
CHAPTER 9

THE TECHNIQUES OF HIGH PRESSURE RESEARCH

9.1 INTRODUCTION

In recent years there has been considerable interest in the study of the high pressure behaviour of solids because of academic, technological and geophysical reasons [1-9]. The academic interest owes its origin to the fact that at high pressures the nature of the interatomic forces in matter change considerably. These changes in solids can be studied in greater detail to reveal several new features of the interatomic forces which are responsible for their diverse physical properties. In addition the high pressure studies are also of great importance to visualise

the mechanism governing the structural changes and to reveal several solid state properties associated with different structures.

The technological interest lies in the synthesis of superhard materials like diamond, cubic boron nitride and their polycrystalline sintered compacts. All these materials are strategic to a country's economic and industrial development.

Considerable interest has also been shown by the geophysicists in high pressure studies to explore the inner mantle of the earth where most of the matter is expected to be confined under very high pressures and temperatures. Under normal conditions, solids exhibit a particular crystal structure for which the total energy is minimum. However, on application of high pressure the atomic arrangement in solid changes resulting into change in interatomic distance and crystal structure. The pressure at which such structural change occurs is called phase transition pressure. Under the high pressure condition, the atoms rearrange themselves and assume a new stable structure for which again the total energy of the solid is a minimum. Such a structural phase transition can be noticed experimentally by observing the X-ray diffraction pattern,

electrical resistivity and optical properties at different pressures. In the present thesis measurements of resistivity and thermoelectric power have been used to study the structural phase transitions in SnSe and WS_2 single crystals.

A brief review of the various methods for the generation and measurement of high pressures upto 100 Kbar has been given below :

9.2 GENERATION OF HIGH PRESSURE

The various methods of generating high pressures can be classified in four groups on the basis of the underlying design principles.

9.2.1 Method - 1

This method of producing high pressure is schematically shown in Fig. 9.1. The sample is surrounded by a pressure transmitting medium contained in a vessel. A piston is forced in the vessel with a force F . The leakage of the pressure transmitting medium past the piston is prevented by the use of a suitable seal. The highest pressure which can be attained is limited by the failure of the pressure vessel or the seal.

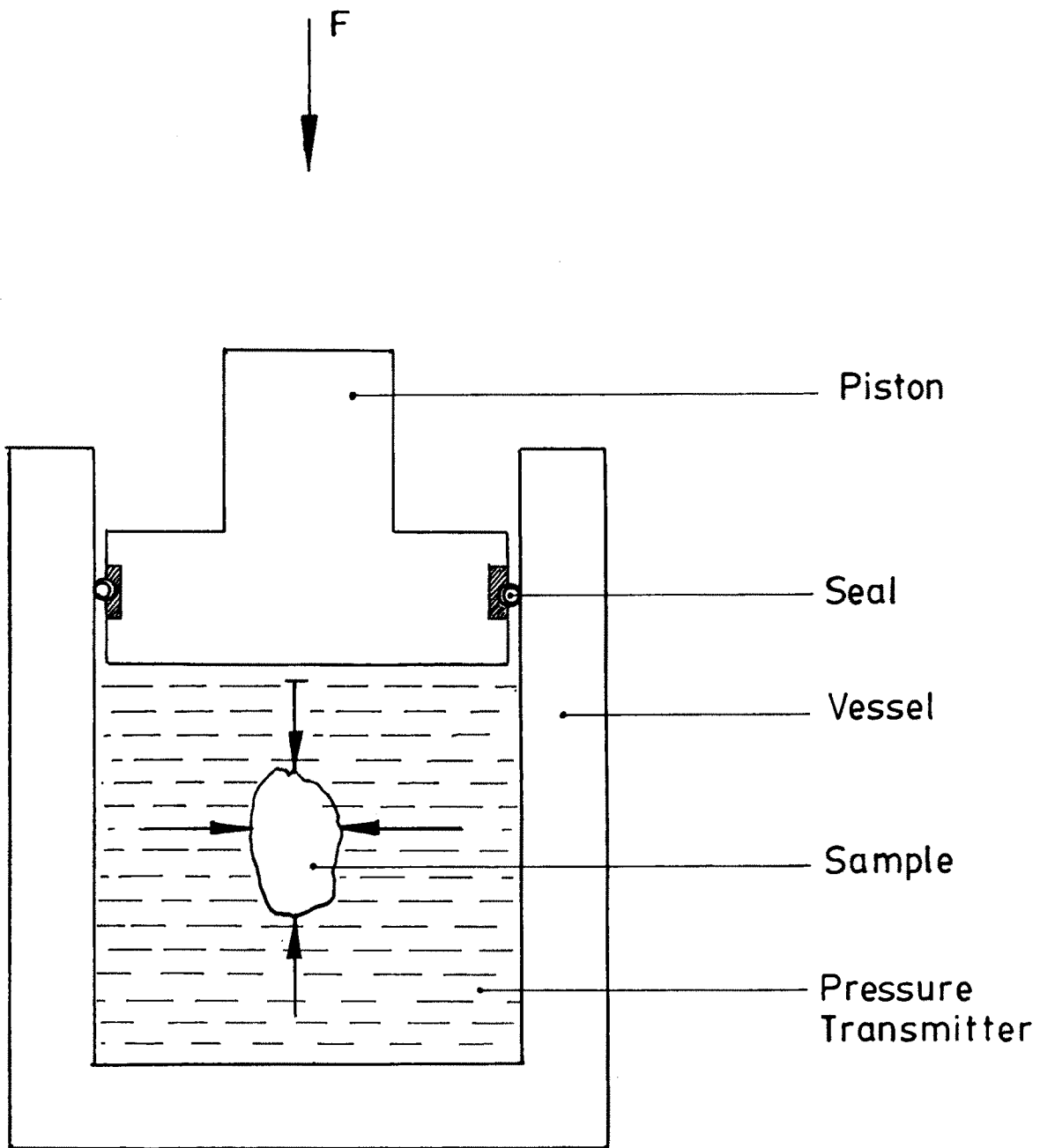


fig. 9-1

Pressure Transmitter

A pressure transmitting medium should be a substance which cannot sustain shear stress. A fluid is therefore an obvious choice as a pressure transmitter. In a variety of high pressure experiments conducted specially at high temperatures, use of fluid as pressure transmitting medium is not possible. In many experiments conducted even at room temperature, use of fluid pressure transmitter becomes difficult because of sealing problems at high pressures. In such cases, solids with low shear strength are used as pressure transmitting medium. The commonly used solid pressure transmitting media are, talc (steatite) boron nitride, silver chloride, lead, indium and epoxy. In general any material which has low shear strength and does not undergo a phase transition can be used as a pressure transmitter.

