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Elastic properties of glasses and glass ceramics at high pressures and high temperatures

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should only enter after the echoes from the previous pulse have died away and this pulse frequency must therefore be set accordingly. If the pulses are sent in at too slow a rate then the human eye does not see a continuous display on the scope, the trace becomes very faint. If sent in too fast, the trace will become unnecessarily bright and the r.f. unit could be overloaded. This frequency is also controlled from the strobe and divider box.

The role of the dual delay generators is to be mentioned here: They are merely a pair of strobes used to intensify a particular portion of the signal. They operate in synchronization with the x-sweep frequency. By means of a variable delay they are used to intensify two chosen portions of the signal such as the first and second echoes. The strobes are located on the two echoes to be overlapped and the intensity of the oscilloscope is reduced. Only the portions illuminated by the strobes will be visible. Their positions, widths and intensity are adjustable using controls.

Sometimes the echo pattern may be quite weak and very little information can be gained. One reason for this is the impedance mismatching of the r.f. unit and transducer which can lead to a significant power loss. To counteract the difference in the impedance of the r.f. unit and the transducer an impedance matching network is included in the circuit. This provides a good match between the high impedance of the transducer and the lower impedance of the r.f. unit.

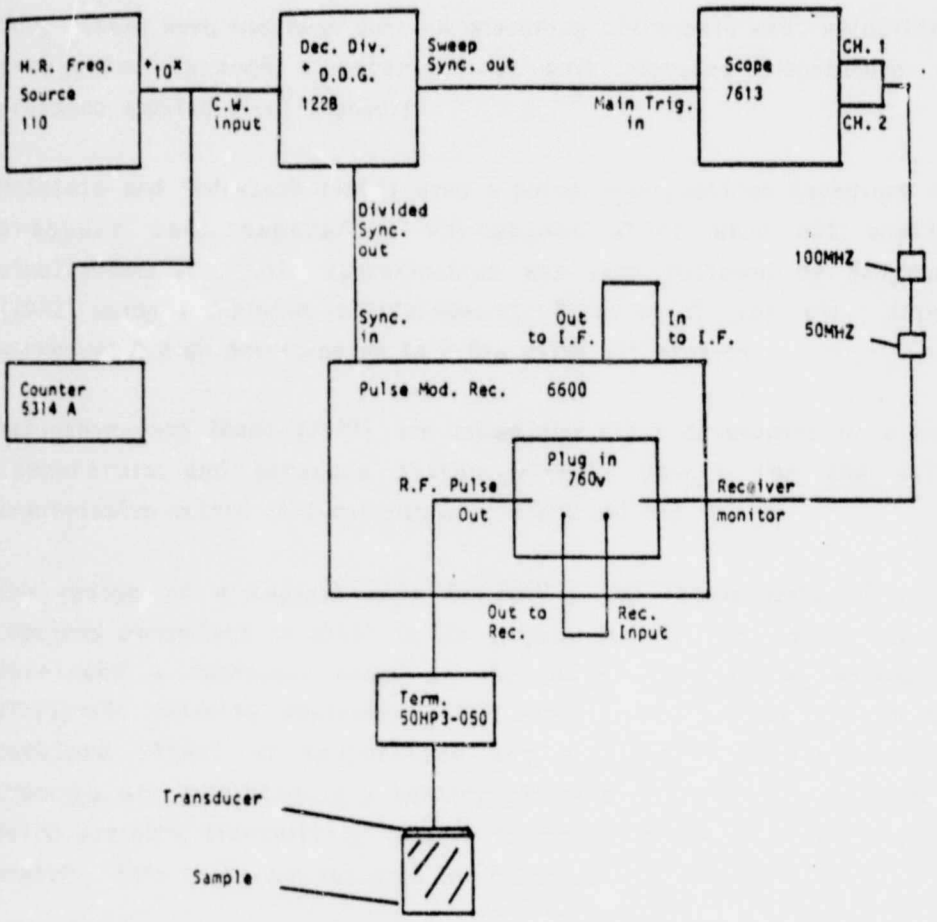


Fig. 3.3 Circuit diagram of the MATEC system

3.1.3 The pulse echo overlap method at high pressure and high temperature

Very little work has been done on measuring ultrasonic wave velocities, propagated through a material, at both elevated temperature and pressure applied simultaneously.

McSkimin and Andreatch (1962) used a pulse superposition technique for pressure and temperature variations which were not applied simultaneously. This approximation was also followed by Manghnani (1972) using ultrasonic interferometry. Kondo et al. (op. cit.) mainly worked with high pressures up to 3 GPa using ultrasonics.

