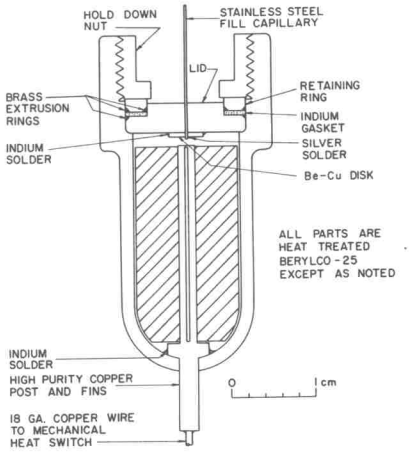

Fig. 1. The details of the high-pressure bomb which contains the solid neon.

to a high purity copper post (2 mm in diameter as it passes through the bomb) which in turn is indium soldered to the bomb for a pressure seal. The hemispherical bottom end of the bomb reduces stress concentrations which were found to be serious in previous purely cylindrical designs. This bomb, which has a diameter ratio of 1.25, was used to 2.5 kbar at 77 K; a previous version with a diameter ratio of 1.23 burst at room temperature at 2.6 kbar.

The room-temperature volume of the bomb was determined by weighing it before and after filling with degassed distilled water. The result as corrected to 0 K[21] is $2.902 \pm 0.002\,\mathrm{cm}^3$. The bomb was immersed in a pyncnometer in an ice bath in a second experiment, and its volume was found to change reversibly and linearly with internal pressures of up to 2.5 kbars with a coefficient $\kappa' = V^{-1}(\partial V/\partial P)_T = 7.36 \times 10^{-6}\,\mathrm{bar}^{-1}$. The assumption is made that the volume of the metal does not change with pressure. This parameter should be proportional to the Young's modulus for beryllium copper,[22,23] and hence should decrease to $\kappa = 6.62 \times 10^{-6}\,\mathrm{bar}^{-1}$ for temperatures below 77 K.

The sample heater (550 Ω, 0.001-in.-diam Pt–8 %W wire) and the germanium resistance thermometer are attached directly to the copper wire which comes from the fins (Fig. 1). This wire also leads directly to a mechanical heat switch which is operated from the top of the cryostat and which is identical with one which we have used previously.[13] Initially, sample heat was supplied by a 1000-Ω heater wound on the heavy top portion of the bomb. Rather long time constants were observed when this heater was used and all data eventually were taken using the sample heater on the wire. The

initial heater proved to be useful, however, to prevent the capillary from blocking prematurely while the sample solidified at constant pressure.

The heater power and the thermometer resistance are determined using conventional potentiometric techniques with current reversal. These, and the timing methods, are almost identical except for a few details with those which we have used previously.[24] The major improvements have involved the use whenever possible of constant current supplies of the type described by Kroeger and Rhinehart.[25] These give indicated thermometer current stabilities of at least 0.001 % (as determined by potentiometer readings across a standard resistance), and allow a choice of 20 different sample heater currents (which vary discretely from 1 μA to 14.5 mA) which are reproducible to 0.01 %. Chart recorders indicate the off-balance readings of null detectors that are associated with the various potentiometers.

The germanium thermometer is calibrated from 0.9 to 20 K in terms of a paramagnetic salt temperature scale[26] that is referenced to the NBS-1955 platinum resistance thermometer scale above 20 K. The thermometer calibration is in terms of this platinum resistance scale from 20 K to 77 K, and a check after these experiments were completed showed it to be unchanged to 0.01 %. The thermometer leads are anchored to the bomb walls to ensure that the thermometer indicates the bomb temperature.

2.2. Procedures and Ancillary Apparatus

Gas pressures of up to 4 kbar can be produced with an air-driven hydraulic pump and a 2-in.-i.d. by 37-in.-stroke, O-ring piston, gas–oil separator manufactured by Autoclave Engineers, Inc. Great care is taken that no oil is transmitted over with the gas, and a special proximity gauge indicates the exact position of the piston for the last two inches of its travel. The bomb is purged with clean neon several times at room temperature, and then is sealed off at approximately 2 kbar pressure prior to cooling to 77 K. Liquid helium then is transferred to the main reservoir and the sample shield is cooled to 25 K where its temperature is held constant. During this time, the desired freezing pressure has been maintained in the separator (as indicated by a Heise gauge in the oil side) and the bomb, and the capillary and the bomb have been heated to prevent their being blocked. The bomb is cooled slowly from the bottom by adjusting the heat-switch pressure, and the onset of freezing is marked by a rapid decrease in the cooling rate of the bomb at a temperature which corresponds with published values.[19,20] The sample again cools rapidly when freezing is completed. Helium exchange gas then is admitted into the capillary vacuum space to block the capillary over a major portion of its length. The melting temperature is redetermined at this point by measuring the heat capacity of the bomb at small intervals of temperature