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**CHAPTER 9**

**INTRODUCTION TO HIGH PRESSURE RESEARCH**

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## 9.1. INTRODUCTION

High pressure research has influentially developed into an interdisciplinary area of science and has led to many insight into the behaviour of matter. The subject acquired a status as an important tool for understanding material properties and phenomena. In the past three decades, there has been considerable interest in the study of the structural phase transition and high pressure behaviour of solids because of academic, technological, astrophysical and geophysical reasons [1-19].

The academic interest owes to the diverse physical properties exhibited by matter under high pressure. The technological interests in high pressure studies are mainly concerned with the applications of the properties of the new phases of matter. In geophysics, this helps towards the understanding of inner evaluation of stellar bodies like white dwarf, neutron stars and black holes. Exceedingly high pressures are their in nature, about 3 million atmospheres at the centre of the earth and 100 million atmospheres at the centre of sun, produced due to forces of gravitational pull.

Irrespective of the definite area of interest, the primary requirements of any investigator in this field are a

suitable means of generating the pressure and a suitable arrangement to contain the generated pressure for its successful utilization . It is therefore essential to understand the status of current experimental methods, their usefulness and technical limitations.

High pressure generation techniques can be widely divided into two major categories, viz static and dynamic. Static high pressure devices consists of a sturdy frame, a device that generates force and container walls for squeezing the enclosed sample. The second technique, encompasses all shock dynamic methods which can produce transient pressure to about five million atmospheres.

In general, the static method includes the piston cylinder (PC), the tungsten carbide (WC) opposed anvil apparatus, diamond anvil cell (DAC) etc. Amongst the dynamic methods, the shock wave methods are most commonly used in the laboratory for the study of the high pressure behaviour in solids.

Before going into the details of the above methods of high pressure generation, it is important to mention some of the units of measurement commonly used in high pressure studies.

## 9.2. UNITS OF PRESSURE

Some of the units commonly used in high pressure studies are, 1 bar =  $10^6$  dynes/cm<sup>2</sup> = 0.9869 atoms.  
= 1.0197 Kg/cm<sup>2</sup>  
= 14.54 psi.

The S.I. unit of pressure is the Pascal (Pa). The Pascal by definition is 1 N/m<sup>2</sup>, and since it is too small a unit, in high pressure work the MPa ( $10^6$  Pa) and the GPa ( $10^9$  Pa) are used. However, the most commonly employed unit in the high pressure literature is the Kiloobar.

## 9.3. GENERATION OF HIGH PRESSURE

The various methods of generating high pressure falling under the above two broad categories, static and dynamic are described in brief.

### 9.3.1. Static Methods Of Pressure Generation

The static methods allow the measurements to be carried out under purely hydrostatic condition.

#### 9.3.1.1. Piston-cylinder method

piston cylinder (PC) apparatus is a versatile high

pressure device used since the earliest compressibility measurements carried out by Bridgman [1,20]. The force or load from a hydraulic press, applied over a large area ram, is transferred to the piston of a small area. The oil pressure in the hydraulic press, multiplied by the area multiplication factor, gives the high pressure after allowing for small corrections due to friction etc.

A piston cylinder apparatus based on the design of Kennedy et al. [21] is shown in Fig. 9.1. The apparatus utilizes two hydraulic rams. The ram at the top  $R_1$  carries the platen  $P_1$ . The pressure vessel is clamped between the platens  $P_1$  and  $P_2$  by operating the ram  $R_1$  such that the tungsten carbide core supports the entire load. This is called end-load. The sample placed in the pressure vessel is pressurised by forcing a tungsten carbide piston into the pressure vessel; the ram  $R_2$  provides the necessary load.

#### **9.3.1.2. Belt and Girdle apparatus**

The belt [22,23] and the Girdle [24,25] apparatus are shown in Figs. 9.2 and 9.3 respectively. In this family of devices the piston (or dies) at the two ends are tapered. As a result of this, the carbide core supports a considerable amount of compressive load, which serves the same purpose as

the end-load in a piston-cylinder apparatus. These devices can be used to generate pressures upto 15 GPa and temperatures upto 3000°C. Because of a comparatively trouble free performance, large sample volume and very high temperature capability, these devices have been extensively used for synthesis of materials such as diamond and cubic carbon nitride.

#### 9.3.1.3. Bridgman opposed anvil apparatus

The principle on which this high pressure instrument is based is shown in Fig. 9.4. A sharp tip made of a high strength material is pressed against an equally hard surface with the sample in between. The stress of the order of  $F/A$ , where  $F$  is the force with which tip is pressed and  $A$  is the area on which the force  $F$  is supported, is generated. The highest stress that can be reached is limited by the yield or the fracture strength of the tip material depending on whether the tip deforms plastically first or fails by fracture. A small area of the tip is desirable to obtain high stresses, whereas the ease of sample loading and introducing the necessary probes in the high pressure region requires a reasonably large area. These considerations have led to the development of device called opposed anvil apparatus. Figure

#### 9.3.1.4. Diamond anvil apparatus

The diamond anvil apparatus, though based on the same principle as that of Bridgman anvils, deserves special mention because it can go to the highest static pressure.