Matsushima and Akeni (1977) are among the first developers of a high temperature and pressure piston-cylinder device for the pulse transmission [illegible]essures of 2.0 GPa and 800 °C.

The design [illegible]e able to fulfil the requirements of these combined parameters is still in its infancy (Levitt, op. cit.). Levitt developed a technique based on the pulse echo overlap method of ultrasonic velocity measurement for materials as a function of the combined effect of temperature and pressure. Quasi-hydrostatic pressure was generated in a Kennedy piston-cylinder apparatus using a solid pressure transmitting capsule incorporating an internal graphite heater. This technique was used in the present study.

3.2 High pressure and high temperature apparatus

The experiments are conducted in a 10 MN Kennedy Press (figure 3.4), where quasi-hydrostatic pressure is applied by driving the piston into the cylinder. The press essentially consists of two rams (a and b), their frames, and hydraulic pumps required to move the rams. The press is specifically designed to deal with piston-cylinder devices.

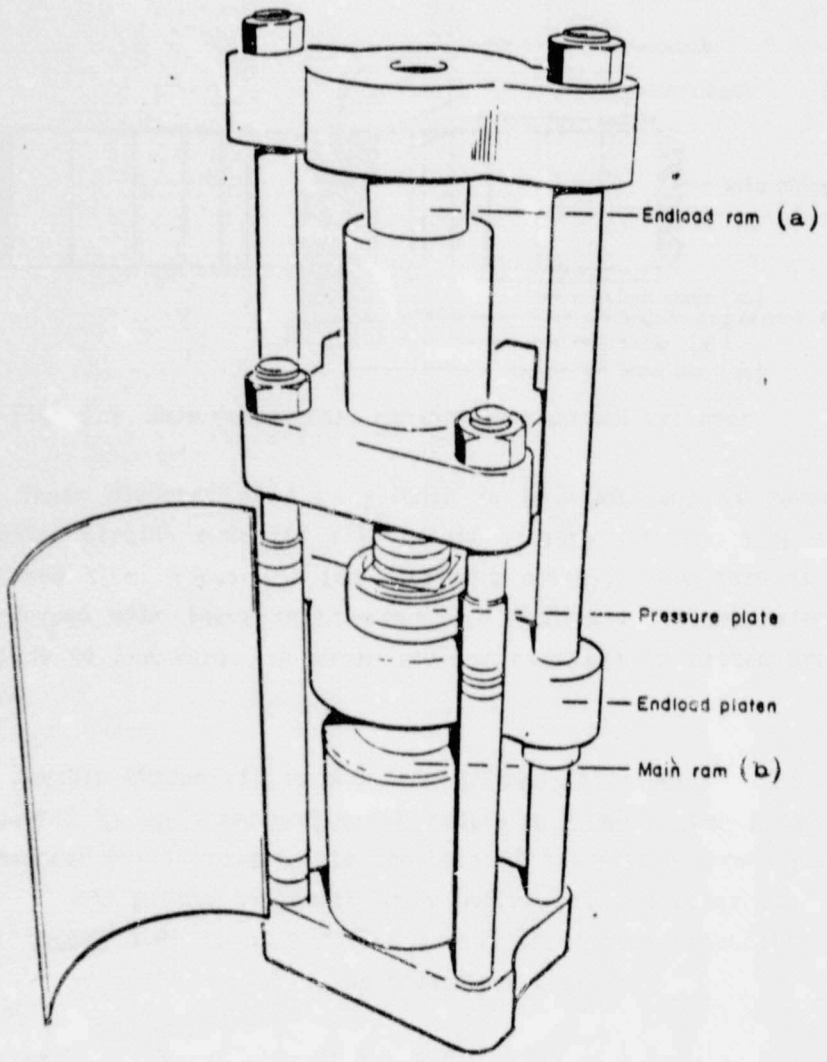


Fig. 3.4 The Kennedy Press

The main components of the piston-cylinder apparatus and the capsule containing the sample are shown in figure 3.5.