Seals

The type of high pressure seals used in an experiment depends on the highest working pressure. Some of the commonly used seals are shown in Fig. 9.2. The simplest is a O-ring with back-up rings shown in Fig. 9.2(a). These are normally used in hydraulic rams and can withstand a maximum pressure slightly over 0.1 GPa.

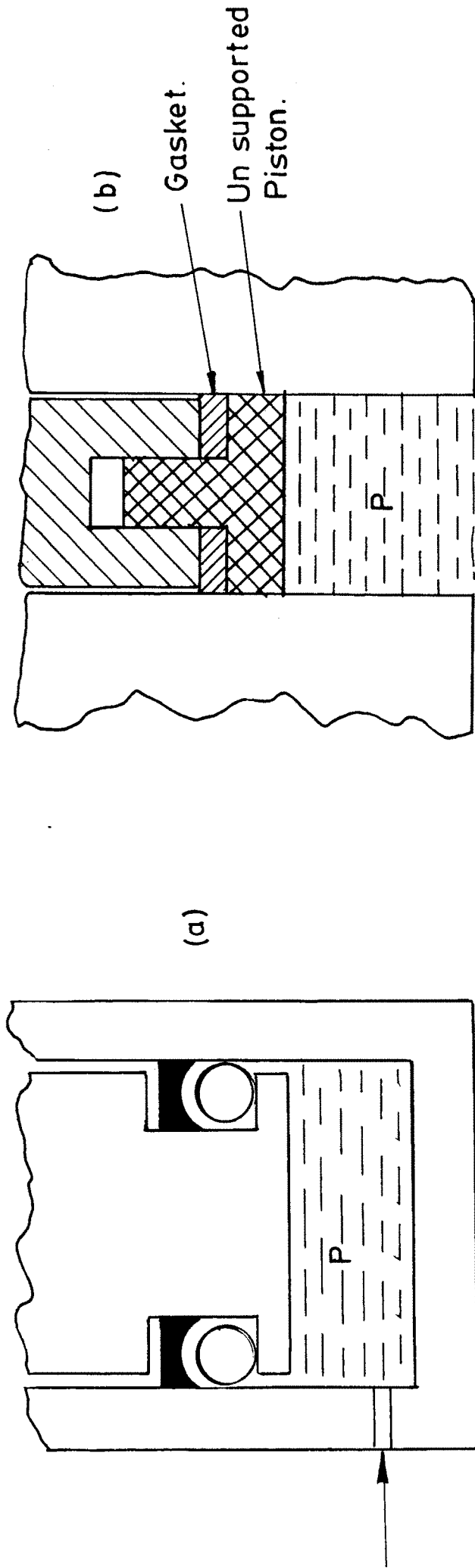


fig. 9.2

The principle of the "unsupported area seal" is shown in Fig. 9.2(b). The compressible gasket area is nearly 10-15 % smaller than the area of the face; consequently, the pressure on the gasket is higher than the pressure in the vessel. This excess pressure prevents the leakage of the fluid past the gasket. The upper pressure limit for the seal is nearly 3.5 GPa.

The teflon-cell technique [10] is often used to seal fluid at high pressures up to 4.0 GPa.

The solid pressure transmitters are easier to contain at high pressures than the fluid pressure transmitters. The solid pressure transmitters such as silver chloride and indium can be successfully sealed with the help of pyrophyllite mitre rings Fig. 9.3. For less efficient pressure transmitters, such as talc, boron nitride, steatite etc. steel mitre rings can be used.

In the discussion so far, an attempt has been made to point out the problems encountered if one tries to extend the upper limit of pressure attainable in a simple concept shown in Fig. 9.1. A complete working apparatus will require not only the pressure vessel, seals, pressure transmitting medium etc. but also an arrangement to apply load on piston which is necessary to generate pressure. The