A simple form of a diamond anvil cell is shown in Fig. 9.6. The diamond pieces, with anvil faces typically 0.3 mm in diameter are placed in hardened steel, boron carbide or tungsten carbide seats with a rocker arrangement which facilitates the alignment of the anvil faces to be parallel to each other. The force on the anvil is given through the lever and spring arrangement.

The introduction of a metal gasket which upon extrusion provides support to the tapered surfaces of the anvil piston, raises the pressure capability from 30 to over 100 GPa. The gasketing techniques also permit experimental studies under a hydrostatic pressure environment upto 10 GPa.

The diamond anvil cell is being routinely used for pressures in the 150-500 Kbar region. The diamond pieces have to be flawless, carefully lapped for parallelism of the end faces and aligned with the greatest care for these high pressure studies. Since the whole arrangement is so compact



that it can go into IR, visible or UV spectrophotometers or into microscope stages [26]. A slight variation in the arrangement is used for X-ray structural investigations. The sample is mixed with NaCl and X-ray pictures are taken. Sodium chloride (NaCl) acts as a pressure transmitting medium and also provides the pressure calibration, since the lattice spacings of NaCl are known as a function of pressure.

The diamond anvil system has been used for other measurements also. By measuring the dimensions of the sample under an optical microscope, pressure-volume data have been obtained. Using foil electrodes, electrical resistivity have been measured. Raman effect and Brillouin scattering studies have been reported on a number of materials.

#### **9.3.1.5. Multi anvil apparatus**

A totally different approach for generating high pressure is illustrated in Fig. 9.7. The sample is placed inside a gasket (made of pyrophyllite or MgO epoxy) which is in the form of a polyhedron. The loads normal to the surfaces of the polyhedron are applied using anvils, the number of anvils equals the number of the plane faces of the polyhedral gasket. This class of instruments are often called multi anvil apparatus. The first multi-anvil device was built with a

tetrahedral gasket configuration [27]. The device is capable of going upto 10 GPa. The movement of the anvils have to be synchronised and directed normal to the tetrahedron faces. This is achieved with the help of the various mechanical linkages or the guides. The cubic presses [28] have also been extensively used to generate high pressures. The pyrophyllite gasket is in the form of a cube. The hexahedral geometry shown in Fig. 9.7 is very useful in X-ray diffraction work [29]. The gasket in this case is in the form of two tetrahedrons with a common face. This geometry permits an unobstructed recording of the diffracted beam.

The devices described so far made use of hydraulic rams to drive the anvils. In other category of the multi-anvil devices, the anvils and the supports can be thought of as forming a sphere. A high pressure at the centre of the sphere can be generated by subjecting the outside of the sphere to hydrostatic pressures [30,31].

### **9.3.2. Dynamics Methods Of Pressure Generation**

These are by (1) shock wave techniques and (2) nuclear detonation. In all these cases, the pressures are generated for a fraction of a second but the pressures are in

the range of Megabar.

#### **9.3.2.1. Shock wave techniques**

The pressure of 500 GPa magnitude can be produced if a solid accelerated by the detonation of some explosive is made to impinge on the sample. This method produces dynamic pressures for a time duration of the order of a few microseconds. The small time duration poses severe experimental problems requiring sophisticated instrumentation. There are serious limitations on the type of information that can be obtained by the technique. So far, attempts have been made mostly to obtain the equation of state of solids from the experimental shock wave data. For these reasons, the shock wave technique do not enjoy the popularity over the other high pressure techniques, eventhough shock wave technique can give extremely high pressures. This technique has therefore not been detailed over here. One can see the details in reference [32].

#### **9.3.2.2. Nuclear detonation technique**

Nuclear explosive experiments are capable of generating the highest pressures of the order of 100 Mbar like the other transient pressure generators. This technique

destroys the specimen in the process of the experiment. However, because of the difficulties in readily having nuclear explosives only a few experiments have been reported so far.

In conclusion, **Table 9.1** summarises the maximum pressure produced by different types of techniques of the pressure generation.

#### **9.4. MEASUREMENT OF PRESSURE**

The promising methods of measuring pressure so far available are

##### **9.4.1. The Mercury Column**

For direct pressure measurements the mercury manometer is limited to about 30 meters in length, which corresponds to nearly 40 bar. It may, however be used as a differential gauge upto pressure of 3 Kbar, giving the most accurate calibration of pressure in that range. Accuracies of nearly 1 in  $10^5$  or better can be achieved at low pressure (< 40 bar). But the useful pressure range covered by this gauge is too low to be of much interest.