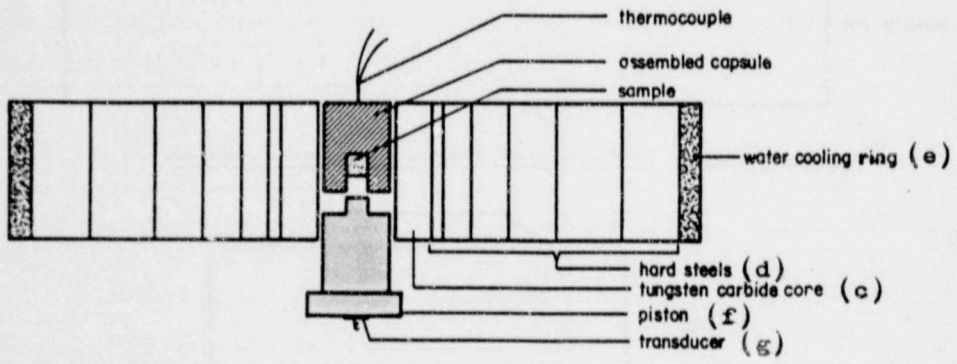


Fig. 3.5 Main components, capsule, piston and cylinder

The inner diameter of the cylinder is 25.4 mm, and is formed by a tungsten carbide core (c) (12% cobalt binder) surrounded by a set of hardened steel rings (d) (the pressure plate). The core is heavily reinforced with these interference-fit binding rings supplying radial support to the core. A water cooling ring (e) is fitted around the plate.

The movable piston (f) is made of hardened T15 steel, and is radially supported by an interference-fit retaining ring on the bottom. The transducer (g) is cemented to the base of the piston with a conductive epoxy. The piston thus acts as a buffer rod. A water cooling ring (see figure 3.8) is also fitted around the piston especially to keep the piston in the region of the transducer cool.

The tungsten carbide core of the pressure plate had to be supported on its flat circular faces, because tungsten carbide can withstand much less tensile stress than compressive stress. Cracking in a plane parallel to the faces is also prevented by this support. Figure 3.6 shows all the components used in the stack. The support on the faces of the core is achieved by clamping the pressure plate and the other stack components in the working space of the Kennedy Press. The clamping force is provided by the secondary hydraulic ram of the press and is transferred as uniaxial support to the core via two hardened steel shims (h) positioned on its flat circular faces.

