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had to be made for the
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A cylindrical geometry
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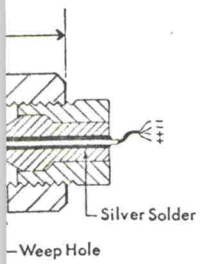

reference. The polymer specimen under investigation must be in the shape of a cylinder to make good thermal contact with the walls of the cell. For the experiments on melting and crystallization of folded-chain polymers under pressure, samples were formed to the proper dimensions in a small stainless steel hand mold. A hole is drilled axially to within 0.125 in. of one end to accept the sheathed thermocouple. Specimens of extended-chain polyethylene were machined to size and drilled to accept the thermocouple. For calibration a dummy specimen was machined from polytetrafluoroethylene. Where the tip of the thermocouple would fit in the usual polymer plug, a cavity of 0.0625 in. diameter and 0.0625 in. length was left in the dummy for insertion of a few milligrams of the calibrant. By this means the sample geometry is preserved for all runs.

The body of the PDTA cell was machined from a high temperature alloy, René 41, which contains 55% Ni, 19% Cr, 11% Co, and 10% Mo. The cell is heated by three strip heaters, each rated for 200 W at 115 V ac. The heating rate is controlled by supplying a chosen voltage to the heaters from a variable autotransformer. The normal heating rate chosen was 4°C/min. To assure good thermal contact, the heaters are bolted to a three-section jacket of pure aluminum which is firmly clamped to the outside of the PDTA cell.

Nitrogen gas under pressure is supplied to the PDTA cell from a 200-cm^3 pressure reservoir. Since the volume of the reservoir is 125 times the volume of the cell, there is no measurable change in pressure as a result of heating the cell during a run. The nitrogen in the reservoir is compressed over oil by means of a high-pressure intensifier in an apparatus described previously.[6] The piston of the intensifier can be manually stroked as many times as necessary to reach the desired pressure. To measure the pressure in the cell, a 6900-bar Bourdon tube gauge (Heise Bourdon Tube Company) is connected directly to the high pressure side of the system. This gauge proved highly reliable in operation and could easily be read to 7 bars. During the course of the present experiments, the gauge was rechecked by the manufacturer by using a dead-weight tester which had been calibrated by The National Bureau of Standards. Up to 5800 bars no measurable deviation was found from the original calibration.

The electronic circuitry of the PDTA apparatus represents a standard DTA measuring circuit.

Analysis of Data and Calibrations

Three points on the ΔT trace were identified: the first deviation from the baseline (A), the peak (B), and the return to baseline (C). For a sharply melting substance, A is the melting point. Figure 2 is a direct copy of the melting and crystallization sections of a PDTA trace made on folded-chain polyethylene at a pressure of 2250 bars. The reproducibility of T_A was usually ± 1°C. The peak in the ΔT trace usually comes at the end of the melting plateau. The peak temperature T_B differs from T_A only