Rev. Sci. Instrum., 40, 169-70 (1969)

Clamp Cell for High Pressure–Low Temperature X-Ray and Mössbauer Resonance Studies*

C. W. CHRISTOE AND H. G. DRICKAMER

Department of Physics, Department of Chemistry and Chemical Engineering and Materials Research Laboratory, University of Illinois, Urbana, Illinois 61801

(12 August 1968; and in final form, 27 September 1968)

THE clamp cell for obtaining high pressures at low temperatures was first developed by Chester and Jones.[1] Buckel and Gey[2] have extended its use to pressures in excess of 160 kilobars. This note describes a cell for x-ray and Mössbauer resonance studies at high pressure and low temperature. The pressure is generated between two Bridgman anvils with flats 0.203 cm in diameter, made from grade 999 Carboloy, work hardened, and loaded as described in a previous paper[3] on Mössbauer resonance equipment for use near room temperature. The pistons make a slip fit in the cell, which is shown in exploded view in Fig. 1. The body contains an inlet hole and a 90° slot for egress of x rays or γ rays. It can be pinned precisely in place in the x-ray spectrometer or Mössbauer apparatus by means of three equally spaced holes in the base.

The cap is hexagonal to permit application of torque. The cell can be made of hardened beryllium copper or of hardened alloy steel depending on the amount of pressure intensification desired in cooling. Above and below the positions are shims. In Fig. 1 the contact parts of these shims are Carboloy, but other materials are possible. The design minimized the possibility of rotating one piston with respect to the other. The pistons and cells can be made in a variety of dimensions without affecting the calibration as long as the flat diameter of the pistons is maintained at 0.203 cm. Two sets are shown in Table I.

Pressure is applied hydraulically with a Carboloy piston through the 1.270 cm diam hole in the cap. Torque is obtained through an hexagonal nut fitting over the cap which is turned by a torque wrench. The pressure calibration procedure used was as follows. The cell was loaded as described in a previous paper[3] on high pressure Mössbauer techniques, then the x-ray spectrum of the marker was obtained. The cell was placed in the press, and pressure, then torque was applied, and a second spectrum taken. If the ratio of torque to applied force was kept in the range of 0.2–0.27 cm (i.e., a ratio of torque to pressure on the flat of 0.006–0.008 cm^3), the exact value did not affect the calibration. The pressure obtained in the cell was 0.82 ± 0.07 times the pressure obtained by direct measurement under pressure[3] using a hydraulic press. Substances used include Al, NaF, MgO, and CaO. The cell was then cooled in place to liquid nitrogen temperature. Because of differences in thermal expansion, a pressure intensification resulted, which was more reproducible than the applied pressure–clamp pressure correlation discussed above. Examples for a beryllium–copper cell and a steel cell are shown in Fig. 2. It is quite practical to reach pressures of 240 kilobars. The limitation is the blowout of the sample during cooling. The use of other pressure media, or of the supported taper cell,[4] could well permit much higher pressures.

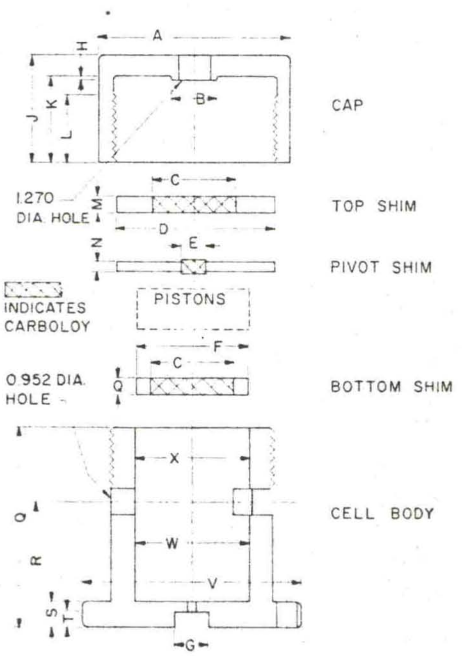

FIG. 1. Cell. Cross-section assembly view.

TABLE I. Typical cell dimensions.

Dimension	Large cell	Small cell
A	7.303 cm	5.397 cm
B	2.381 cm	2.381 cm
C	3.175 cm	—
D	6.180 cm	3.925 cm
E	0.952 cm	0.900 cm
F	4.330 cm	2.857 cm
G	1.270 cm	1.270 cm
H	0.100 cm	0.100 cm
J	4.128 cm	4.921 cm
K	3.334 cm	3.334 cm
L	2.540 cm	2.857 cm
M	0.635 cm	0.476 cm
N	0.430 cm	—
P	0.635 cm	0.635 cm
Q	7.620 cm	6.509 cm
R	4.763 cm	3.492 cm
S	0.952 cm	0.952 cm
T	0.635 cm	0.635 cm
U	3.651 cm	2.540 cm
V	8.414 cm	6.350 cm
W	6.350 cm	4.128 cm
X	4.445 cm	2.857 cm