CHAPTER – II

DESCRIPTION OF THE 200 TON HYDRALIC PRESS AND THE DTA HIGH PRESSURE CELL

Part – I. 200 Ton Hydraulic Press

1. Introduction:

The most widely used technique for producing high pressure is the piston cylinder device. [1-4] Due to the non-availability of any commercial apparatus it became necessary to design, fabricate and assemble a single acting 200 ton hydraulic press incorporating the piston cylinder device with a view to carrying out a variety of experiments like differential thermal analysis (DTA), optical, volumetric studies, etc. on liquid crystalline materials under pressure. In this section we shall give in detail the constructional as well as the design aspects of the press and describe its working.

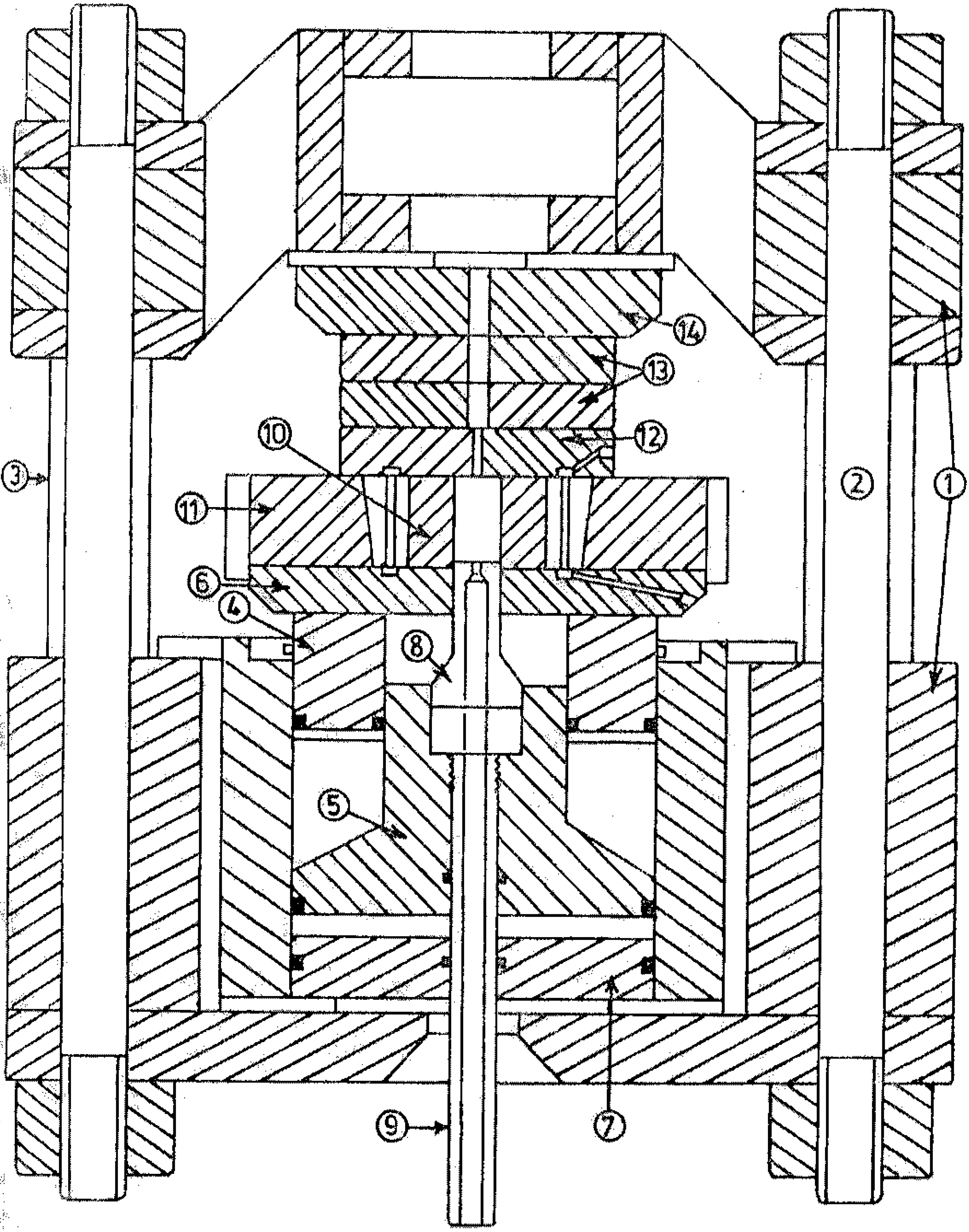
2. Description of the press

The schematic diagram of the assembled press is shown in figure 1. Each individual part of the press has been labeled and the details of each of these components will be described below in sequence. Table I lists the components of the press, and the material used in each case along with its hardness.