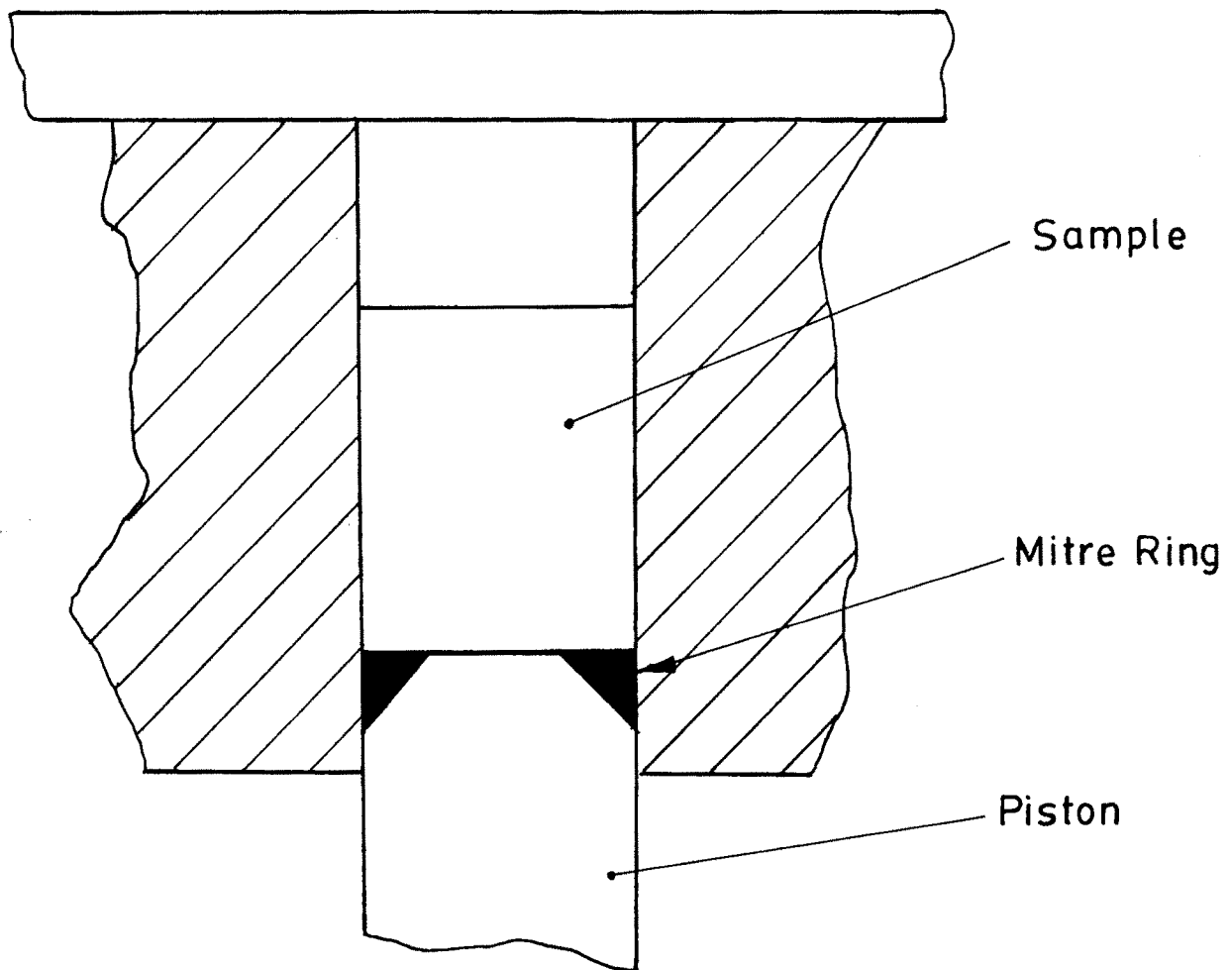


fig. 9-3

high pressure systems which are based on method - 1 are, piston-cylinder apparatus, belt apparatus and girdle apparatus.

A piston-cylinder apparatus based on the design of Kennedy and La Mori [11] is shown in Fig. 9.4. 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.