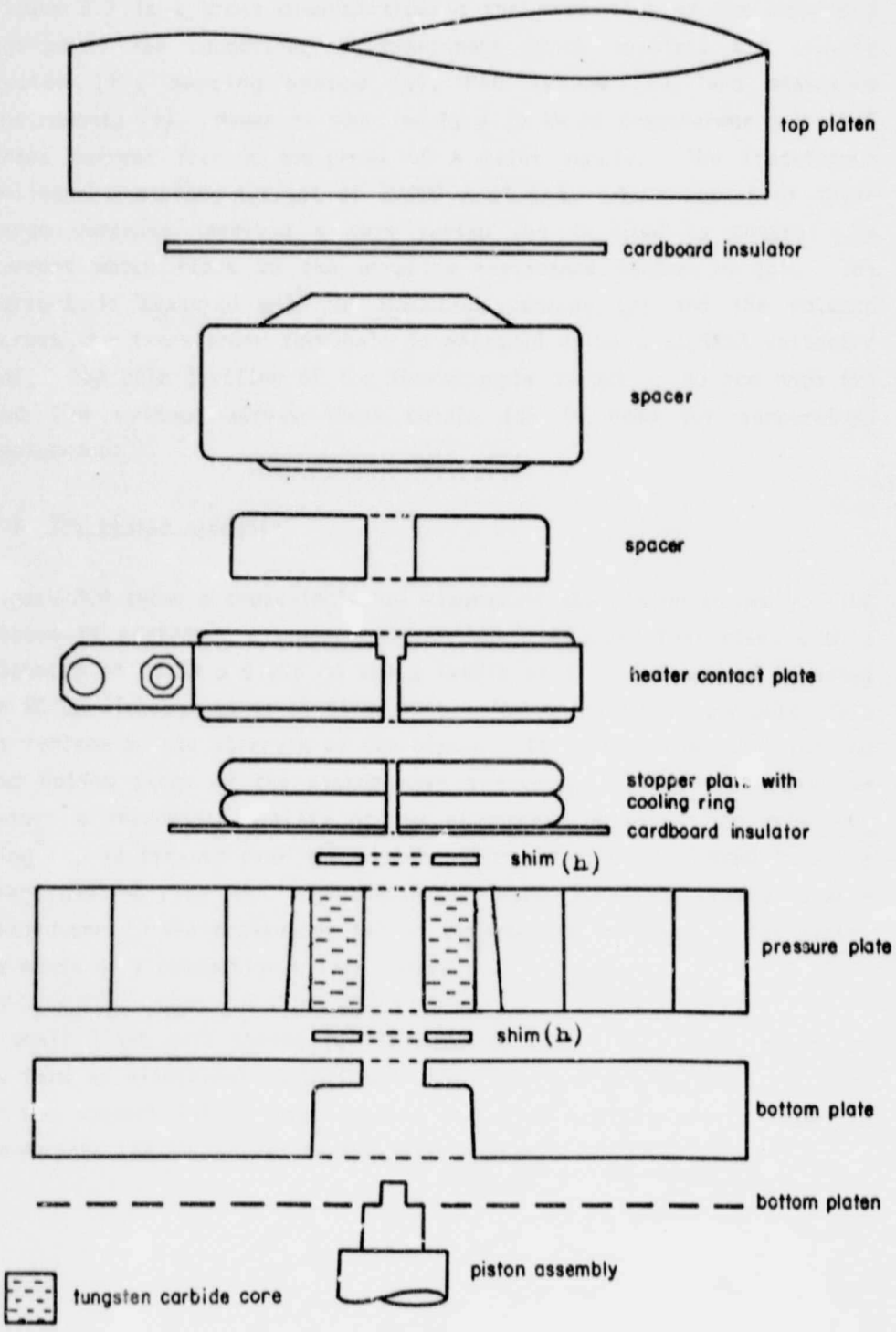


Fig. 3.6 The cylinder stack

Figure 3.7 is a block diagram showing the connection of the auxiliary equipment for conducting an experiment which involves the cooling system (i), heating system (j), PEO system (k), and measuring instruments (l). Power is supplied by a 10 kW AC transformer (m) which draws current from a two-phase 60 A mains supply. The transformer delivers a maximum current of 2 000 A at 5 V. As a result of these large currents involved a twin variac (n) is used to control the current which flows to the graphite resistance heater in (o). The current is measured with an induction ammeter (p) and the voltage across the transformer terminals is measured using a digital voltmeter (q). The cold junction of the thermocouple is put in an ice bath (r) and the voltage across these points (s) is used for temperature measurement.

3.3 The piston assembly

Figure 3.8 shows a cross-sectional diagram of the piston assembly. The piston is a stepped cylinder made of T15 high speed tool steel with a diameter of 25.28 ± 0.005 mm and a length of 57 ± 0.1 mm and hardened to RC 68 although it is still brittle. The maximum pressure attainable is limited by the strength of the piston. It is important that the top and bottom faces of the piston have a good quality ground finish to ensure a reasonable quality of the ultrasonic pulses. The retaining ring (t) is pressed over the piston before being press-fitted into the brass cooling ring (u). The transducers used are 10 MHz coaxial quartz transducers. The transducer (v) is cemented to the base of the piston by means of a conductive silver epoxy.

A small light coil spring (w) is cemented centrally on the transducer to form an electrical contact with the copper contact plate (x) on the piston support (y). This copper plate makes contact with a lead (z) connecting the transducer to the MATEC system.