##### **9.4.2. The Pressure Balance**

The pressure balance is also known as the free-

piston gauge. A typical free-piston gauge is shown in Fig. 9.8. A free-piston closely fitted in a bore in the pressure vessel is retained in position by weights placed on it. The absence of sealing ring greatly reduces the friction between the free-piston and the bore. A further improvement is brought about by either rotating or oscillating the free piston. The design of the bore allows a component of pressure  $p$  to act on the region marked C such that the clearance between the free-piston and the bore is controlled at various pressures. The pressure medium however leaks past the piston slowly because of the absence of any seal. This does not pose any serious problems if the leakage of the pressure medium is slow. However at high pressure to compensate for the leakage past the free piston, a continuous supply of pressure medium is required. A carefully designed and fabricated free-piston gauge can be used upto nearly 3 GPa.

#### **9.4.3. Simultaneous Measurement Of Compressibility And Relative Volume Change**

The idea of obtaining thermodynamic pressure from compressibility and relative change of volume data was first suggested by Smith et al. [40] as early as 1954. But the

credit of realizing this method in practice goes to Ruoff et al. [41,42]. Independent measurements of compressibility (K) and relative change of volume of a substance under identical conditions of pressure and temperature were used to obtain the pressure through the relation

$$P_1 - P_2 = \int_{P_1}^{P_2} dp = - \int_{V_1}^{V_2} \frac{dV}{kV}$$

#### 9.4.4. The Equation Of State

The equation of state for a suitable solid calculated from first principle will be the ideal thermodynamic basis for absolute pressure measurement. But in practice, for useful substance in high pressure, calculations of equation of state becomes a formidable problem, and vice versa. The best that is available is the Decker NaCl equation of state [43,44]. The results of the calculation are in such good agreement with experimental results over a wide pressure and temperature range that NaCl scale is regarded as thermodynamic pressure scale.

#### 9.4.5. Piston-Cylinder Gauge

The use of a piston-cylinder system with either piston packing or a solid medium pressure environment

represents the best approximation to a primary scale at pressures above 25 Kbar. Several piston-cylinder devices have been used upto 80 Kbar, particularly with solid pressure transmitting media [45,46]. Such an apparatus uses a hydraulic ram to move the piston. By rotating the ram and averaging data from the forward and backward strokes, pressures at 50 Kbar may be estimated to within around 0.5 Kbar. At 80 Kbar uncertainties are probably as high as  $\pm 3$  Kbar. An excellent review of this method is given by Richter and Clark [47].

#### **9.4.6. Galvanic Cell As Primary Pressure Indicator**

The technique offers a very simple and vigorously defined means of determining absolute pressure but at present cannot be put to practical use because of experimental difficulties. Continued research on solid state electrolytes may result in a cell that is feasible for primary pressure measurement.

### **9.5. SECONDARY GAUGES**

The gauges so far considered are primary gauges in the sense that pressure is derived from the measurement of the fundamental quantities. In many other types of high pressure set up such an approach is not feasible. This has led to the

development of secondary pressure gauges. Description of several secondary pressure gauges used in practice is given below.

#### **9.5.1. Measurement Based On Non-Uniform Strain**

For pressure measurement less than 10 Kbar the most widely used pressure gauges depend on effects resulting from non-uniform strain.

The most common gauge of this kind is the Bourdon gauge, which is normally in the form of a hollow tube bent into a spiral. One end is fixed, the other free to move under the deforming action of the pressure. Indication of the pressure is usually made via a mechanical linkage. The Bourdon gauge may use other geometries, such as a flat tube twisted about its length, a spiral, or a straight tube with an eccentric bore.

#### **9.5.2. Electrical Resistance Gauges**

In principle any physical quantity that is a function of pressure can be used as a basis for a secondary gauge. The changes in electrical resistance can be measured with a high degrees of accuracy.



Bridgman identified use of manganin (84 % Cu, 12 % Mn and 4 % Ni) in pressure gauges. The variation of resistance with pressure is linear upto nearly 2GPa with a pressure coefficient of resistance nearly  $0.023 \text{ (GPa)}^{-1}$ . However, the pressure coefficient depends on the exact composition and the manufacturing details of the wire. Many investigations have reported differences of a few percent between two gauge coils prepared from the same spool. For these reasons, it is not possible to have an universal value of the pressure coefficient of resistance. A careful seasoning by pressure cycling and temperature annealing is required before the determination of the pressure coefficient of resistance for each coil. The variation of manganin resistance in solid pressure transmitting medium is much more complex and is not discussed here.