Figure – 1

Schematic diagram of the 200 Ton hydraulic press

1. Platens (top and bottom)
2. Tie rods
3. Spacers
4. End Load Ram
5. Master ram
6. End load plate
7. Static seal plate
8. Piston
9. Piston connecting rod
10. Pressure die
11. Binding ring with cooling jacket
12. Insulating groove pad
13. Support pads
14. Top platen pads



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One of the most important stages in the design of apparatus and instruments used in conjunction with high pressure is, generally the selection of suitable materials. [5] This becomes an acute problem when the pressures exceed 10 kbars and even more acute when high temperature are also required.

The materials used by us in the construction of these components have been chosen considering various factors like the appropriate pressure and temperature ranges, corrosion and fatigue resistance, ability to weld and machine and the extent to which heat treatment can be carried out. Most of the components are machined out of a low alloy hardenable Indian steel called EN-24, which has the composition of 0.4% carbon, 0.2% silicon, 0.55% nickel. This steel which is roughly equivalent to the widely used AISI 4130 ·type, can be hardened to great strength by means of martensitic transformation. The alloying elements, in addition to increasing the hardenability of the alloy also contributes with some solid solution strengthening. The pressure die (10) and the piston (8) were machined out a high strength steel, namely High carbon-High chromium (HC-HC) consisting of 2% carbon and 12% chromium.

a) Platens and Tie-rods

The upper and lower platens in figure 1 are shown in detail in figures (2) and **(3).** Both the platens were made from 1" mild steel (MS) plates which were hand welded together and normalized before machining. For the purpose of welding a silver type alloy Eutec rod 1801 of Larsen and Toubro with very high tensile strength of 90,000 psi and with very smooth filling property was used. It was ensured that the Eutec alloy covered all the crevices so that the possibility of air pockets acting as vulnerable points was completely precluded. Both the platens have matching holes at the centre for visual observation when an optical cell is used in the set up. The bottom platen is scooped out for positioning the master cylinder (fig 4) made from EN-24 and heat treated to a relatively low RC value of 30-35. This cylinder has eight through holes made at 150 PCD on both ends in order to bolt it to the pattern. The master cylinder is also provided with two pressure connections, leading to the master ram and end load ram. The platens are separated by tie rods (fig 5a). The tie rods after heat treatment to RC 35-38 are then turned and threads cut between centres. The tie rods are clamped to the platen by means of nuts (fig 5b), the nuts are being heat treated first to the same hardness as the tie rods and then machined and threads cut into it. Since the nuts would be taking a lot of load, it is ascertained that the bottom face of the nuts were squared to the axis of the thread to better than ± 0.02mm. Also six slots are cut on the OD of the nut using a milling machine. These slots suit in turn a hook spanner which facilitates complete tightening of the nut on to the platen.

