HIGH-PRESSURE DIFFERENTIAL THERMAL ANALYSIS

PDTA Cell Design

DTA has been performed in pressure equipment of various geometries by other researchers[4,5] but generally some concession had to be made for the sake of attaining high pressures. The present apparatus was designed to provide optimum geometry and thermal characteristics for DTA in a cell which can be easily pressurized and manipulated. A cylindrical geometry with thick walls provides for symmetrical placement of sample and reference substances and a uniform temperature gradient. Uniform pressure at the surface of sample and reference is assured through the use of nitrogen gas as the pressure-transmitting medium. The apparatus has been used up to 3700 bars and 200°C. By proper design of all components of the system it should be possible to reach 6500 bars and 500°C with this type of high pressure DTA cell.

As shown in Figure 1, the cell is a cylinder 6.25 in. in length, 2 in. in outside diameter, with an 0.1875-in. bore. The bore is closed at both ends by identical plugs which form "cone-in-cone" high-pressure seals. Seals of this type function by forcing a plug with a taper of 58 or 59° into a seat tapered at 60°. The interface is a narrow annulus which carries the entire sealing force on a small area. The two plugs of this cell are drilled axially for the insertion of Chromel–Alumel thermocouples which are encased in a stainless steel sheath of 0.0625 inch outside diameter. The gap between the thermocouple sheath and the bore of the closure is sealed by brazing with a silver alloy. The junction of the thermocouple is electrically insulated from the sheath by a packing of finely powdered magnesium oxide. Four wires emerge from each junction so that the temperature T and temperature difference ΔT circuits originate at the same point in the thermocouple. (The cell was fabricated by Pressure Products Industries of Hatboro, Pennsylvania, who also assisted in its design.)

The tip of one thermocouple is surrounded by a reference substance which does not suffer any thermal transitions in the range of temperature under investigation. A tightly wound cylinder of asbestos paper is suitable as a

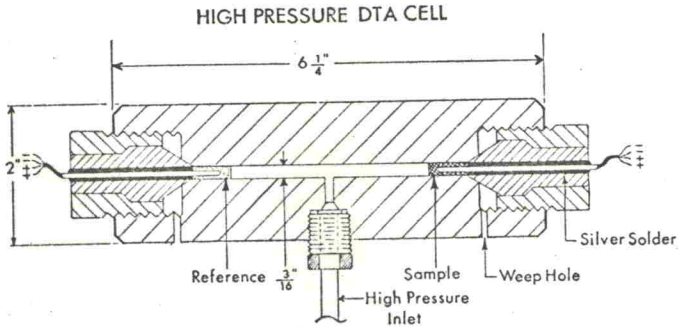

Fig. 1. PDTA cell.