

sure region. In the coaxial heater, the heating current flows in opposite directions in inner and outer sleeves. This greatly reduces inductive pickup in the test wires.

All materials subjected to high temperatures inside the pressure cell were previously fired to avoid their giving off water during the course of the experiment. Small traces of water quickly react with Chromel and Alumel at high temperatures causing contamination and decalibration. The massive talc pressure medium did not get hot enough to dewater and was not fired.

In the experimental assembly shown in Fig. 3 there are two pressure seals, one at high temperature and the other at low temperature. The temperature of each of these seals was measured with a butt-welded Chromel-Alumel thermocouple. Their location is indicated in Fig. 4. The leads of these thermocouples were taken out of the atmospheric pressure side of the seal. Thus pressure did not affect their calibration.

Substantial care was taken to avoid parasitic emf's and leakage currents in the low-level measuring circuits. The emf from each thermoelement in the test was measured with an absolute accuracy of $\pm 2 \mu\text{V}$ on a 1-mV span strip chart recorder. Polarity is indicated in Fig. 2 and is in accordance with that of Bridgman.⁵ At the highest furnace currents, a small amount of inductive pickup was noted. This was removed by a one-stage R-C filter.

EXPERIMENTAL MEASUREMENTS

Four thermoelement materials were tested simultaneously: Pt, Pt10Rh, Chromel-P, and Alumel. The noble metal thermoelements were 0.305-mm-diam reference grade thermocouple wire secured from Englehard Industries: Pt, bar number 72385 and Pt10Rh, bar number 73740. The Chromel and Alumel were supplied by Hoskins Inc. in the form of 0.320-mm-diam wire: Chromel-P coil, number 3831 and Alumel coil, number 6404. All four materials were received in the annealed condition. No further annealing was done, though care was taken to avoid undue bending of the wires during assembly of the experiment. The thermoelements were cleaned with petroleum ether before assembly and then handled only with cleaned tweezers. This was done to avoid the introduction of contaminants on the surface of the wires.

Whereas Pt and Pt10Rh seemed clean initially, Chromel and Alumel required substantial cleaning. Argon was flushed through the 1-atm high-temperature region for 1 h before the first temperature excursion. This was done to insure effective removal of oxygen. The argon flow was then continued throughout the experiment.

Pressure was first raised to a nominal value of 1 kbar. The pressure on the wires inside was probably variable and less than 1 kbar. The first temperature cycle was made at this extremely low pressure to the highest temperature to make sure there was no single-wire emf in the absence of pressure. Data was recorded on the

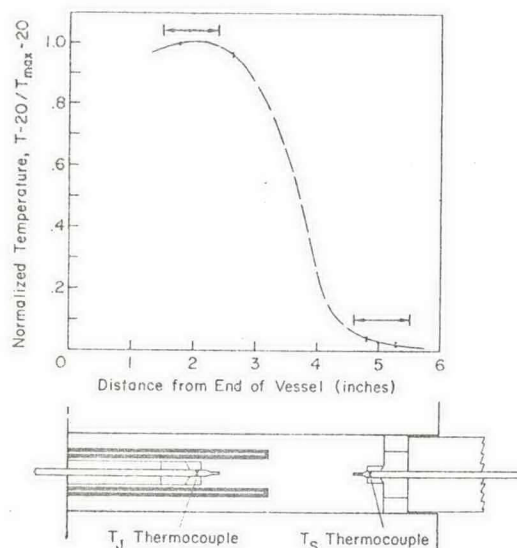


Fig. 4. Single-wire experiment temperature distribution, normalized temperature along the pressure cell as determined by five fixed thermocouples. Data was taken at maximum temperatures of 300°, 600°, and 900°C. Arrows indicate maximum extent of the pressure seals.

increasing and decreasing parts of the cycle. The pressure was then raised.

Both isobaric and isothermal excursions were made across the explored region of the pressure-temperature plane.

In the isobaric excursions the piston load was generally taken to a fixed value and maintained. This meant regulating the oil in the piston ram to counter the effects of thermal expansion and contraction. Temperature was typically cycled between room temperature and 1000°C.

At each data point the hot seal temperature was held constant for $\frac{1}{2}$ –1 min before the single-wire voltages were recorded. During this time, one of the single-wire voltages was monitored on the strip chart recorder in order to observe any time dependency. One-half minute was typically sufficient to establish suitable stress and temperature equilibrium in the cell to terminate all short-term drift of the single-wire voltages. Occasionally the hot-seal temperature was maintained constant for 15 min to observe any longer-term changes in the voltages. This was generally done at the highest temperature of each cycle. Data were recorded on both the increasing and decreasing parts of each temperature cycle to observe any hysteresis introduced by thermal expansion induced changes in stress on the wires. Temperature excursions were made at 12, 23, and 33 kbar; and then again at 23 and 12 kbar to check reproducibility.

The values recorded at each data point were the single-wire voltage for each of the four thermoelements, hot pressure seal temperature T_H , cool seal temperature T_C , oil pressure in the piston ram, and the position of the piston as monitored with a dial gauge.