### 9.5.3. Gauges Based On Optical Properties Of Materials

The changes in the optical properties of materials caused by pressure, such as the shift of absorption lines and change in refractive index can be used for pressure measurement. The pressure scale based on the shift of the absorption band of nickel dimethyl glyoxime developed by Lippincott and Dueckar was quite useful in the past.

Recently, ruby-fluorescence technique [48-50] has been extensively used in the measurement of pressure. A sharp line fluorescing material is placed in the high pressure cell along with the sample and fluorescence is excited by a high intensity light source. The shift under pressure in the wavelength of the fluorescent radiation is detected. Once the shift in wavelength is calibrated in terms of pressure, it can be used to estimate pressure by measuring the shift in any experiment.

The fluorescing material could be ruby  $\text{Al}_2\text{O}_3$  (0.5 % Cr),  $\text{YAl}_2\text{O}_3$  (0.2 % Cr), YAG (0.38 %  $\text{Cr}_2\text{O}_3$ ),  $\text{YAl}_2\text{O}_3$  (2.5 % Cr) etc. However, the most commonly used material is ruby. The early experiments showed that the shift under pressure of the R-lines (doublet : 6923 and 6942<sup>0</sup>A) was 3.6<sup>0</sup>A/GPa and the shift in wavelength versus pressure relation was linear over a very wide range of pressure (upto nearly 20 GPa).

#### 9.5.4. Phase Transition And Fixed Points

Many types of phase transitions may be used as reference points for high pressure measurement and many different methods may be used for detection (e.g. based on

changes in volume, resistivity, optical or acoustical constants or on latent heat). Following the symposium on accurate characterisation of the High Pressure Environment in 1968 the transition pressure values listed in Table 9.2 are proposed as fixed points. The situation is comfortable for pressures upto 100 Kbar.

For work above 100 Kbar, many fixed points have been tentatively proposed. The review by Le Neindre et al. [51] discusses the status of the fixed points above 100 Kbar.

#### 9.6. CONCLUSIONS

The methods of generation and techniques for measurement of high pressure discussed briefly in this chapter will be used to study the transport properties behaviour of CVT grown  $\text{CuInS}_2$  single crystals. The results of this measurements will be discussed in detail and will be the subject matter of the next chapter.

TABLE 9.1 Highest Pressures Achieved Through Various Experimental Techniques

Pressure (Region)	Method (type)	Technique	Maximum pressure produced (GPa)	Typical compression achieved ( $V/V_0$ )	References
Low pressure (0-4) GPa	Static	Piston Cylinder (PC)	4.5	0.95	[ 33 ]
High pressure (4-100) GPa	Static	Tungsten Carbide Anvil cell	100	0.7	
	Static	Diamond Anvil Cell (DAC)			[ 34 ]
Ultra High Pressure > 100 GPa	Dynamic	Magnetic Compression	500	0.25	[ 35 ]
	Dynamic	Gas Gun	500	0.5	[ 36 ]
	Dynamic	Shock wave Chemical explosive	1000	0.5	
	Dynamic	Laser	3500	0.3	[ 37 ]
	Dynamic	Under Ground Nuclear Explosive	7000	0.3	[ 38 ]
	Dynamic	Electric Gun	1000-5000	-	[ 39 ]
	Dynamic	Rail Gun	1000-10,000	-	

**TABLE 9.2** Fixed Point In The Pressure Range  
(10-300) Kbar at 25°C.

## REFERENCE POINTS

Material and transition	Fixed point pressure Kbar
Hg freezing point at 22°C	11.8
Bi I-II at 25° C	25.5
Tl I-II at 25° C	36.7
Cs II-III, IV	42
Ba I-II at 25° C	55
Bi III-V at 25° C	77
Sn I-II	94
Fe	110
Ba II-liq.	120
Pb I-II	130
Rb II-liq.	146
Fe <sub>15</sub> Co	150
Fe <sub>20</sub> Co	190
Fe <sub>40</sub> Co	290

**CAPTIONS TO THE FIGURES**

- Fig. 9.1.        Piston-cylinder apparatus
- Fig. 9.2.        The Belt apparatus
- Fig. 9.3.        The Girdle apparatus
- Fig. 9.4.        Principle of an anvil set up
- Fig. 9.5.        Principle of the Bridgman anvil apparatus
- Fig. 9.6.        Sketch of a diamond anvil cell
- Fig. 9.7.        Geometry showing tetrahedron, cube and Hexagonal presses for generating high pressure.
- Fig. 9.8.        The Pressure Balance : A typical free piston gauge

