The cell is mounted on a particularly stable goniometer head with a height adjustment of 10mm and fits on a Phillips single crystal diffractometer, which has a rather small circle. So the whole equipment can easily be used on different types of four-circle diffractometers.

In our construction the gasket is the only strong absorber left in the paths of the X-rays. The use of beryllium reduces this shortcomings dramatically. Recently pressures up to 80kbar have been reached with these beryllium gaskets.

Fig. 2

16.6-6  IN SITU HETEROGENEOUS CATALYSIS STUDIES AT VARIABLE TEMPERATURES BY AUTOMATED X-RAY DIFFRACTION SPECTROMETRY. By Glover A. Jones, Central Research & Development Department, E. I. du Pont de Nemours & Company, Experimental Station, Wilmington, Delaware 19899, U.S.A.

A fixed-bed microreactor, capable of operating with mixtures of reducing or oxidizing liquids or gases at pressures between 100 millitorr and one atmosphere, and at temperatures between 80° and 1000° Kelvin, has been constructed especially for x-ray diffraction applications. In a model system study, the crystalline phase catalyst transformations during the oxidation of tetrahydrofuran (THF) to Y-butyrolactone over a V.O2-SiO2 catalyst have been examined. The fixed-Bed reactor, operated in isothermal mode and computer-controlled at temperatures between 443° and 488° Kelvin at atmospheric pressure, was used to collect x-ray diffraction data at each programmed setpoint. XRD measurements in situ show significant proportion of V(IV) on carrying out the oxidation of THF on the catalysts. Reduction in H2 at temperatures above 443° Kelvin gives rise to V.O2, which is similar to the effect when the reduction is induced by THF.

16.6-7  HIGH-PRESSURE CELL FOR GUINIER X-RAY DIFFRACTION. By E. Knorre, Institut fUr Kristallo grafie der Universitat Tubingen, West-Germany.

Diamond anvil cells for x-ray diffraction are available up to very high pressures. The disadvantages as very small samples, nonuniformity of the pressure, and inaccuracy of the pressure determination are well-known. For a great number of experiments e.g. investigations of plastic crystals or incommensurate structures pressures below 4 kbar are sufficient. For this range it is possible to construct small gas-pressure cells for hydrostatic pressure conditions and with large sample areas suitable for Guinier diffraction geometry. We have designed and tested such a cell for applications in the temperature range from 100 to 400 Kelvin at pressures up to 4.1 kbar at all temperatures. This pressure cell with a diameter of 26 mm and a height of 20 mm is machined from CuBe alloy, the windows from 2 mm thick Be plates. The slits for the diffracted and the primary beam, respectively, allow diffraction angles from 20 = 60° to 30° and oscillation of the sample of ± 10° relative to the primary beam. As the experiments have shown this movement is necessary because many powder samples develop texture effects as a result of recrystallisation during pressure and temperature treatment. He gas is used as the pressure medium. The pressure of 4 kbar is generated by a small transportable (weight 35 kp) supply unit which is operated manually. The pressure cell has been tested successfully in a reinvestigation of the ammonium nitrate p-T-diagram.