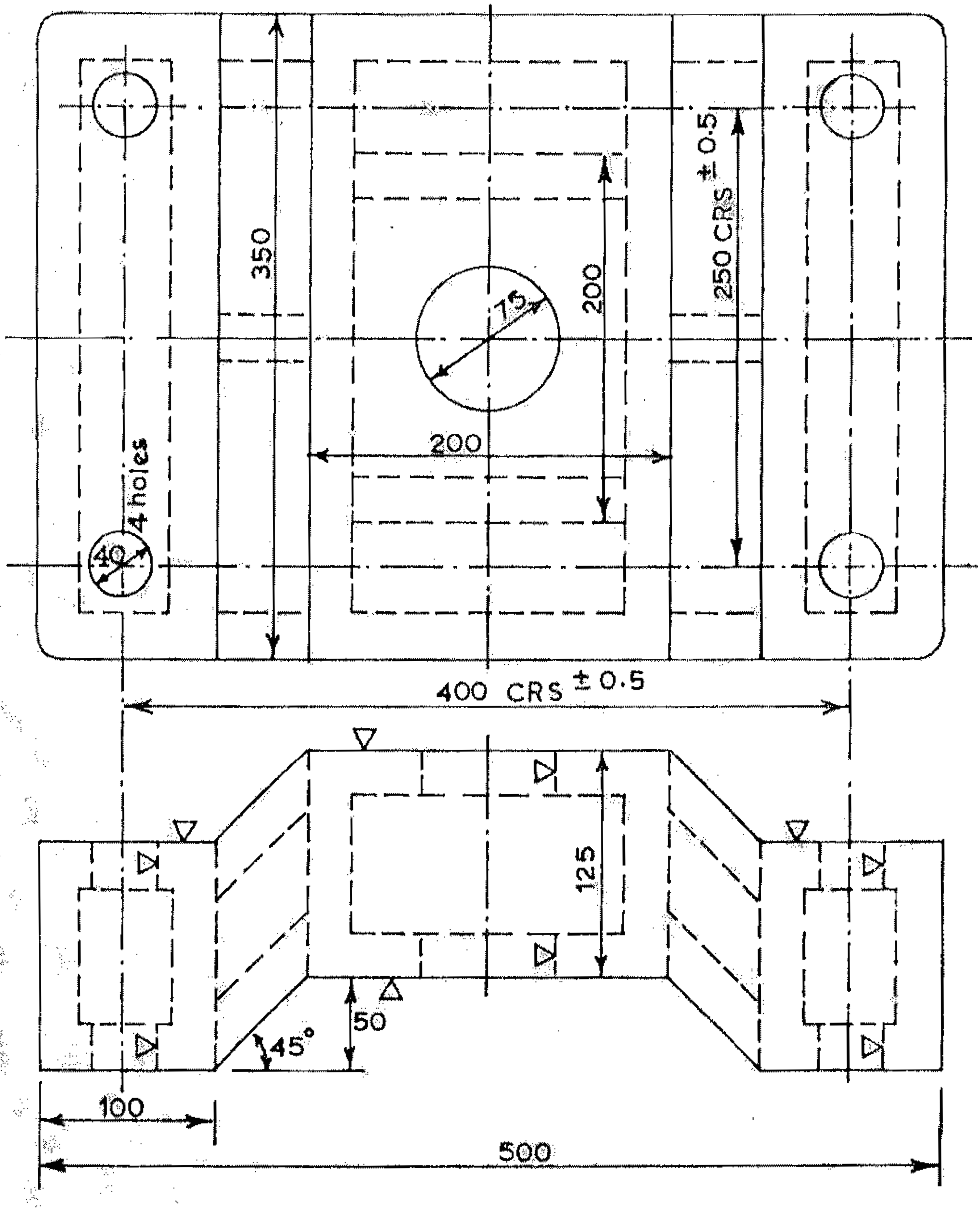


Fig.2: Top platen

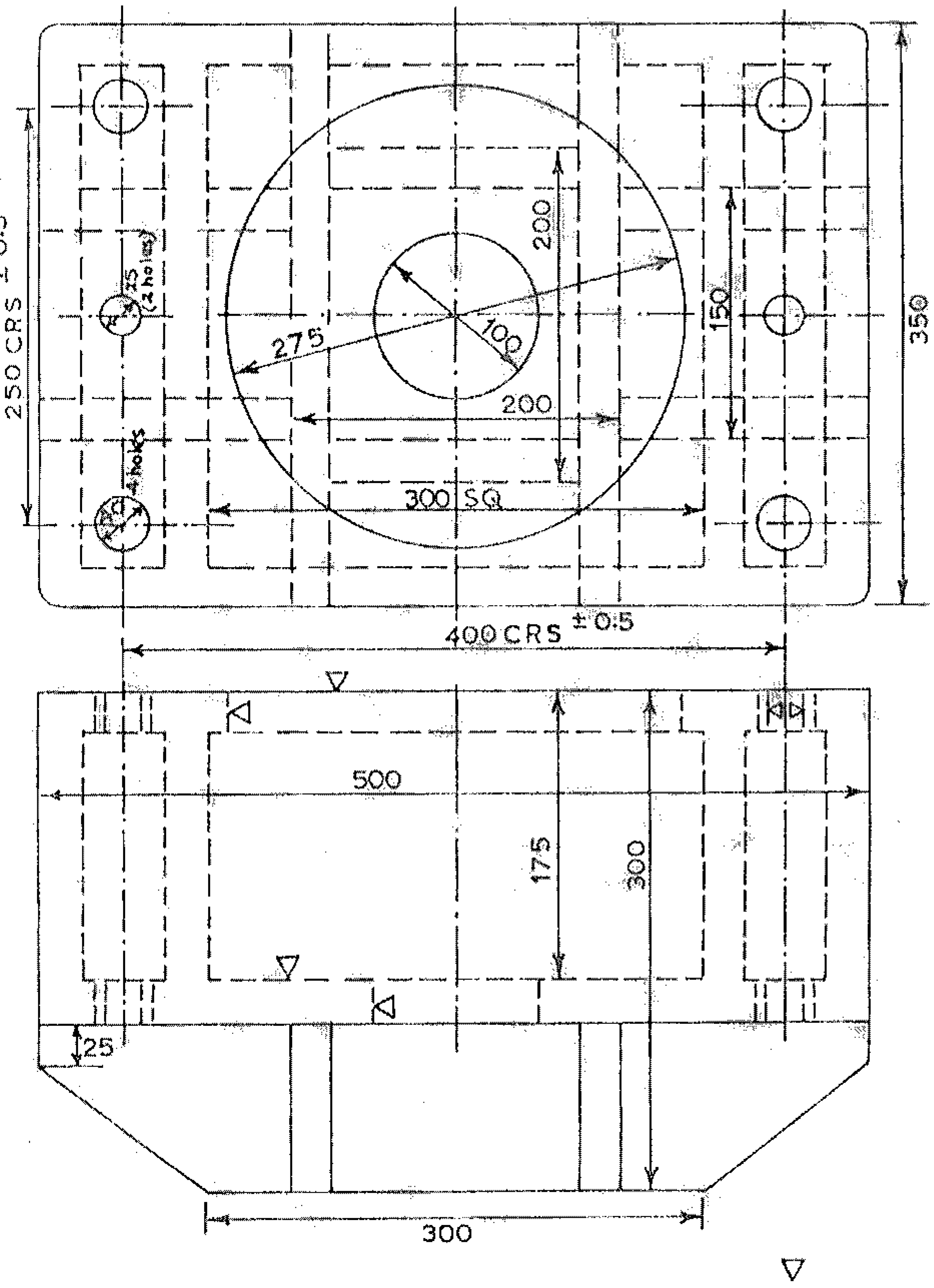


Figure 3: Bottom Platen

and normalized before machining. For the purpose of welding a silver type alloy Eutec rod 1801 of Larsen and Toubro with very high tensile strength of 90,000 psi and with very smooth filling property was used. It was ensured that the Eutec alloy covered all the crevices so that the possibility of air pockets acting as vulnerable points was completely precluded. Both the platens have matching holes at the centre for visual observation when an optical cell is used in the set up. The bottom platen is scooped out for positioning the master cylinder (fig 4) made from EN-24 and heat treated to a relatively low RC value of 30-35. This cylinder has eight through holes made at 150 PCD on both ends in order to bolt it to the pattern. The master cylinder is also provided with two pressure connections, leading to the master ram and end load ram. The platens are separated by tie rods (fig 5a). The tie rods after heat treatment to RC 35-38 are then turned and threads cut between centres. The tie rods are clamped to the platen by means of nuts (fig 5b), the nuts are being heat treated first to the same hardness as the tie rods and then machined and threads cut into it. Since the nuts would be taking a lot of load, it is ascertained that the bottom face of the nuts were squared to the axis of the thread to better than ± 0.02mm. Also six slots are cut on the OD of the nut using a milling machine. These slots suit in turn a hook spanner which facilitates complete tightening of the nut on to the platen.

