licating a critical end point for although Pontiatovskii stated ›int where the heat of transi- ected was not necessarily the he end point.

ork by HERMANN and SWEN- r and SWENSON(23) confirms sition volumes decrease with re and pressure along the and Swenson extrapolate and 20,000 atm as the point ›lume change. Of importance vill be the fact that Beecroft ed a spreading out of the higher temperatures and a in thermal expansion of hase II) at 20,000 atm, over pressure. The earlier work WENSON(22) revealed con- the transition at room tem- ater work, using the cerium revealed little or no hys- e.

NTAL METHOD

r to that previously described A sketch of the supported is given in Fig. 1. The com- ds less than 6 cm high and is at the base of a small press. spindle is inserted into the elco diffractometer as seen in rrangement, Fig. 2.

is encased in a steel jacket into the hardened steel outer gement there is a selection of C and corresponding piston or different sample sizes and mp system now in use. The r used throughout the study pellet is separated from the ·25 mm. beryllium pellet to X-rays. The X-ray beam outer casing (Fig. 1, inset), der and pellet, and onto the th is similar. The dial gauge cator, the main function of ve sample extrusion or de- ts of the vessel.

e heating jacket made from) Nichrome V wire wound hioned into a zig-zag belt. n inner and outer layer of)°C, or by layers of Transite loth held together by an

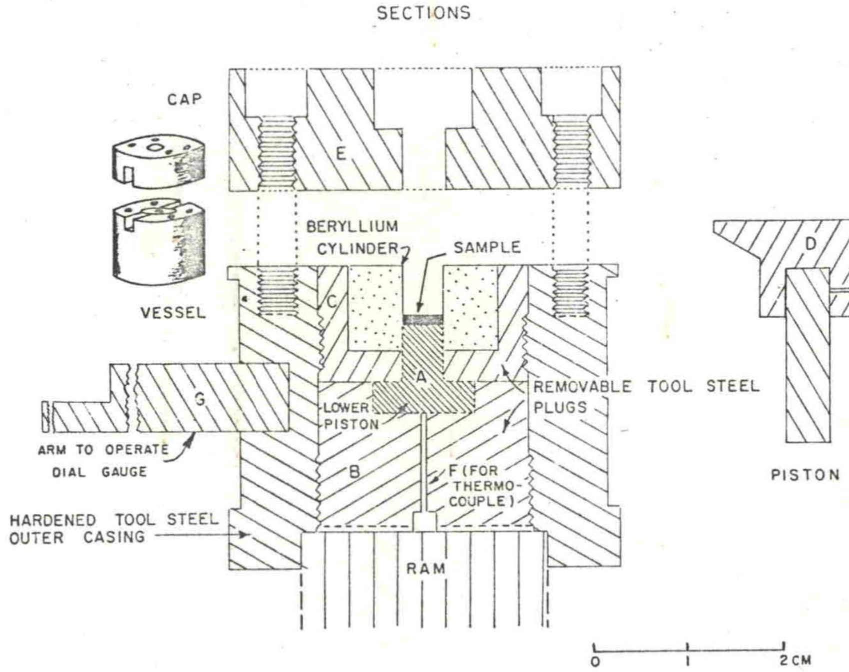

FIG. 1. Beryllium pressure vessel, sectional view, including cap and piston. Beryllium cylinder is stippled. Inset shows view of vessel and cap, and relations between casing slots and beryllium cylinder.

exterior band of copper or brass sheeting (see Fig. 2). A special cooling jacket was constructed to fit around the ram and goniometer spindle.

The temperature was measured by means of a Chromel-alumel thermocouple insulated and inserted into hole F of plug B, Fig. 1. The temperature was read from a Leeds-Northrup potentiometer (with a "reference junction" to approximate the cold-junction correction). Insertion of a mercury thermometer into the bomb window at 175°C gave results within two degrees of that read from the potentiometer. Work with the transition point of KNO_3–II–KNO_3–I (128·3°C) showed that thermocouple readings were correct to within ±2 deg. A similar uncertainty can be assigned to the results of this investigation. Excessive heat flow out through the vessel was eliminated by placing a 4-mm disk of Transite between the piston base and plug base (B, Fig. 1).

Preliminary experiments were carried out to determine the difference between nominal and internal pressures. This was done by making a standard run using a highly compressible material, such as KI or RbI, as a sample. Previously determined equations giving $\Delta v/v_0$ as a function of pressure allow calculation of internal pressures from peak shift on the diffraction chart. To correct for the peak shift due to change in sample height (mainly due to compression of Transite pad and beryllium pellet above the sample) a thin layer of diamond powder was placed on top of the sample pellet. Effective reflecting depths of diamond powder and sample material would be almost identical after a slight amount of pressure was applied to imbed the diamond into the sample.

The calibration runs carried out specifically for this study agreed closely with results obtained previously with the same vessels. Using a piston that fits easily into the cylinder, there is very little frictional loss of pressure on going up in pressure. After perhaps 20 kb had been attained the return run indicated considerable lag of the internal pressure above nominal pressure. At $P = 0$ (nominal) on the return run, the internal pressure may be as much as 4 kb. One up-pressure run at 150°C indicated that there is less frictional resistance than at room temperature, after taking into account the change in compressibility of sample with temperature. There is still some pressure uncertainty stemming from the deformation of the beryllium pellet and vessel walls, especially at higher temperatures. This deformation is most likely responsible for the pressure lag on the return leg of the runs as well as increased frictional resistance when repeating a run with the same sample and beryllium pellet used previously. A pressure uncertainty of ±1 kb is appended to all pressure values, and at temperatures below 100°C this figure is considered to be quite conservative.

Radiation used was MoKα, with applied power of 40 kV and 20 mA. A scintillation counter combined with pulse-height analyser comprised the detection,