The belt [12,13] and the girdle [14,15] apparatus are shown in Figs. 9.5 and 9.6. In this family of devices the pistons (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

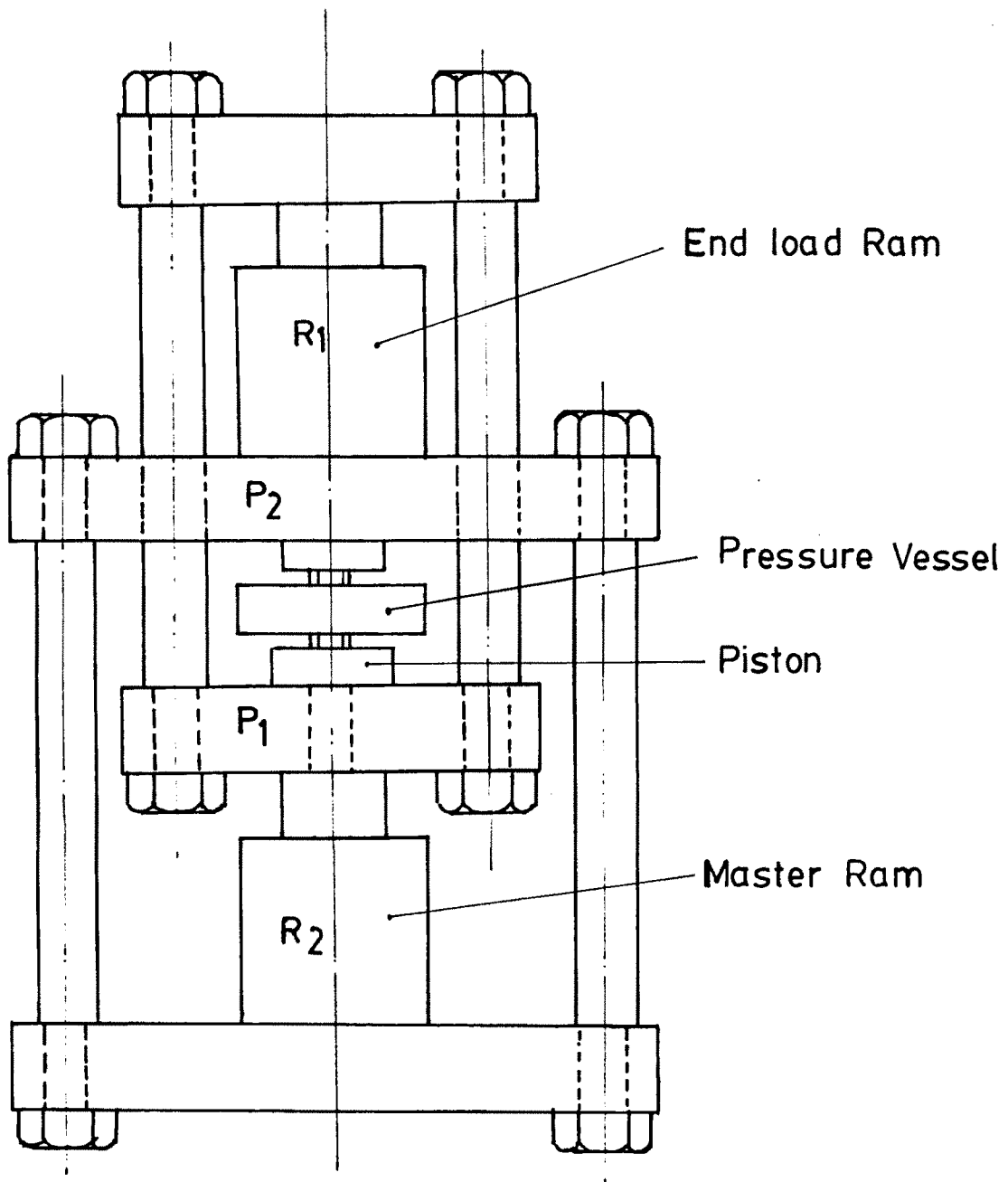
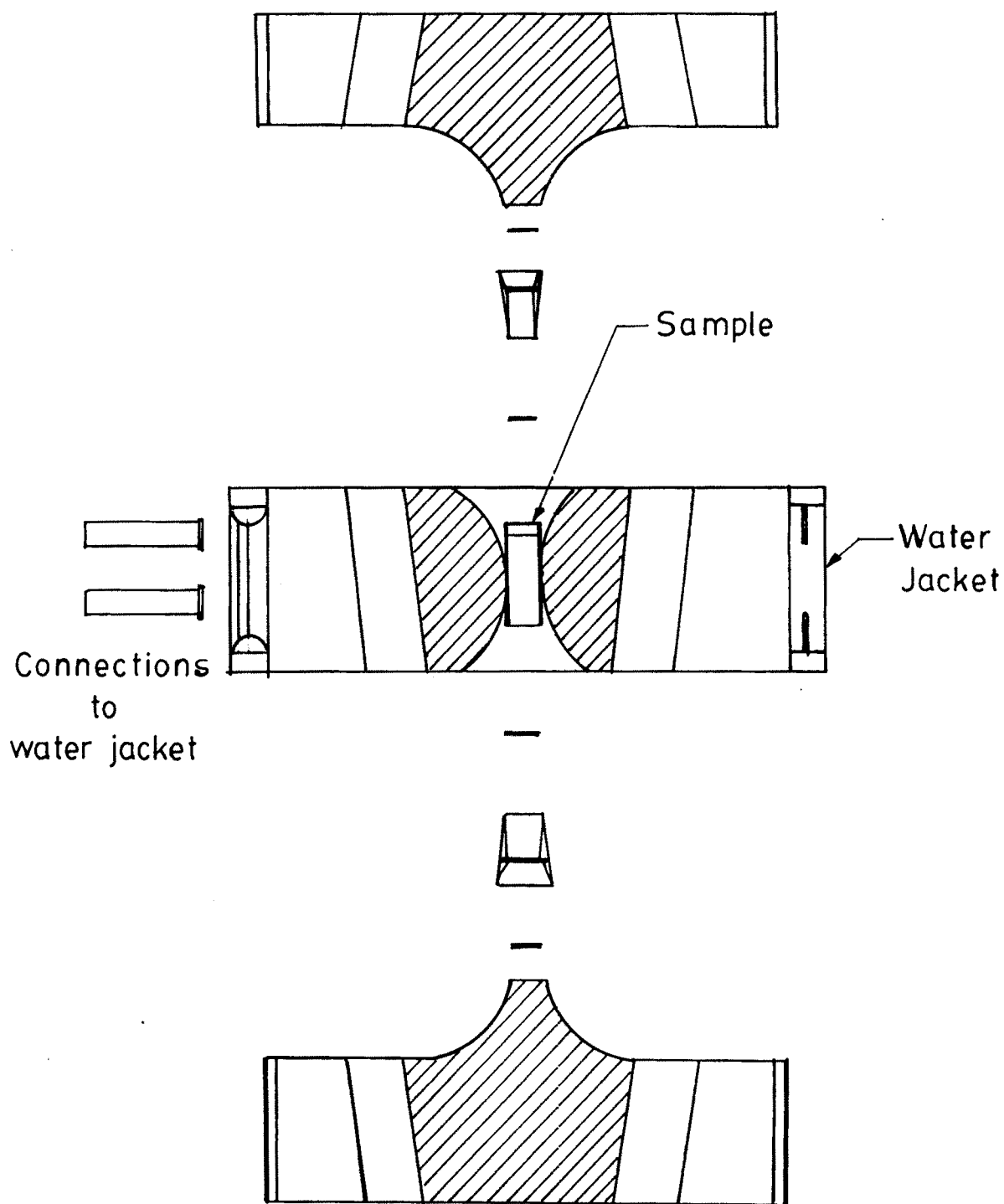


