SAPPHIRE CELLS FOR HIGH-PRESSURE OBSERVATIONS

F.E. Wubbolts[1]*, P-J Smits[1], M.J.E. van Roosmalen[2], M.V. Fernandez Cid[2]

[1]FeyeCon D&I BV
Rijnkade 17A
1382 GS Weesp, the Netherlands

[2]Laboratory for Process Equipment, Delft University of Technology
Leeghwaterstraat 44
2628 CA Delft, The Netherlands

Frank@feyecon.com

Measurement of phase equilibria and observation of processes in high-pressure systems yield information that is important for the development of new applications. Two cells of thick-wall sapphire tube were developed, one for fluid phase equilibrium studies and another to observe processes in compressed gases. These systems are designed for a pressure of 350 bar at 150 °C. The equipment is placed in an oven to maintain a constant temperature. The design of the cells allows fast, easy opening and cleaning.

INTRODUCTION

Visual observation of processes at high pressure requires the use of strong and transparent materials. Although Borosilicate glass and Quartz are already quite strong, sapphire has unsurpassed physical properties. In particular its tensile strength is very high and remains almost unchanged up to the melting point of around 2000 K. This makes it the preferred material for visual observations at a high temperature and pressure.

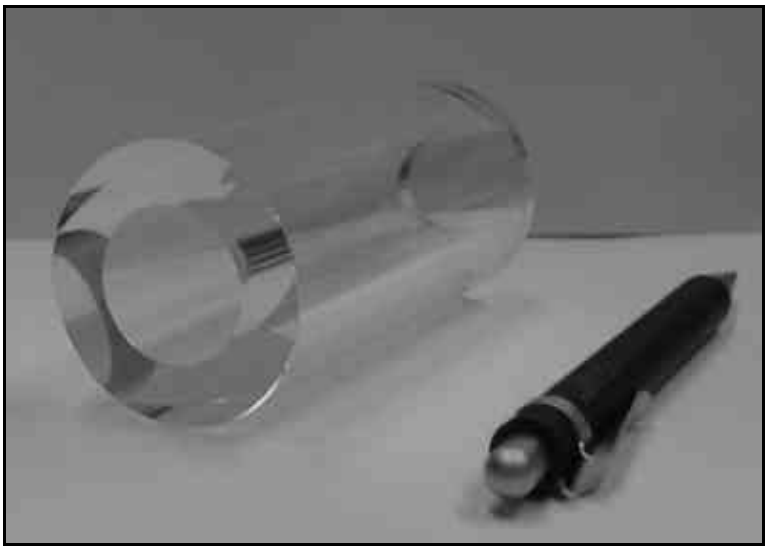

Figure 1 : Thick wall sapphire tube

Autoclaves can be equipped with small sapphire disk windows to observe the processes inside. This allows only a limited view of the contents. Manufacturing technology exists to produce thick-wall tube, like the one shown in figure 1. Based on such a tube a completely transparent pressure vessel has been designed.

Two different vessels have been manufactured based on thick-wall sapphire tubes. One is for high pressure phase equilibria and has a sampling system to analyse the composition of the phases. The other is for the observation of high-pressure processes like textile dyeing.

I – PHASE EQUILIBRIUM EQUIPMENT

Figure 2 shows the phase equilibrium vessel. It consists of two sapphire tubes stacked on top of each other to have an internal volume to 100 ml. The tubes are closed at the ends by two flanges, which are held together by four bolts. Both the cell and sampling system, with Anton-Paar density meter, are placed in an air-oven.

A movable sample rod runs through the cell, with two small tubes inside. These tubes can be positioned in one of the phases. A sample is then taken from and returned to the cell to circulate it over GC and HPLC equipment.

The overall composition is known from the amount of material charged into the equipment. The content of the cell is circulated with a small external gear pump to equilibrate the contents.

The transparent cell permits a clear view of all phases. The tubes´ thick wall even magnifies the contents, so phase changes can be observed clearly.

For cleaning the sapphires can be removed without taking the equipment apart. The way of construction ensures that the sapphire tubes are always mounted in the right position.

Figure 2 : Transparent vessel for phase equilibrium measurement.

II – HIGH PRESSURE OBSERVATION

A second cell of 50 ml was constructed to observe the dyeing of textile with compressed CO_2 as a solvent. The dye-content of the gas phase can be determined with the chromatographic equipment to determine its' distribution coefficient over CO_2 and textile.

Other applications of our sapphire tube arrangement are in polymer engineering to observe mixing of polymers in a static mixer.