

Table 1. Characteristics of high-pressure apparatus of type a and type b

Types	Method	Pressure limit	Effective volume	Press	Temperature controller	Pressure cell
Type a	Multianvil system; the area of anvil tip is 20 x 20 mm <sup>2</sup>	Because of the principle of massive support, the pressure (4.5 ~ 40 kbar) higher than the withstand pressure of tungsten carbide can be obtained	$\pi \times (11/2)^2 \times 13 \text{ mm}^3$	Six 600-t cubic presses	Yes	complicated (Fig. 1)
Type b	Piston cylinder system; cylinder metal diameter is 20 mm	The pressure range is limited to 0 ~ 30 kbar because of the withstand pressure of tungsten carbide which forms a high-pressure vessel	$\pi \times (12/2)^2 \times 18 \text{ mm}^3$	500-t Kennedy dual presses	No	simple (Fig. 3)

Lead foils are attached around the capsule cap to shield the vaseline as well as to facilitate smooth insertion of the cap into the capsule body.

Vaseline (fluid pressure medium) allows one to apply the same static pressure as oil. Even a fragile crystal plate 0.16 mm in thickness can be tested without damage by applying high pressure.

When a cubic press is used, one of the sample electrodes is connected to the ground via one of the anvils and the other electrode is connected to ⊕ terminal of the meter via one of the anvils. In our experiment, one of the electrodes ③ of the sample ④ is grounded together with all anvils via 0.3 mm-diameter copper wires imbedded in pyrophyllite. The other electrode is connected to the ⊕ terminal of the meter via gaskets using 0.5 mm-diameter nichrome wire ⑩ covered by a Teflon tube ⑪ which can reduce stray capacitance and leakage conductance. Nichrome wires can withstand a large tensile stress.

Below 4.5 kbar gaskets are not effectively formed in the pressure cell and unstable pressure is applied to the sample. The stray capacitance due to the pressure varies from cell to cell, and therefore, no dielectric measurement was made below 4.5 kbar.

Figure 2 shows the pressure dependence of stray capacitance of the positive lead wire in the apparatus without the sample. The variation of the stray capacitance  $\Delta C_s$  increases with the pressure. When the sample is inserted into the apparatus, the variation of the effective stray capacitance of the sample is evaluated as  $\Delta C - \Delta C_s$ , where  $\Delta C$  is the capacitance of the sample including the stray capacitance of the lead wire.

Denoting by  $C_0$  the capacitance of the sample at the atmospheric pressure, and by  $\epsilon_{r0}$  specific permittivity, the specific permittivity of the sample at the pressure  $p$  (kbar) above 4.5 kbar is

$$\epsilon_{rp} = \epsilon_{r4.5} + \epsilon_{r0} \Delta C_s / C_0$$

where  $\epsilon_{r4.5}$  is the specific permittivity at 4.5 kbar,  $m$  is the material constant and  $\tau$  is the correction factor of the capacitance

$$\tau = 1 - mp$$

where  $m = 3.0 \times 10^{-4}/\text{kbar}$  for crystal and  $7.5 \times 10^{-4}/\text{kbar}$  for powder [8].

#### (b) Type-b, high-pressure apparatus

A cylindrical pressure cell is placed inside a pressure vessel to which pressure is applied by a Kennedy-type 500 t press (see Fig. 3). The cell consists of a pyrophyllite cylinder ⑨ (o.d. 20 mm, i.d. 13.2 mm and length 28 mm) with both ends covered by steel caps ⑦ and ⑧. The cell is filled with silicon grease in which sample ⑩ is imbedded. The inner wall of the cell is tapered at 1° along the axis. The lead foil ⑥ is attached on the contacts between the cell inner walls and caps. This foil facilitates realizing good contact between the cell and caps when high pressure is applied to caps via the pressure vessel.

An  $\text{Al}_2\text{O}_3$  insulating tube (dia. 2 mm) ① is inserted into the sample cell via four holes (dia. 0.4 mm) of the upper cap ⑦. This tube contains a pair of enamel copper wires which connect a thermocouple ② on the sample surface to the microvolt meter. Gaps between the holes and two pairs of lead wires and between the holes and the insulating tube are filled by epoxy resin to shield the silicon grease.

Manganin-wire coil ③ is installed on the inner wall of pyrophyllite cylinder ⑨ inside a cell to control the sample temperature. The lead wires are connected to the upper and lower caps which supply power to the wires. The pressure vessel ④ and end plate ⑤ are electrically insulated by use of mica plate ⑥.

The oil pressure was corrected on the basis of the principle that electrical resistance changes rapidly at the phase transition points I-II (25.5