fig. 9.4







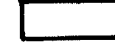
-  - Hard steel plug
-  - Tungsten carbide
-  - Carbon
-  - Pyrophyllite
-  - Steel binding rings

fig. 9.5

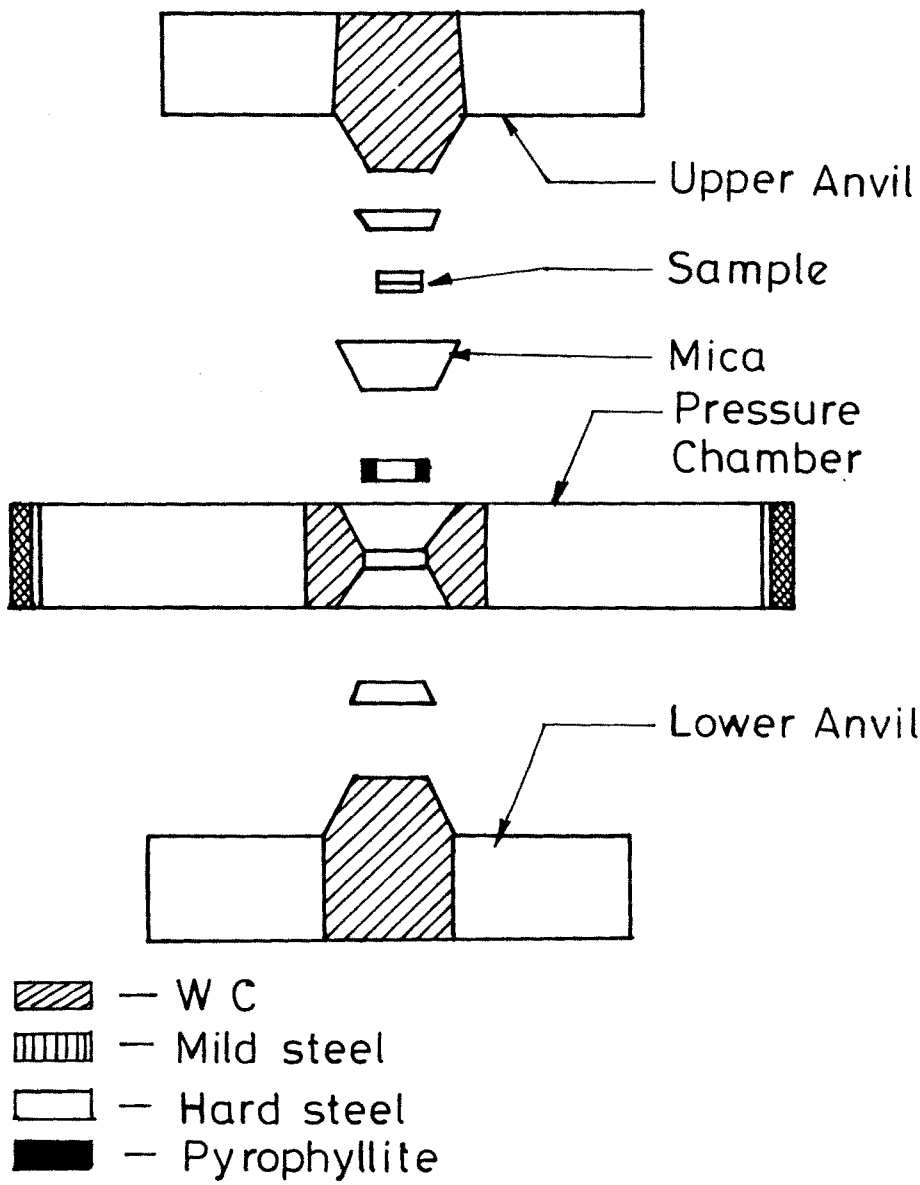


fig. 9.6

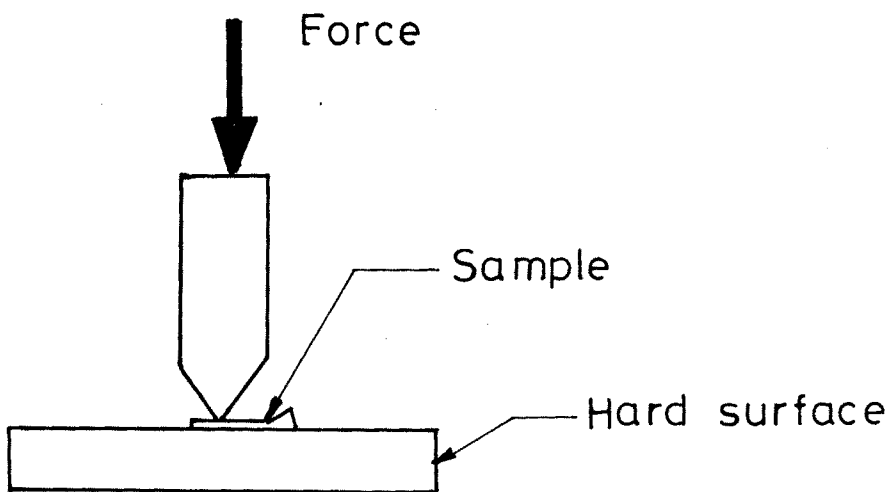
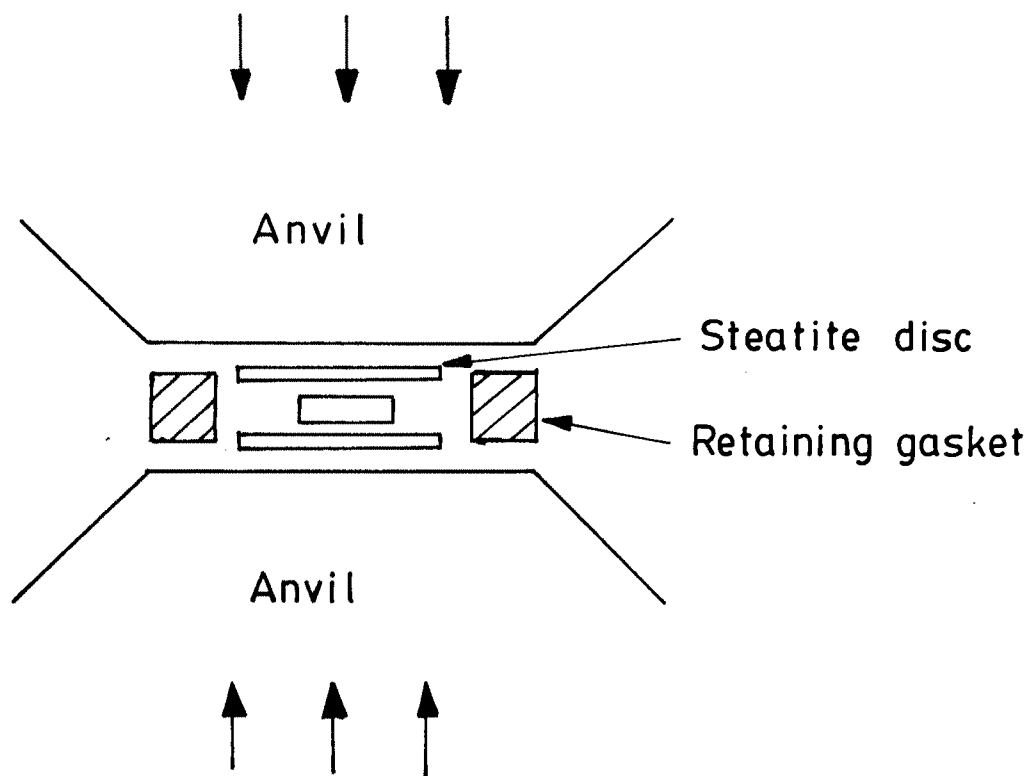


fig. 9.7

used for synthesis of materials such as diamond and cubic boron nitride.

9.2.2 Method - 2

The principle on which another group of high pressure instruments are based is shown in Fig. 9.7. 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 where as the ease of sample loading and introducing the measuring probes in the high pressure region (electrical or optical) requires a reasonably large area. These considerations have led to the development of devices called opposed anvil devices. Fig. 9.8 illustrates the principle of the Bridgman opposed anvil apparatus. The anvil consists of a tungsten carbide cylindrical piece, one face of which is ground in a tapered form ending with a flat anvil surface. In a typical anvil, the anvil surface has a diameter of say 4 mm, the cylinders



Principle of the Bridgman anvil apparatus

fig. 9·8

of diameter 15 mm and the taper angle about 10° . The anvil face experiences a high pressure but the material is heavily supported to prevent its failure. Thus, the anvil faces can support pressures in excess of 100 Kbar.

The anvils are pressed against each other and samples are typically 1 mm x 0.5 mm x 0.1 mm in size. The sample is enclosed with steatite discs and surrounded by pyrophyllite gaskets. The frictional forces acting between the anvil face and the gasket material prevents the extrusion of the material and the high pressure is transmitted to the substance under investigation. Naturally, the nature of the gasket material is important. By carefully controlling the dimensions of the gaskets and the amount of the pressure transmitter, a reproducibility of nearly 1 % can be obtained.

The Bridgman opposed anvils are used upto about 100 Kbar. With lateral support for the tungsten carbide anvils, one can go to about 150 Kbar and with slight modification as suggested by Drickamer, the pressures can be pushed to 300 Kbar. For higher pressures, the diamond anvil arrangement is needed as discussed below. :