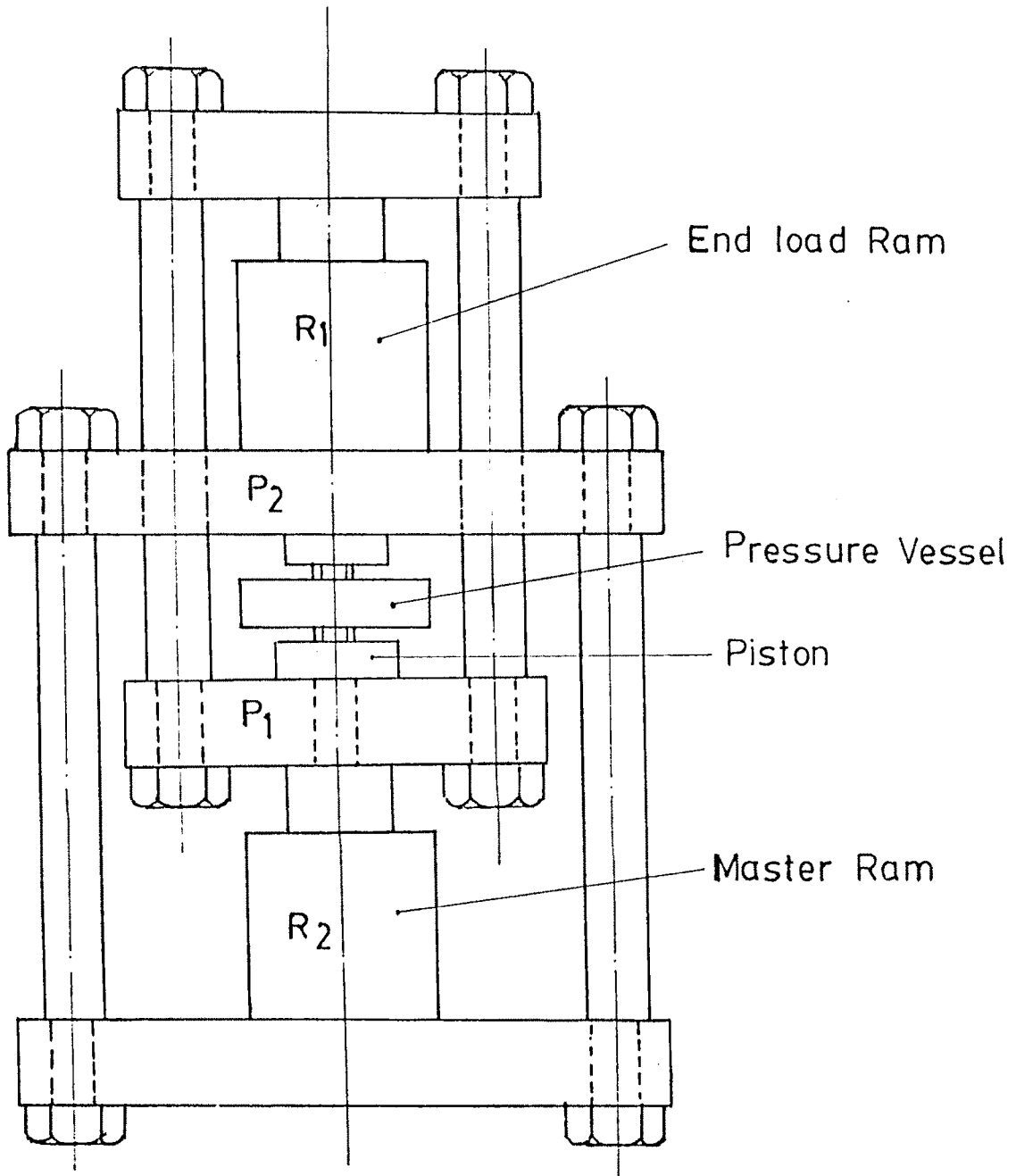