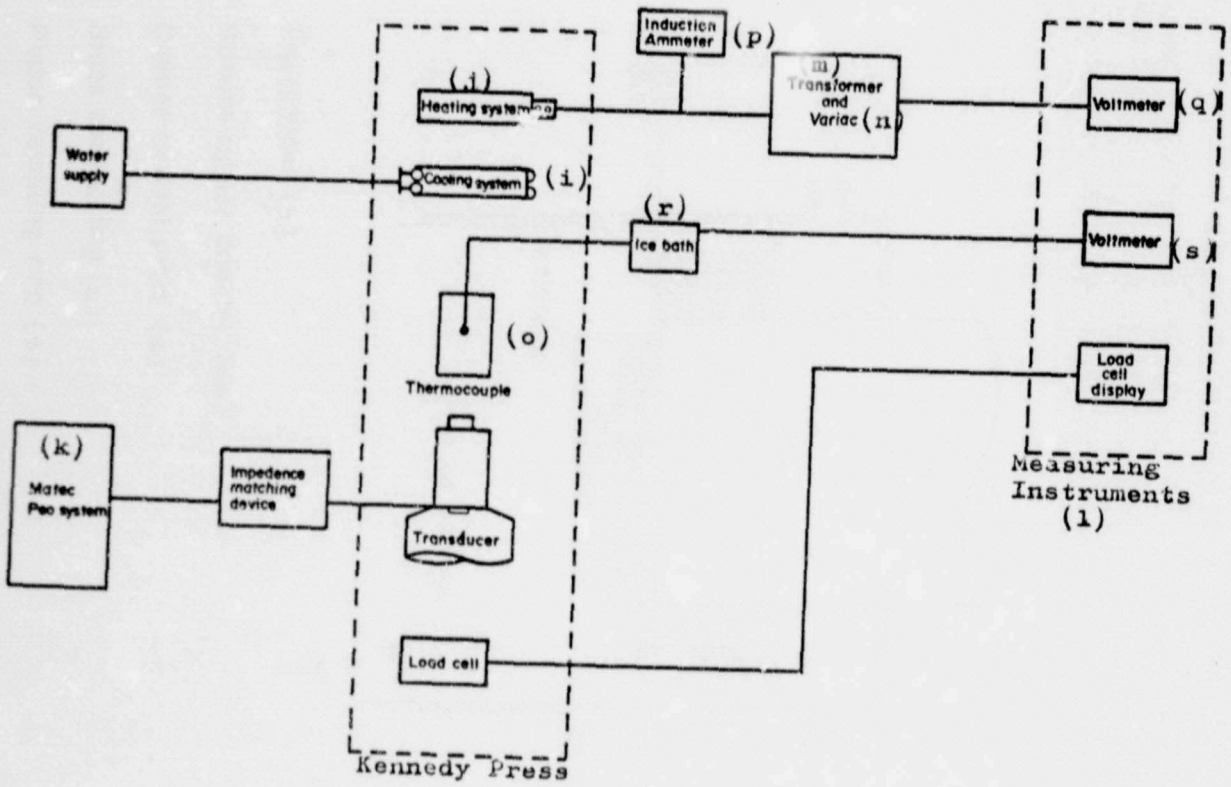
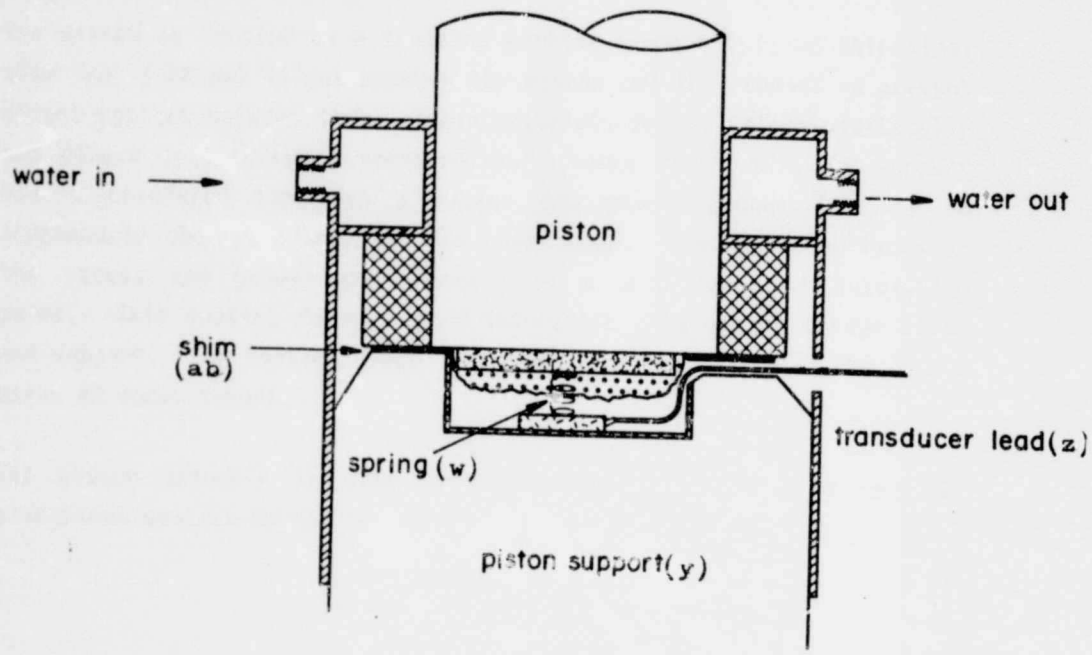


Fig. 3.7 Block diagram of the auxiliary equipment



Transducer (v)

Silicone rubber damper (aa)

Copper contact plate (x)

Brass cooling ring (u)

Piston retaining ring (t)

Fig. 3.8 Cross-sectional diagram of the piston assembly

To prevent "ringing" of the transducer, it is usually "backed" on the rear face by a solid material with, ideally the same impedance (C ecraft, op. cit.). Vibrations at the rear face are then ideally, coupled completely into the backing. In this study a 1 mm thick layer of silicone rubber (aa) was used which does not have the same impedance exactly, but nonetheless reduces the "ringing" by damping the transducer.