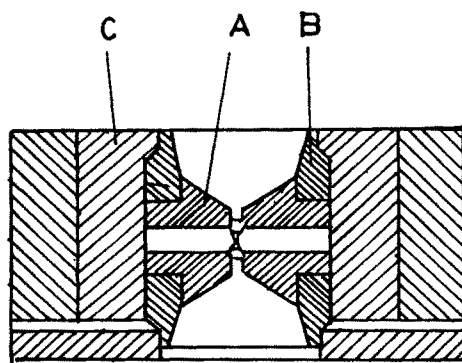
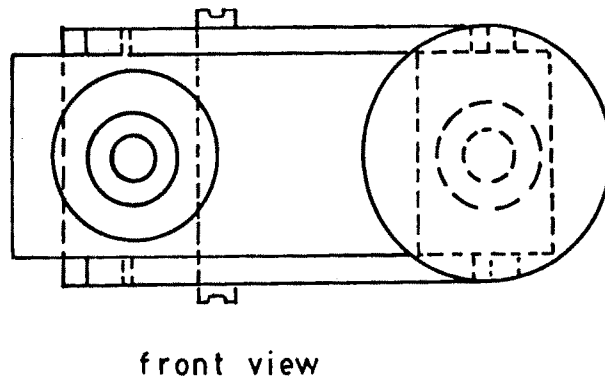
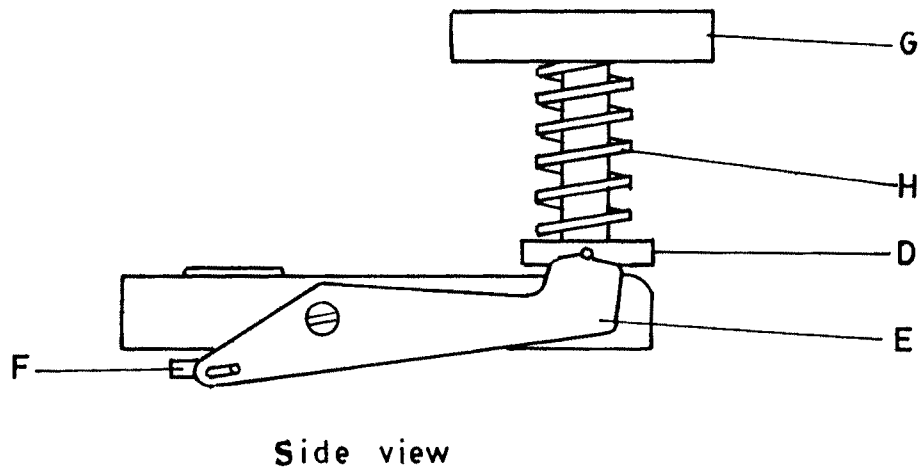
DIAMOND ANVIL APPARATUS

The diamond anvil apparatus, though based on the same massive area support principle as the Bridgman anvils, deserve special mention because it can go to the highest static pressures.

A simple form of a diamond anvil cell is shown in Fig. 9.9. 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 anvils 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 pistons, raises the pressure capability from 30 to over 100 GPa. The gasketing techniques also permits 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 of care for these



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 sprig.

fig. 9.9

high pressure studies.

9.2.3 Method - 3

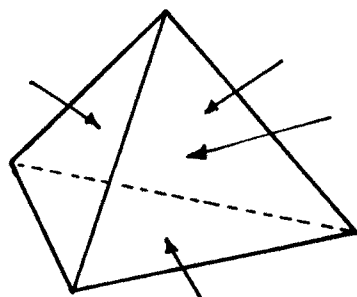
A totally different approach for generating high pressures is illustrated in Fig. 9.10. The sample is placed inside a gasket (made of pyrophyllite or MgO-epoxy) which is in the form of a polyhedron. The loads normal to the surfaces of the polyhedron are applied using anvils; the number of anvils equals the number of the plane faces of the polyhedral gasket. This class of instruments are often called multi-anvil devices. The first multi-anvil device was built with a tetrahedral gasket configuration [17]. This device is capable of going up to 10 GPa. The movements of the anvils have to be synchronized and directed normal to the tetrahedron faces. This is achieved with the help of the various mechanical linkages or the guides. The cubic presses [18] 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. 9.10 is very useful in X-ray diffraction work [19]. 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

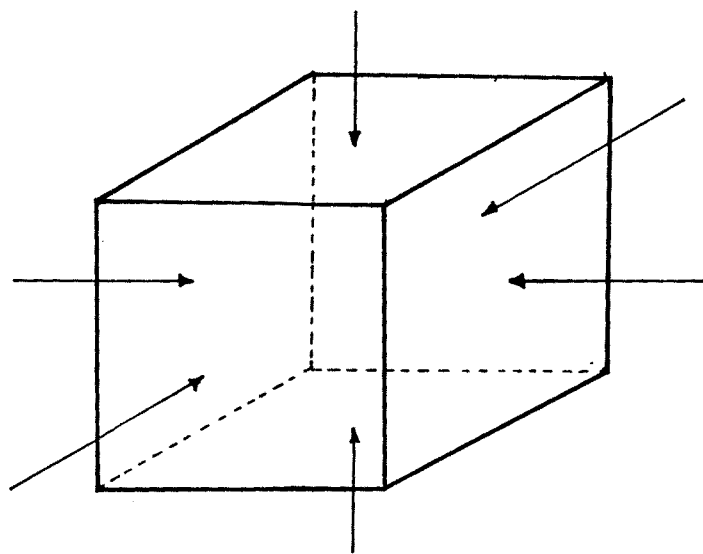
hydraulic rams to drive the anvils. In other category of the multi-anvil devices, the anvils and the supports can be thought of as forming a sphere. A high pressure at the centre of the sphere can be generated by subjecting the outside of the sphere to hydrostatic pressure [20-21].

9.2.4 Method - 4

The pressures in the range of 500 GPa can be generated by subjecting a sample to shock wave produced by detonating explosives. The pressures of similar magnitude are also produced if a solid accelerated by the detonation of some explosive is made to impinge on the sample. In contrast to the methods described earlier, this method produces dynamic pressures with a time duration of the order of a few micro-seconds. The small time duration poses experimental problems requiring sophisticated instrumentation. The propagation of shock waves through solids is quite complicated which leads to involved data reduction procedures. There are serious limitations on the type of information that can be obtained by this 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 techniques do not enjoy the popularity of other high pressure techniques, even though shock wave techniques can give extremely high



Cube



Hexahedron

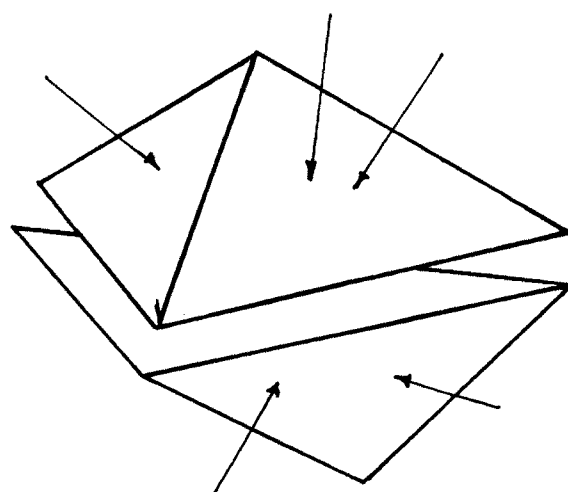


fig. 9-10

pressures. This technique has therefore not been detailed over here. One can see the details in reference [22].

In conclusion, a mention of pressure region, type and technique of pressure generation with maximum pressure produced is shown in Table 9.1.