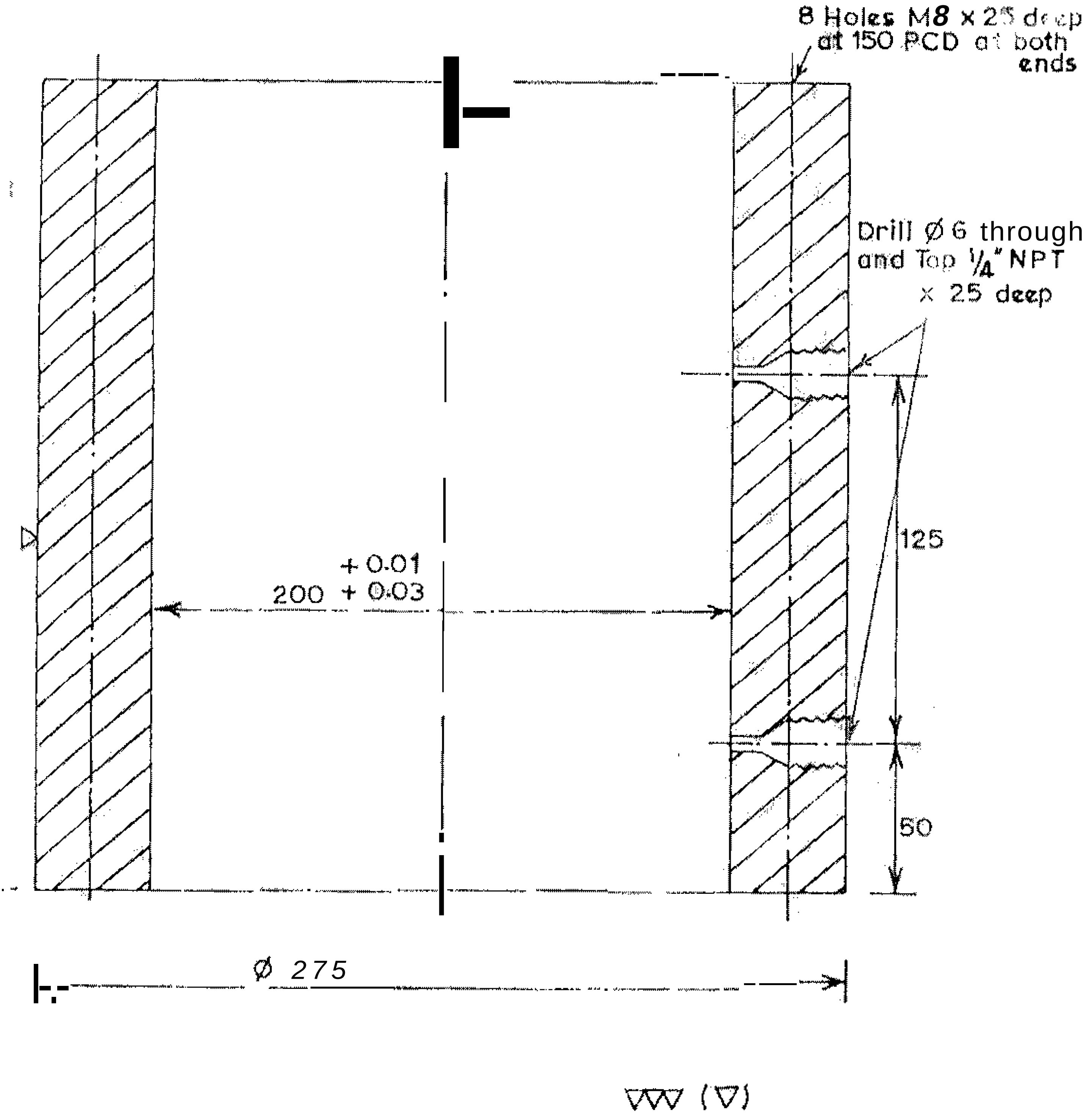
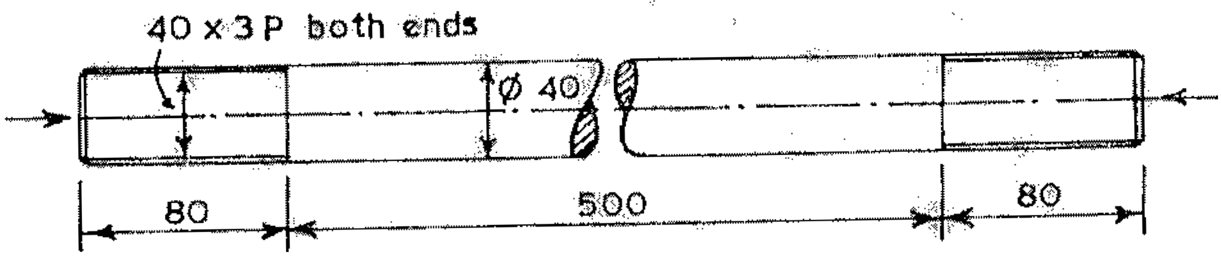


Figure 4: Master cylinder



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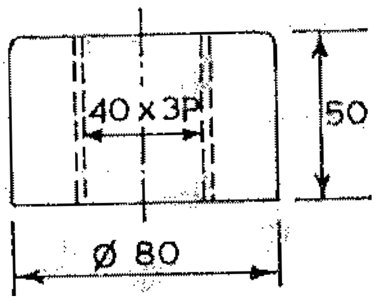


(b)

Figure 5: (a) Tie rod (b) Nuts for the tie rods

b) <u>End load ram and the end load support plate</u>

The end load ram (fig 6) has a high hardness of RC 50-55 and is machined such that all the diameters are concentric to 0.05 TIR and all faces are square to the axis within 0.5 TIR. This ram floats at the top of the master cylinder and moves up on pumping the hydraulic oil into the chamber through the top opening of the master cylinder (fig 4). The bottom of the end load ram has a groove for seating an ‘O’ ring which acts as the seal. The sealing property of the O ring is based typically on the initial squeeze of the O ring in the assembly. [(6)] The depth of the groove made in the end load ram for the O ring is slightly less (about 10%) than the diameter of the O ring. As the pressure is applied, O ring deforms assuring a leak tight seal at high pressures. The O ring used in our set up was made of neoprene. To prevent an extrusion of the O ring due to high pressures an 'anti-extrusion' ring [(7)] of aluminium was used between the neoprene ring and the groove. The use of this ring was extremely successful and even after about 3 years of continuous use of perceptible leakage was observed. The upper end of the end load ram has eight M8 holes at 150 PCD on both