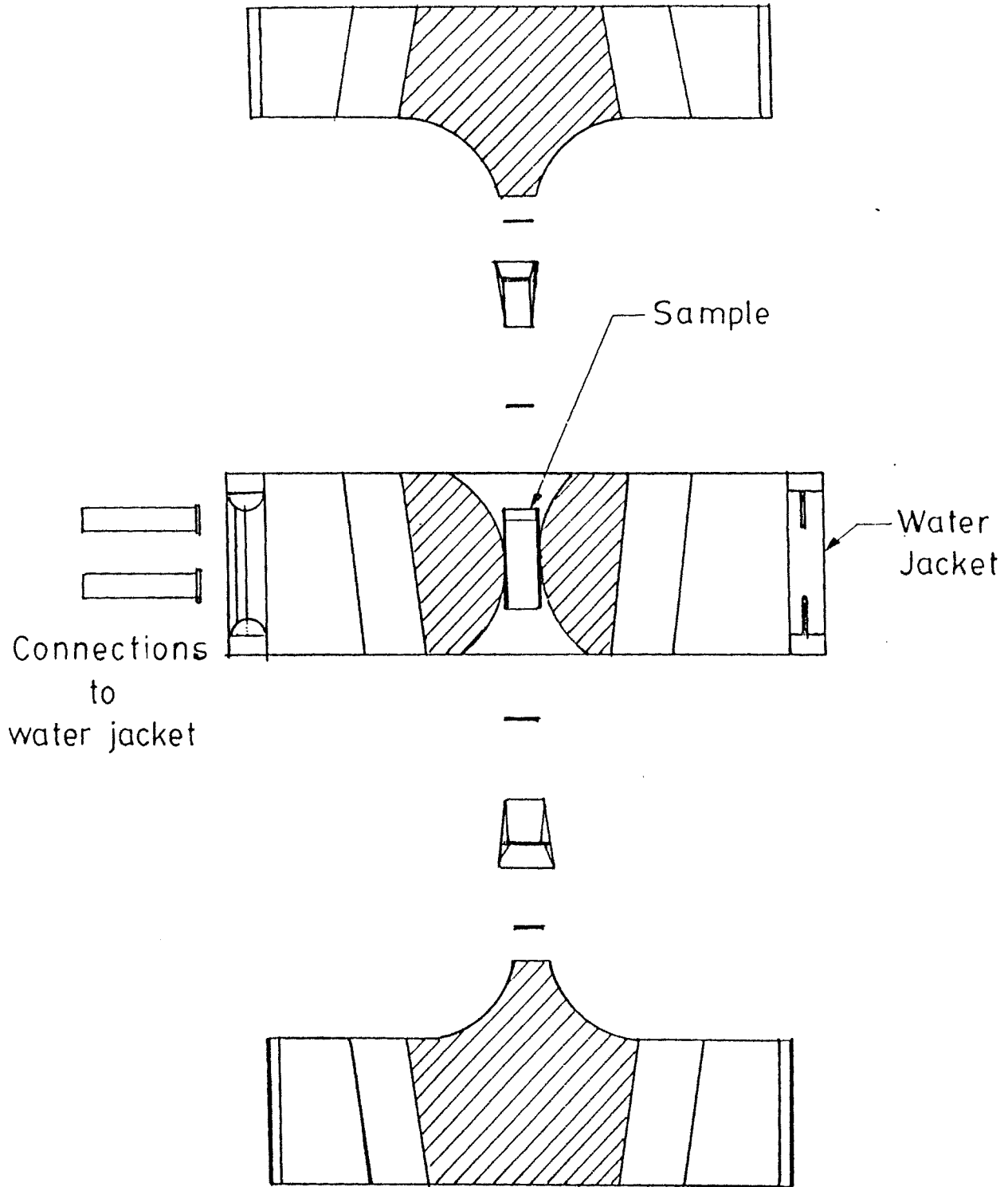