The piston is located on a tungsten carbide support (y). A thin steel shim (ab) (~50 μm) is put between the piston and the support to prevent direct contact between them. These materials do not deform plastically and direct contact under pressure would cause fracture. The support has a chamfered top with a recess and groove ground into it to accommodate the transducer and its power lead. The inside surfaces of the recess and groove are coated with a thin layer of insulating paint. This coating forms a good insulation between the copper plate and support. The contact plate is cemented in the centre of the recess using an epoxy cement.

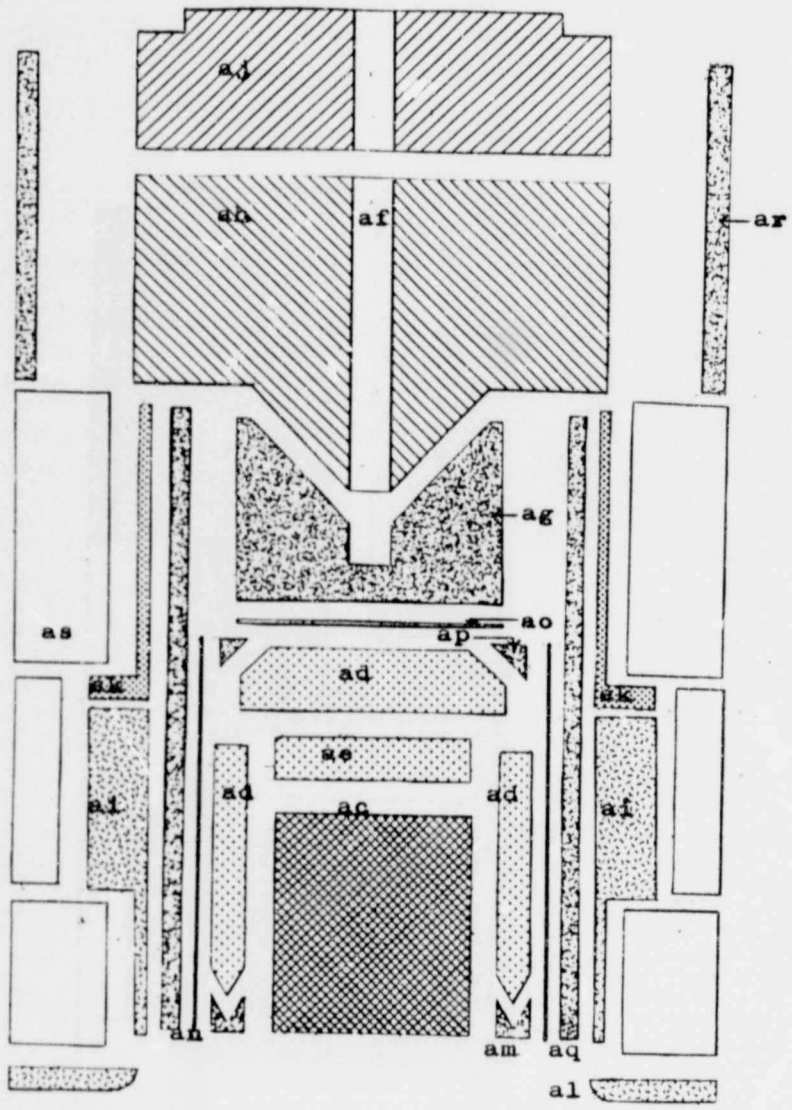
The piston assembly is slid in place over the support to initiate electrical contact to the transducer.

3.4 The high pressure and temperature ultrasonic capsule

The ultrasonic high temperature and pressure capsule designed in this laboratory generates a hydrostatic pressure on the sample, contains a heating element for high temperature work, and allows the passage of ultrasonic pulses to the sample and back.

Figure 3.9(a) shows schematically an exploded diagram of the cross-section of the high pressure and temperature ultrasonic capsule and figure 3.9(b) is the assembly diagram. The capsule is cylindrical in shape, 48 mm long and 25.4 mm in diameter. The recess at the base of the capsule allows the stepped piston to sit directly against the sample (ac), and ensures the transmission of ultrasonic waves to the sample from the transducer located at the bottom of the piston.

