



Fig. 2. Cross-sectional view of the assembled high-pressure vessel and the high-temperature internal furnace.

was 1 in. in diameter by  $6\frac{1}{2}$  in. in length. The vessel was water-cooled during each of the runs.

Thermocouple and power leads were conducted from the vessel through Chromel, Alumel, and steel cones seated in the pressure plug with unfired lava A (American Lava Corporation) conical insulators. Chromel and Alumel (B. & S. gauge No. 24) were selected as the thermocouple materials because of the small pressure effect on the emf output reported by Bundy.<sup>21</sup> The high-pressure side of the plug was covered with Eccobond 76 epoxy to fill the gaps between the plug and the metal cones. These gaps were caused by a crumbling away of the insulating cones when the metal cones were pressed in initially with an arbor press. This application of the epoxy virtually eliminated electrical arcing of the power lead cones to the plug, which had frequently occurred prior to this modification.

The internal furnace consisted of a  $\frac{1}{2}$ -in. inside-diameter-threaded core closely wound (16 turns per in.) with 0.016-in. platinum wire, an insulating sheath surrounding this core, and a three-piece sample holder and thermocouple support assembly. All five pieces were machined from lava A. Figure 2 shows a cross-sectional view of the internal furnace and pressure vessel.

The diffusion specimen was situated near the center of the furnace with its axis of symmetry normal to the horizontal axis of symmetry of the furnace. Its bottom face, the face to be sectioned, rested on a platinum disk 0.003 in. thick. Chromel and Alumel wires were spot-welded to the bottom side of this platinum disk, creating a large thermocouple junction

spread across the face of the specimen. This type of thermocouple was adopted for several reasons. Early tests with spot-welded Chromel-Alumel junction thermocouples indicated that temperature gradients of several degrees existed near the center of the furnace over a region as small as the extent of the specimen diameter. Also, large vertical temperature gradients were observed between a specimen and a Chromel-Alumel junction when the specimen and junction were separated by a thin mica sheet. Furthermore, the early Chromel-Alumel junction thermocouples, used primarily in conjunction with mica sheets, showed large and irregular deteriorations in their emf characteristics in subsequent calibrations. The platinum disk thermocouple assembly was found to reduce greatly the magnitude of the thermocouple deterioration which had been observed with the previously used thermocouple assemblies. The disk also provided a means to eliminate the vertical temperature gradients and to give a better indication of the average temperature of the face of the specimen to be sectioned.

After most diffusion anneals, the platinum disk was found to be adhered slightly to the face of the specimen, but it was always possible to remove it with little or no discernable damage to the specimen face. A new thermocouple, including platinum disk, was used for each run.

A special calibration furnace was built in an effort to approximate the steep temperature gradient in the internal furnace from the center of the furnace to the plug. Each thermocouple was calibrated in this furnace after the diffusion anneal against new Chromel and Alumel from the same spools which were spot-welded symmetrically with the original wires on the opposite

<sup>21</sup> F. P. Bundy, *J. Appl. Phys.* **32**, 483 (1961).