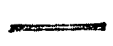
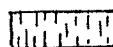

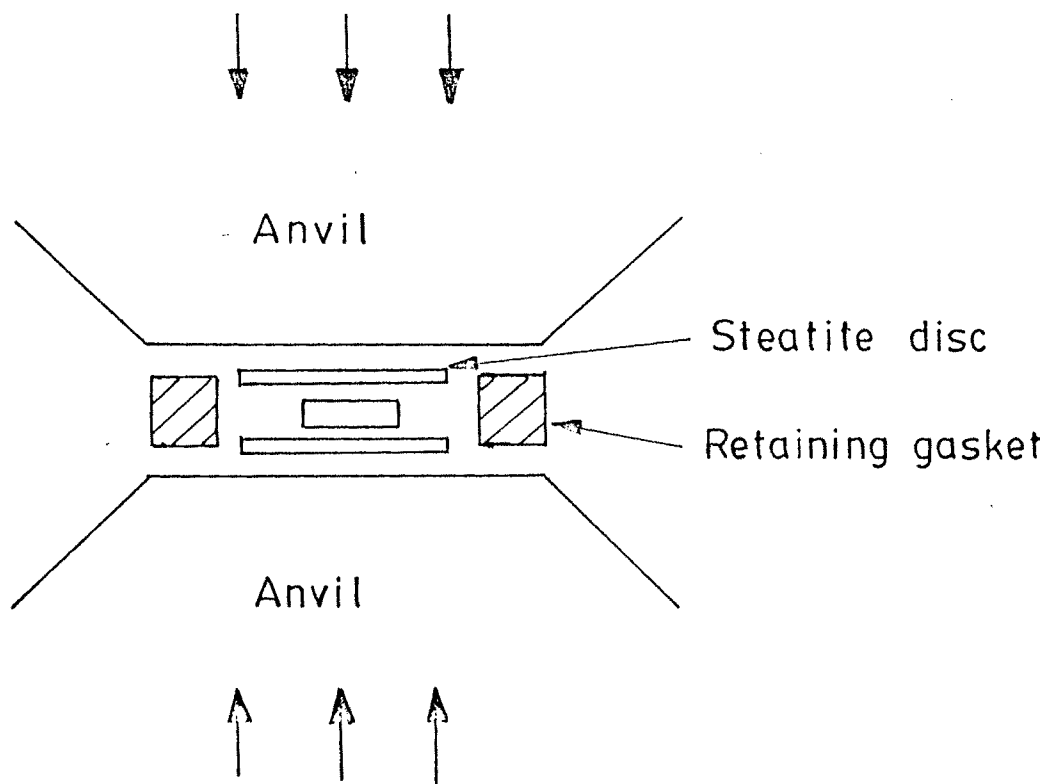


fig. 9.1



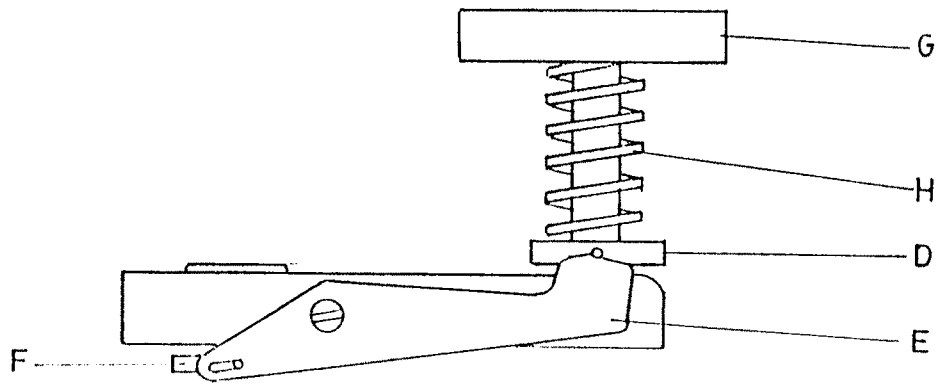
-  - Hard steel plug
-  - Tungsten carbide
-  - Carbon
-  - Pyrophyllite
-  - Steel binding rings