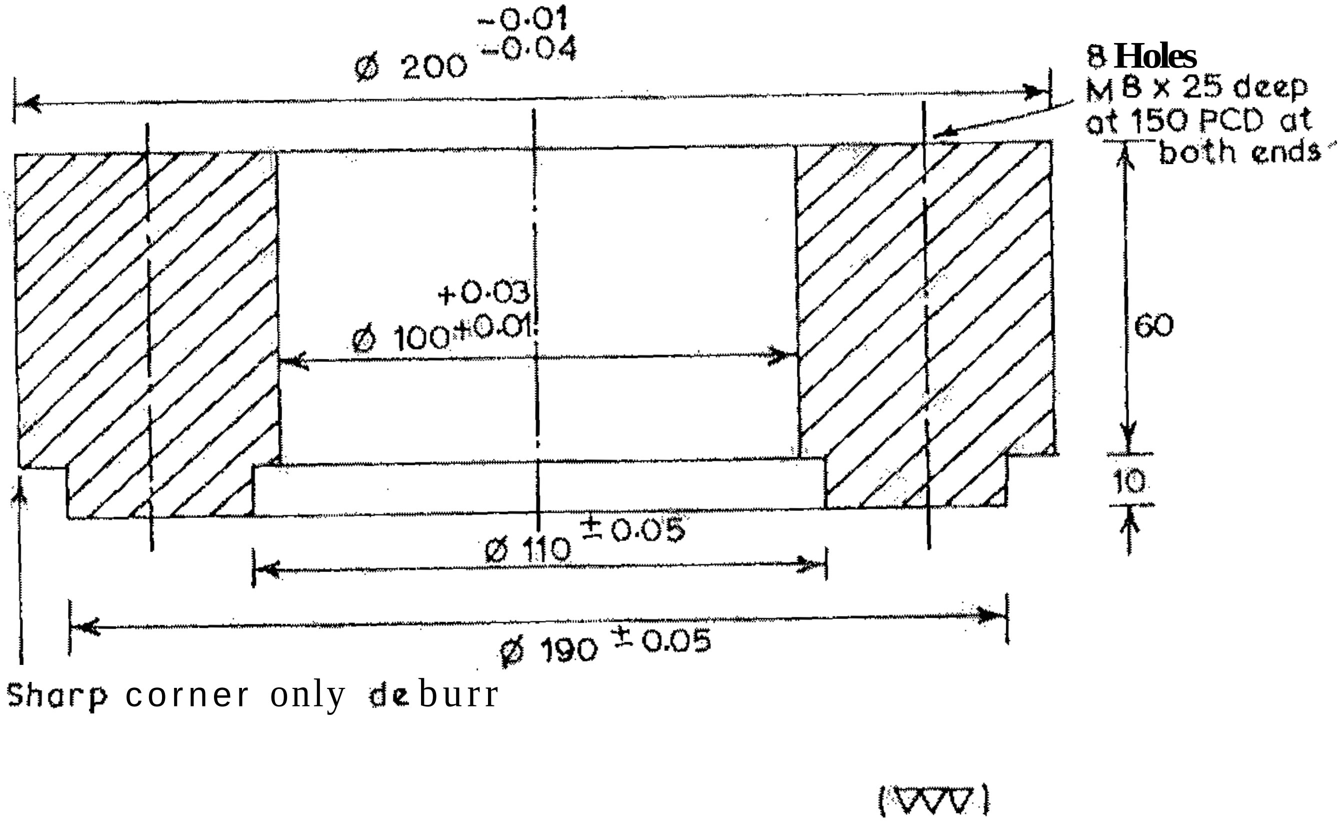


Figure 6: End load ram

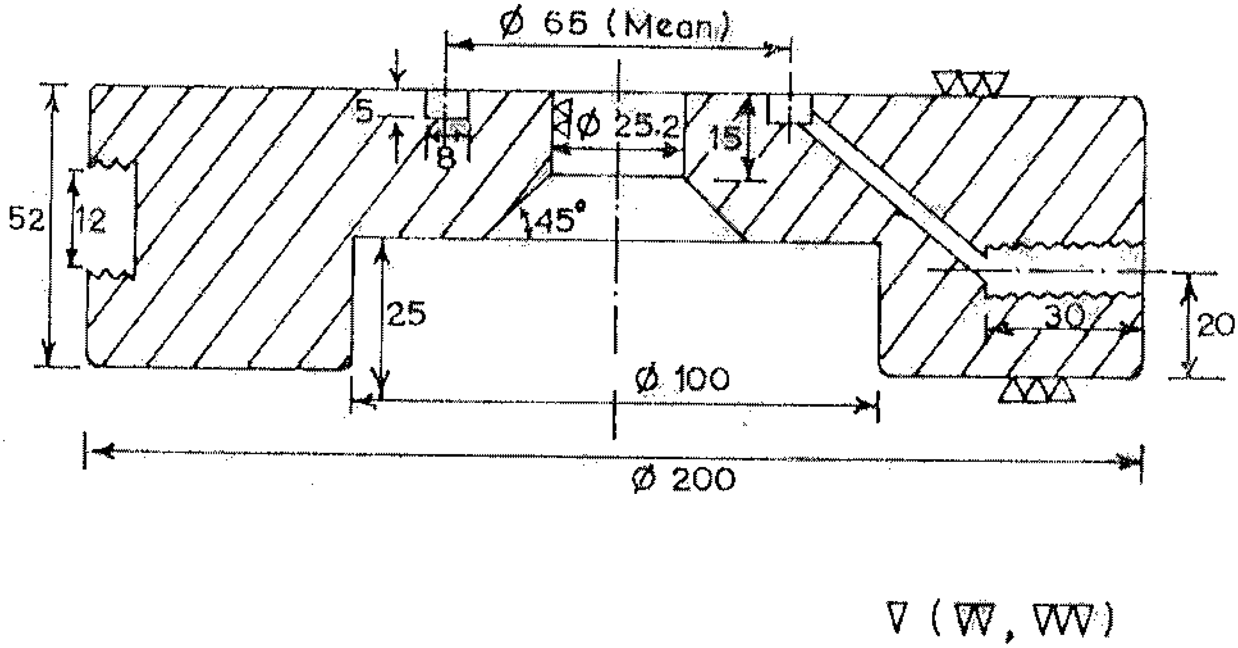


Figure 7: End load support plate

ends for fixing the end load support plate (fig 7) which acts as a pad for the pressure plate. The end load plate has a groove scooped out concentrically, the groove being connected to a water inlet through a slot made at an angle in the interior of the plate. There is a cavity on the outside surface of the plate into which ½ " BSW threads are cut to enable to take electrical connections.

c) Master ram

The Master ram (fig 8) has a 100mm diameter on the surface and rests on a static seal plate (fig 9), which in turn is fastened on to the master cylinder (fig 4). The area between the lower surface of the master ram and the upper surface of the static seal plate serves as the chamber into which oil can be pumped thereby raising the master ram. The grooves in the master ram and the seal plate in which the O rings are seated have their edges deburred in order to prevent the cutting of the O ring. The top of the master ram has a step in which a steel pad (fig 10a) is inserted.

d) High pressure piston and the pressure die

The high pressure piston (fig 11) which is heat treated to high RC 62-65 rests on the pad (fig 10a). It is an unsupported [8-9] piston which, when the master ram is raised advances into the pressure die. The piston is coupled through a connecting rod (fig 10b) to a long stroke linear displacement meter to facilitate accurate monitoring of the