9.3 UNITS OF PRESSURE

Some of the units commonly used in high pressure studies are $1 \text{ bar} = 10^6 \text{ dynes/cm}^2 = 0.9869 \text{ atm}$
 $= 1.0197 \text{ Kg/cm}^2$
 $= 14.54 \text{ psi.}$

The S. I. unit of pressure is the Pascal (Pa). The Pascal by definition, is 1 N/m^2 , 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 kilobar. Table 9.2 gives the various units of pressure and their interconversion factors.

9.4 MEASUREMENT OF PRESSURE

The primary methods of measuring pressure so far available are :

9.4.1 The Mercury Column

For direct pressure measurements, the mercury

Table 9.1 Experimental Techniques

Pressure (Region)	Method (type)	Technique	Maximum Pressure produced (GPa)	Typical compression achieved (V/V_0)
Low pressure (0 ~ 4) (GPa)	Static	Piston Cylinder (PC)	4.5	0.95
High pressure	Static	Anvil Cell Tungsten carbide (WC)	100	0.7
(4 ~ 100) (GPa)	Static	Diamond Anvil Cell (DAC)		
Ultra High Pressure > 100 GPa	Static	Magnetic Compression Gas Gun	500	0.25
	Dynamic	Shock wave chemical explosive	500	0.5
	Dynamic	Laser	~1000	0.5
	Dynamic	Under Ground Nuclear Explosive	~3500	0.3
	Dynamic	Electric Gun	~7000	0.3
	Dynamic	Rail Gun	~1000-5000	-
			~1000-10,000	-

Table 9.2 Principal Units of Pressure and Conversion Factors

	Bar	Nm ⁻² (Pa)	Kg cm ⁻²	Atm.	Lb. in ⁻²	in Hg	mm Hg
1 bar	1	10 ⁵	1.01971	0.98692	14.5038	29.530	750.059
1 Nm ⁻¹ (Pa)	10 ⁻⁵	1	1.01971x10 ⁻⁵	0.98692x10 ⁻⁵	14.5038x10 ⁻⁵	29.530x10 ⁻⁵	750.059x10 ⁻⁵
1 Kg cm ⁻²	0.98067	0.98067x10 ⁵	1	0.96784	14.2234	28.959	753.56
1 Atm	1.01325	1.01325x10 ⁵	1.03322	1	14.6960	29.921	760.0
1 lb in ⁻²	0.68947	0.68947x10 ⁵	0.070306	0.068045	1	2.0360	51.716
1 in Hg	0.33864	0.33864x10 ⁵	0.034531	0.033421	0.49116	1	25.40
1 mm Hg	0.0013332	0.0013332x10 ⁵	0.0013595	0.0013158	0.019337	0.03937	1

1 bar = 10⁶ dynes cm⁻². The Pascal (1 Pa = 1 N.m⁻²) has been adopted as the unit of pressure in the International system of Units (SI), Paris, 1960.

manometer is limited to about 30 meters in length, which corresponds to nearly 40 bars. It may, however, be used as a differential gauge up to pressures 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. 9.11. A free-piston closely fitting 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 regions 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 problem if the leakage of the pressure medium is slow. This will however, require a

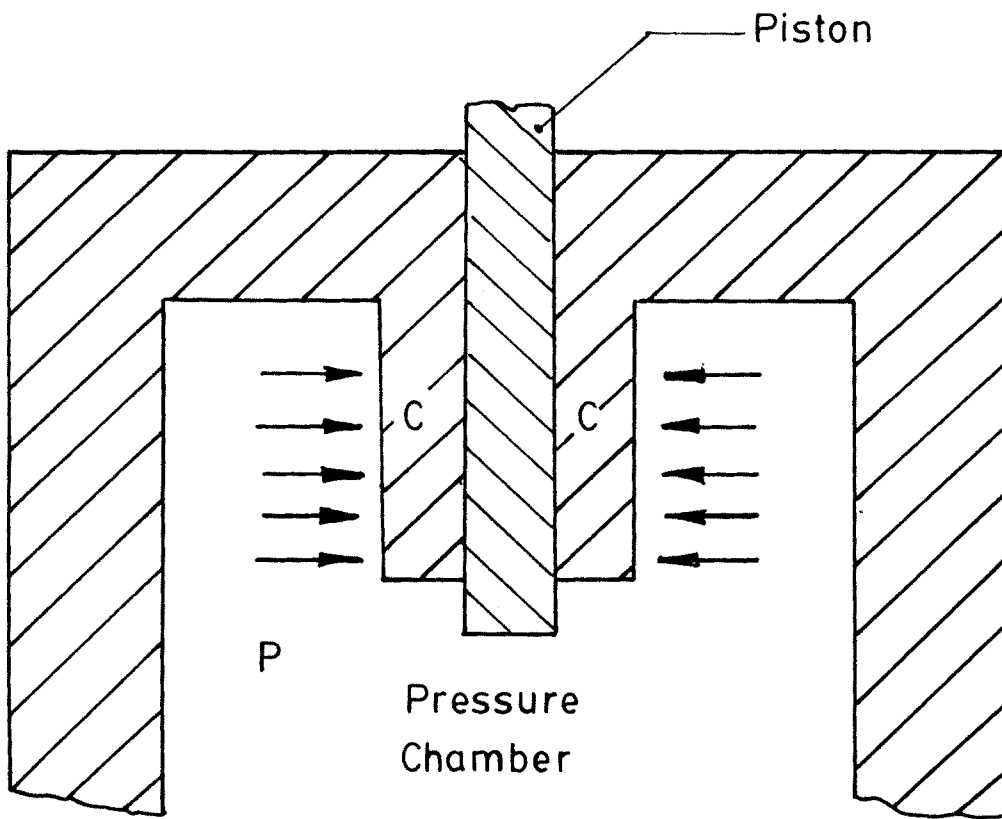


fig. 9-11

continuous supply of pressure medium at high pressure to compensate for the leakage past the free-piston. A carefully designed and fabricated free-piston gauge can be used up to 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 and Lawson [23] as early as 1954. But the credit of realizing this method in practice goes to Ruoff et al [24,25]. 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

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

9.4.4 The Equation of State

The equation of state for a suitable solid calculated from first principles will be the ideal thermodynamic basis for absolute pressure measurement. But

in practice, for useful substances in high pressure, calculation of equation of state becomes a formidable problem, and vice versa. The best that is available is the Decker NaCl equation of state [26,27]. 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 [28,29]. Such an apparatus uses a hydraulic ram to move the piston. By rotating the ram and averaging data from the forward and backward strokes. (to estimate frictional forces) pressure at 50 Kbar may be estimated to within around 0.5 Kbar. At 80 Kbar uncertainties are probably as high as ± 3 Kbar. An excellent review of this method is given by Richter and Clark [30]

9.4.6 Galvanic Cell as Primary Pressure Indicator

This technique offers a very simple and rigorously 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 gauges used in practice is given below :

9.5.1 Measurement Based on Non uniform Strain

For pressure measurement < 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 degree 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 2 GPa with a pressure co-efficient of resistance nearly 0.023 (GPa)^{-1} . However, the pressure coefficient depends on the exact composition and the manufacturing details of the wire. Many investigators 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 Duecker was quite useful in the past.