The lead (ad) around the sample ensures a good approximation of hydrostatic stress in the region of the sample. The lead disk (ae) at the back of the sample acts as a reflector to enhance reflections from that end of the sample. The stainless steel tube (af) holds the chromel-alumel thermocouple wires. The wires are fed through an alumina insulation tube with two holes. The wires are spot-welded together to form a small thermocouple bead at the one end of the alumina tube. A small droplet of alumina cement is placed over the bead to protect it. The stainless steel tube is also welded shut at the bottom end. The alumina tube is then inserted into the stainless steel one. The ends of the wires that protrude from the tube are painted with an insulating paint and covered with a heat shrink insulation. The thermocouple bead is eventually located in the recess in the particular pyrophyllite part (ag). The purpose of the conical interface between this pyrophyllite part and the silver steel part (ah) is to clamp the tube in place at high pressures. Current to the heater (ai), in the region of the sample, passes into the capsule via the top steel parts (ah, aj) and the brass conductor (ak) and generates heat in the graphite resistance heater (ai). The heater is grounded via the piston.



Hardened silver steel
Silver steel
Salt
Pyrophyllite
Graphite
Lead
Tantalum
Sample
Brass

Fig. 3.9(a) Exploded diagram of the cross-section of the high pressure and temperature ultrasonic capsule

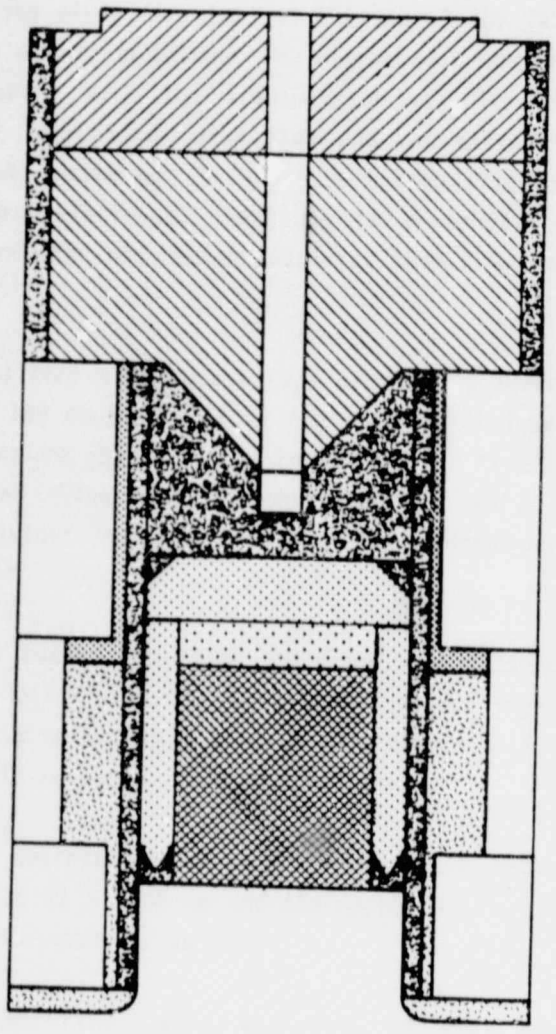


Fig. 3.9(b) Assembly diagram of capsule

The graphite bush (al) at the bottom spreads the current from the heater over a larger area so that the heat would not be concentrated on one spot of the piston.

The pyrophyllite mitre ring (am) at the end of the lead sleeve prevents the lead from extruding between the piston face and the sample and thus from interfering with the transmission of ultrasonic waves. The tantalum foil (an), steel disk (ao) and top mitre ring (ap) prevents the lead from extruding through the surrounding pyrophyllite parts which can cause electrical shorts. The pyrophyllite tube (aq) just inside the conductor and heater insulates them from the piston step and lead sleeve.

The hardened silver steel piece (aj) provides a strong capping to the capsule. The lug on top of it is for locating an insulating shim which is used to prevent current flow to the heater from grounding via the pressure plate. The thin pyrophyllite ring (ar) around the steel components provides insulation between these components and the walls of the cylinder.

The three salt bushes (as) have low shear strengths which is important for converting uniaxial force from the piston driving into the cylinder into a hydrostatic stress acting on the sample. They are formed by compressing salt in a specially designed die.

