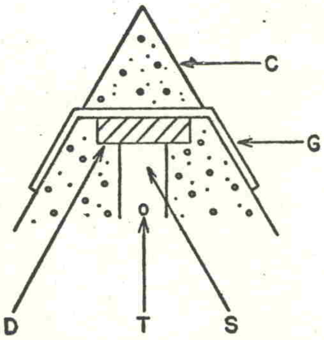

FIG. 2. Detail of sample assembly, without graphite heating tube.

the pyrophyllite is forced to flow into the space between adjacent anvils when they are driven together, thus forming a compressible gasket. The pyrophyllite has sufficient internal and surface friction to do this, and yet not so much as to be unsuitable in its pressure-transmission properties. The surface friction of the pyrophyllite, which is particularly important for the formation of a gasket that will hold in place between adjacent carbide faces without blowing out, is enhanced by painting the outside surface of the tetrahedron with red iron oxide powder. The gasket thus formed, in a run up to 105 000 atmospheres, is about $\frac{1}{4}$ in. in width and 0.020 in. in thickness.

The sample itself is in the form of a small cylinder, $\frac{1}{4}$ in. long and $\frac{1}{8}$ in. in diameter, aligned through the center of the pyrophyllite sample-holder, coaxial with a line joining the mid-points of two opposite edges. As shown in Fig. 2, the sample S is simply placed into a cylindrical hole drilled in the pyrophyllite, and bounded on the ends by mild steel plugs D, which are $\frac{1}{16}$ in. thick and $\frac{5}{32}$ in. in diameter. (These were found to effectively prevent extrusion of the sample through the ends of the container.) Electrical connections are made to the sample through 0.005-in. metal "contact tabs" G, and thermal insulation is provided at the ends of the sample container by the pyrophyllite prisms C. The metal tabs G from the ends of the sample each make contact with the faces of a pair of anvils which bring in the ac heating current.

It has also been found advantageous to place triangular steel "clamping tabs" (cut from 0.005-in. shim stock) over the two edges containing the removable pyrophyllite prisms (see Fig. 3). When the anvils are just being driven in against the pyrophyllite tetrahedron, particularly before the gasket is formed, there is a tendency for the edge assembly on each end of the sample to become misaligned, sometimes allowing the sample to extrude. These steel tabs seem to prevent this problem by holding the assembly in place during the formation of the gasket. When significant pressures are applied, the pyrophyllite breaks through the tab and forms a normal gasket, so that the clamping tab does not interfere with a symmetrical load being applied to the sample.

The temperature of the sample is measured by means of a platinum-platinum+10% rhodium (P-PR) thermocouple, the hot junction of which is embedded directly in the center of the sample at T. The wires, each 0.010 in. in diameter, are fused together at the hot junction. The cold junction is maintained at 0°C in an ice-water bath outside the apparatus, and the thermal emf is recorded automatically on a strip-chart recorder. The leads from the hot junction are brought out through opposite edges of the pyrophyllite tetrahedron, through the gaskets formed between adjacent anvil faces. It was found that with this arrangement, the sample had a tendency to flow out along the thermocouple leads upon melting, thus short-circuiting the thermocouple. (This effect was particularly pronounced with the indium, which is the more fluid of the two substances investigated. In some cases, the flow was of such magnitude that indium could be found out at the edges of the tetrahedron following a run.) This would cause the thermocouple to read some kind of an average temperature over the short-circuited region, which would introduce error by yielding a temperature reading considerably lower than the sample temperature. In attempts to eliminate this source of error, two other types of sample assemblies were used. In one case, the sample was contained in a Nichrome sleeve, of $\frac{1}{8}$ in. outside diameter and 0.005-in. wall thickness, containing end caps, with the thermocouple junction spot-welded to the outside of the sleeve. The other assembly contained the sample in a graphite sleeve of $\frac{1}{8}$-in. o.d. and about 0.014-in. wall thickness, bounded by $\frac{1}{32}$-in. graphite end plugs (as shown in Fig. 1), with the thermocouple junction embedded in the pyrophyllite just outside and adjacent to the sleeve. (In both of these arrangements, assembly was effected by using two pyrophyllite half-tetrahedrons, such that the axis of the sample lay in the dividing plane.) All three types of sample assembly were used with each of the materials investigated.

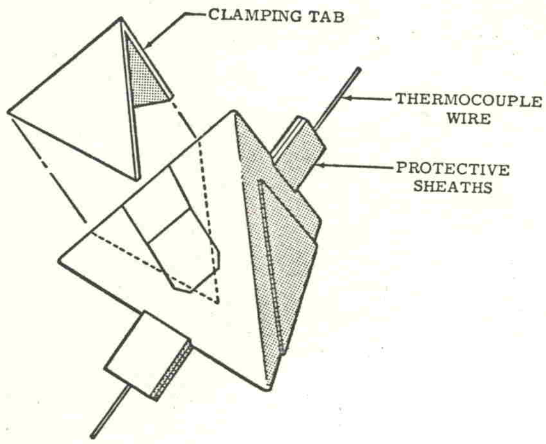

FIG. 3. Assembled sample, showing protective sheaths for thermocouple.

In order to prevent the thermocouple leads from being