Recently, ruby-fluorescence technique [31-33] has been extensively used in the measurement of pressure. A sharp line fluorescing material is placed in the high pressure cell alongwith 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 (Al_2O_3 : 0.5 % Cr), YAl_2O_3 (0.2 % Cr), YAG (0.38 % Cr_2O_3), YAl_2O_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 : 6928 and 6942 Å) was 3.6 Å /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 Transitions 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). Whereas in temperature measurement there are several defining fixed points, fixed points for pressure measurements had to be agreed upon. Following the symposium on the Accurate Characterization of the High Pressure Environment in 1968, the transition pressure values listed in Table 9.3 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 [34] discusses the status of fixed points above 100 Kbar.

Tables 9.4 and 9.5 summarise the range,

Table 9.3 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 $\alpha \rightarrow \epsilon$	110
Ba II-liq. (?)	120
Pb I-II	130
Rb II-liq. (?)	146
Fe ₁₅ Co $\alpha \rightarrow \epsilon$	150
Fe ₂₀ Co $\alpha \rightarrow \epsilon$	190
Fe ₄₀ Co $\alpha \rightarrow \epsilon$	290

Table : 9.4 Primary methods of pressure measurement- their range, sensitivity and accuracy.

Type of gauge principle	Pressure range (kbar)	Sensitivity (bar)	Possible accuracy	Limiting factor in Temperature effect	Temperature control (K) for measurement to 1 in 10 ⁴	Other remarks
Mercury manometer	0-0.04	10 ⁻⁶	1 in 10 ⁵	Thermal expansion of mercury, container and scale.	0.5	
Differential mercury manometer and pressure balance	0-3	10 ⁻⁶	1 in 10 ⁵	Thermal expansion of mercury, container, scale, piston and cylinder	0.5	Equation of state of mercury is the limiting factor in the accuracy
Pressure balance	0-26					
3 kbar		0.01	0.1 bar	Thermal expansion of piston and cylinder	0.5	Commercial units working to 15 kbar are available
10 kbar		0.01	1 bar			
26 kbar		0.1	25 bar			
Simultaneous measurement of volume and compressibility	Not stated but should be usable to 20kbar and to 80 kbar in modified form	Not stated but 0.1 bar	± 1.6 bar at 7.5 kbar	Thermal expansion of specimen	5 x 10 ⁻³	Ruoff et al (1973) ; further work is necessary before its adoption as a primary measurement system.
Equation of state of solids (volume pressure)	0-300	100(a)	1% below about 30 kbar. 10% at about 30 kbar	Thermal expansion coefficient		Scales for very high pressure range not fully developed. Scale depends on knowledge of equation of state of substance.

(a) This value refers to a typical system for use to over 20 kbar. Much higher sensitivity may be obtained in the low-pressure range (see Ref (35))

Table : 9.5 Secondary methods of pressure measurement - their range, sensitivity and accuracy.

Type of gauge Principle	Pressure range (kbar)	Sensitivity (bar)	Possible Accuracy	Limiting factor in temperature effect	Temperature Control reqd. for measurement	Subsidiary Comments	Other remarks
Melting line of mercury	0-15	0.5	1 bar	Slope of melting line	0.01 for ± 1 bar	Provisional pressure scale 0-15 kbar	Pressure determined from solid-liquid transition temperature
Strain of elastic element (e.g. Bourdon tube)	0-15	10^{-6}	1 in 10^3	Thermal expansion of element and reference plate	0.5 for typical gauge for ± 1 bar	Most widely used type of gauge.	Maximum sensitivity obtained with a strain capacitance gauge
Resistance element (e.g. Manganin or gold-chromium alloy)	0-30	10^{-2}	1 in 10^4	Temperature coefficient of resistivity	0.1 (depends on pressure) for ± 1 bar	Probably the most reliable secondary gauge from 10 to 30 kbar	Temperature coefficient of both alloys very small, but increases with temperature.
Capacitance gauge (e.g. CaF ₂)	0-2.5	10^{-1}	1 in 10^4	Temperature coefficient of capacitance	0.01 for ± 1 bar	New promising type of gauge	The range of this very accurate, simple gauge could perhaps be extended to 10 kbar
Ultrasonic velocity measurement	10-40	500(a)	1000(a) bar	Coefficients of thermal expansion and compliance	0.5 for ± 1 bar	Scales for very high pressure range not fully developed	Above 12 kbar quartz transducer must be outside pressure vessel: can be used to detect phase transition
Optical : Shift of ruby R lines with pressure	0-100	500	scale above 40kbar	Shift of line with temperature	5 for 1kbar	Should be capable of use upto 300 kbar in the diamond cell	

(a) This value refers to a typical system for use to over 20 kbar. Much higher sensitivity may be obtained in the low-pressure range (See : Ref : (35))

sensitivity and accuracy of primary and secondary pressure gauges developed so far.

9.6 CONCLUSIONS

The techniques of high pressure generation described in this chapter will now be used to study the behaviour of tungsten disulphide (WS_2) and tin monoselenide (SnSe) single crystals under high pressure. The results obtained during these studies will form the subject matter for Chapters 10 and 11 respectively.

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CAPTIONS TO THE FIGURES

Fig. 9.1 Schematic diagram illustrating the method of producing high pressure.

Fig. 9.2 The Commonly used Seals.

Fig. 9.3 Schematic diagram showing pyrophyllite mitre rings used for sealing solid pressure transmitters such as silver chloride and indium.

Fig. 9.4 Piston-Cylinder apparatus.

Fig. 9.5 The Belt apparatus.

Fig. 9.6 The Girdle apparatus.

Fig. 9.7 Principle of an anvil set-up.

Fig. 9.8 Principle of the Bridgman anvil apparatus.

Fig. 9.9 Sketch of a diamond anvil cell.

Fig. 9.10 Geometry showing Tetrahedron, Cube and Hexahedral presses for generating high pressures.

Fig. 9.11 The Pressure Balance : A typical free-piston gauge.