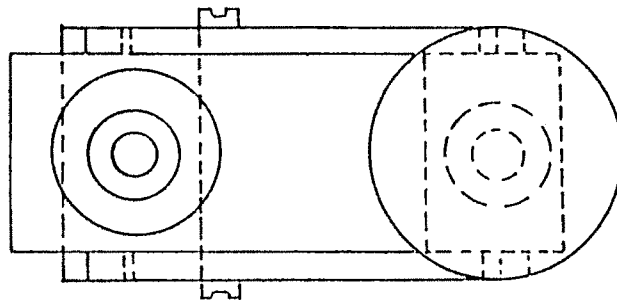


Principle of the Bridgman anvil apparatus

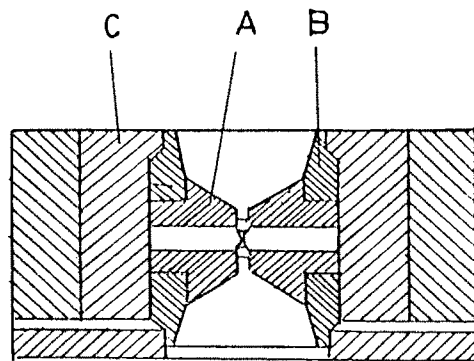
fig. 9-5



Side view



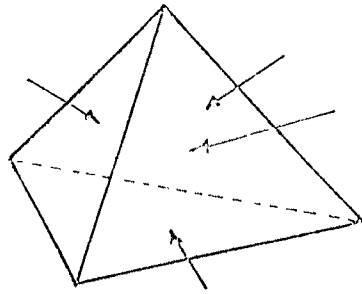
front view



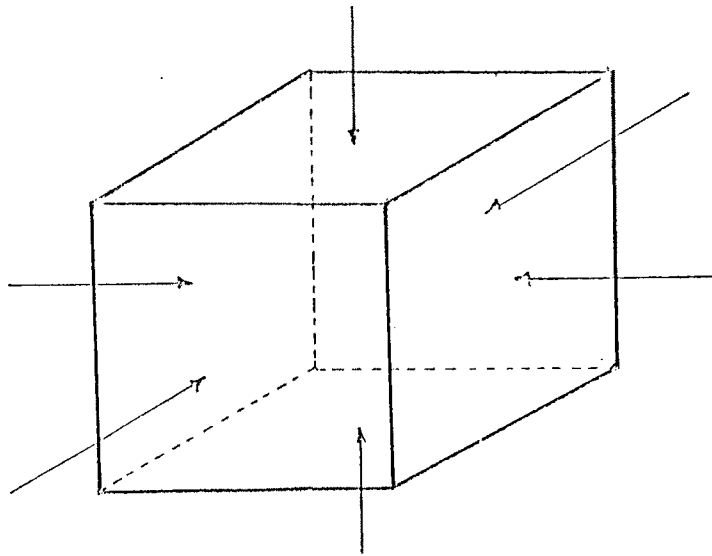
Detail of diamond cell

Sketch of a diamond anvil cell. A: Support to diamond pieces. B: Mounting of the support. C: Hardened alloy insert. D: Pressing plate. E: Lever. F: Pressing plate on the anvil side. G: Screw head. H: Calibrated spring.

fig. 9.6



Cube



Hexahedron

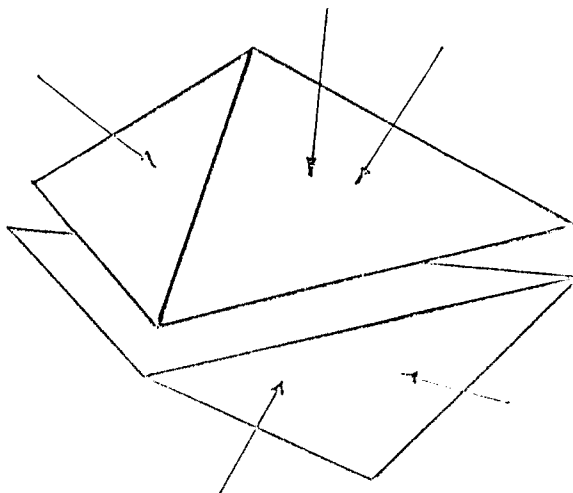


fig. 9·7

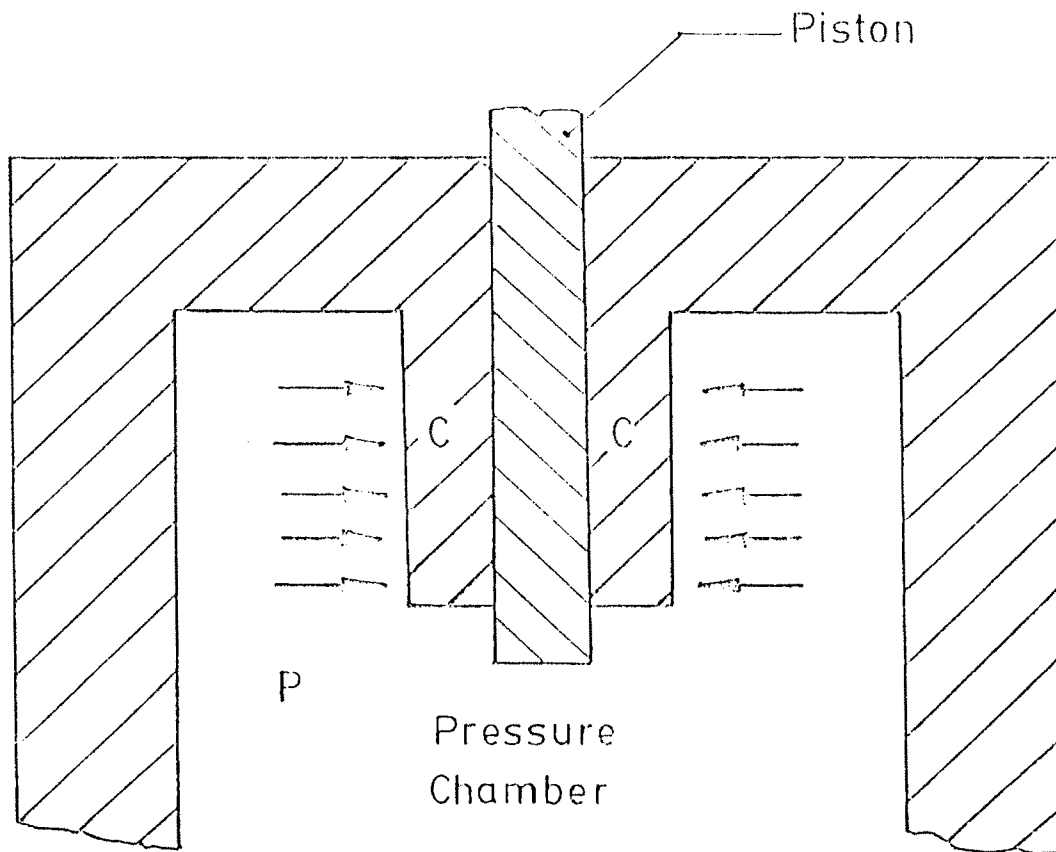


fig. 9-8

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