The sample is cylindrical with varying diameter between 6.5 and 10.0 mm, length of 10.00 ± 0.05 mm and the opposite faces polished flat and parallel to 1.8 minutes of an arc.

All the pyrophyllite parts are made of pink pyrophyllite, i.e. pyrophyllite that has been baked in an oven, following a fixed procedure, to get rid of chemical water. The different parts are made to a slide fit, and have to be put together carefully. The capsule is then wrapped in lead foil along its length, the purpose of which is to reduce friction inside the cylinder. Figure 3.10 shows some of the components, and an assembled capsule (at) on the pressure plate (au). The piston with retaining ring and transducer stuck to the piston's base (av) is also shown.

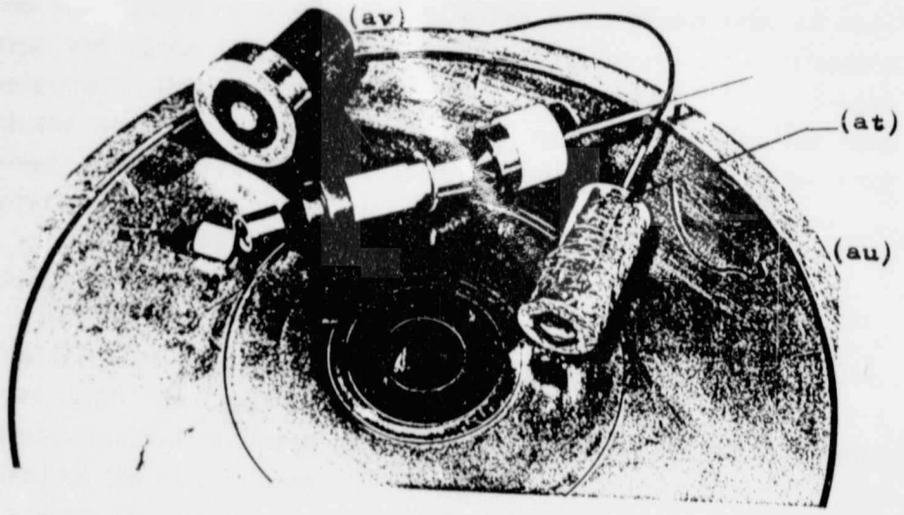


Fig. 3.10: An assembled capsule and components on the pressure plate

3.3 Experimental procedure

The data obtained from this experiment are:

3.5.1 Pressure

Pressure is measured by the load cell which has been calibrated so that the true pressure exerted on the sample can be calculated. An oil pressure gauge is also used which gives an indication of the pressure.

3.5.2 Temperature

The thermal variation between the ice point and the chromel-alumel thermocouple produces a voltage of a few mV. Measurement of this voltage and using tables to convert this value yields a reference temperature. This reference temperature is measured in the region above the sample with the thermocouple. An equation which has been obtained from a reference run is then used to calculate the true temperature inside the sample.

3.5.3 Frequency

The velocity of the ultrasonic wave travelling through the sample, changes with the application of pressure and temperature. This manifests itself as a change in the overlap frequency of the two echoes examined on the MATEC system. By retuning the frequency to overlap the echoes again the variation of velocity can be recorded as a function of temperature and pressure. After particular corrections have been applied to this data, the appropriate elastic modulus, longitudinal or shear, is calculated and considered as a function of pressure or temperature.

Reference equations and corrections to be applied will be discussed in Chapter 4.

It is good practice to first apply some pressure on the capsule before starting to raise the temperature. Since the melting point of

lead increases with increasing pressure, this precaution is taken to prevent lead from extruding and causing electrical shorts. In order to compact the capsule the temperature was initially raised and stabilized at 100 °C before pressurizing the capsule to maximum pressure (3 GPa). The other isotherms followed and ranged between room temperature and 250 °C for pressures varying between 0.5 GPa and 3 GPa. Pressure increments of 0.1 GPa were spaced one or two minutes apart so that the pressure within the capsule could equilibrate.

As the temperature rises it becomes more difficult to operate the experiment between 0 GPa and 1 GPa, due to the possibility of electrical shorts occurring. It is preferable to determine isotherms rather than isobars, since the latter are very time-consuming. After every current setting a few minutes must be allowed for the temperature to stabilize at the sample.

This experiment is very delicate and complex, involving numerous precision operations and component assemblies. Many runs failed as a result of lead extrusion, electrical shorts, transducer cracking, electrical contact failure, water leakage, loss of signal due to attenuation, acoustic coupling and sample cracking.

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