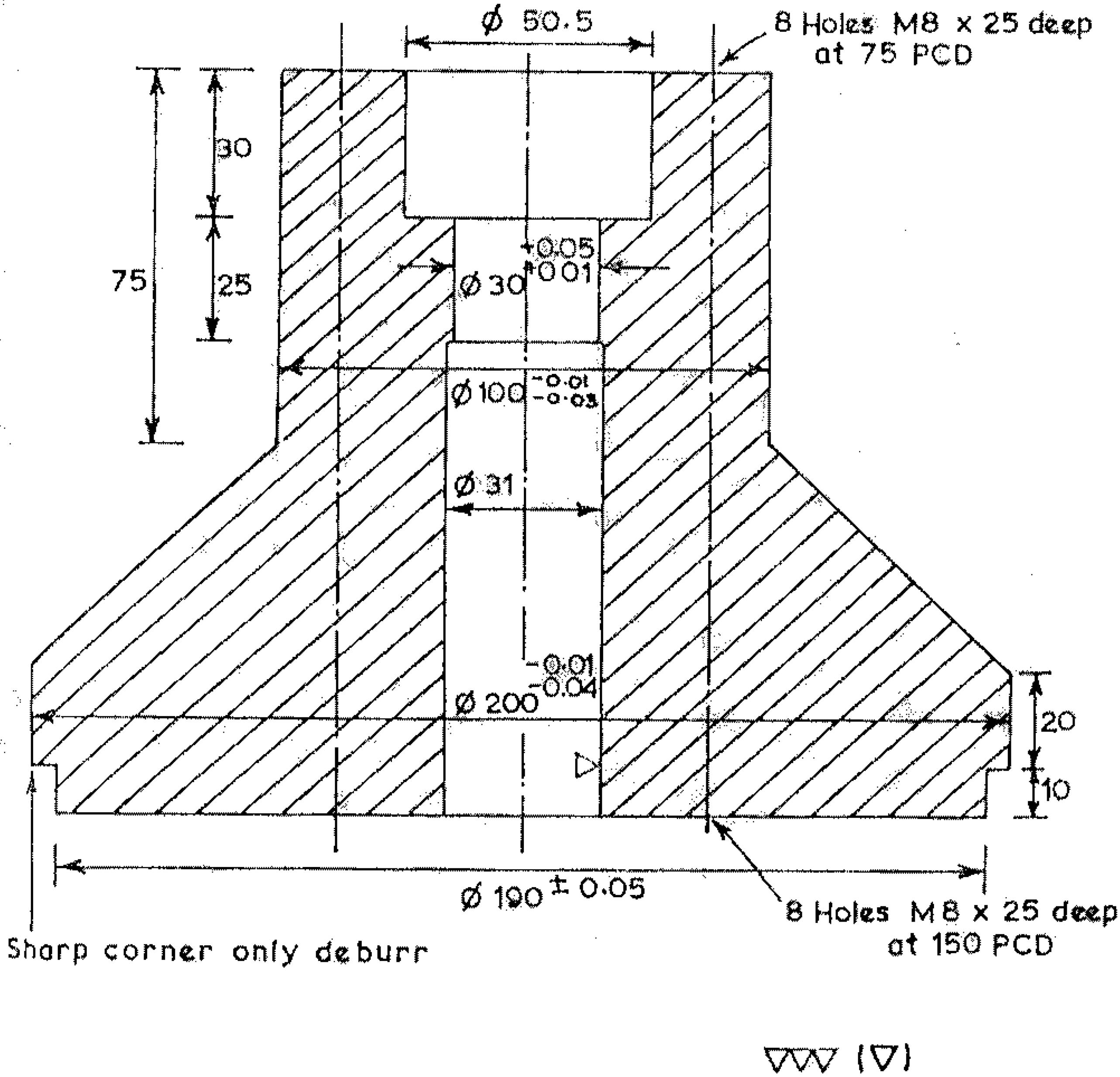


Figure 8: Master Ram

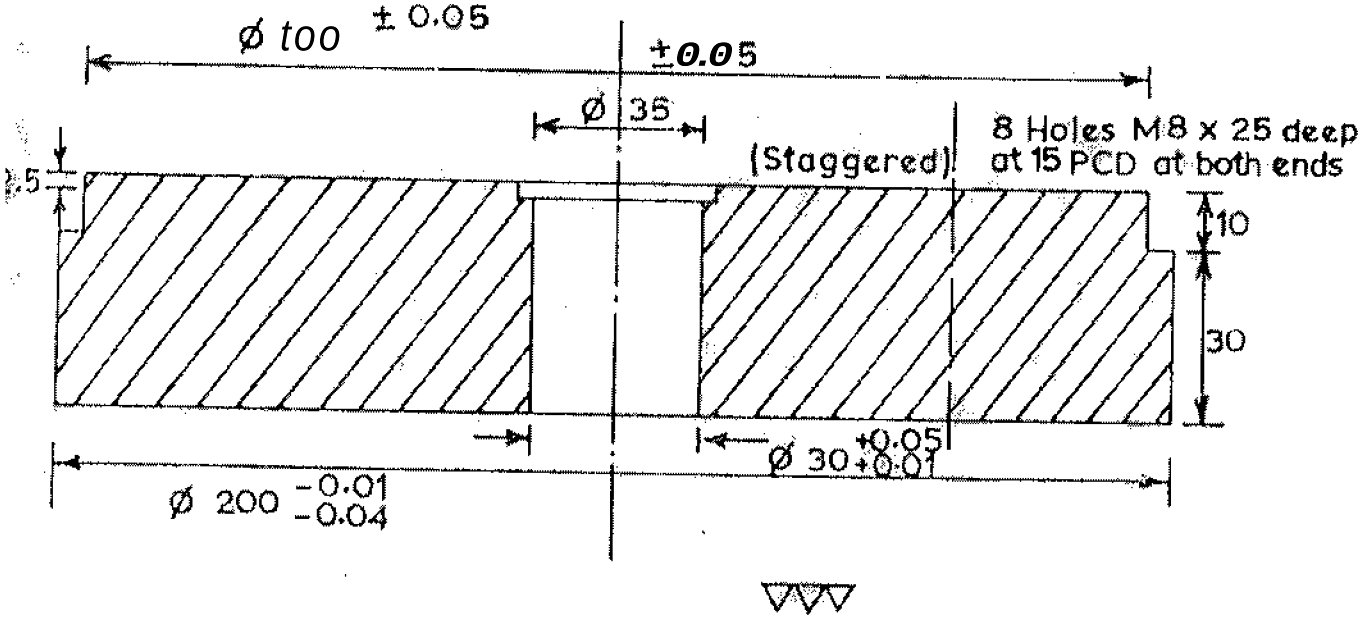
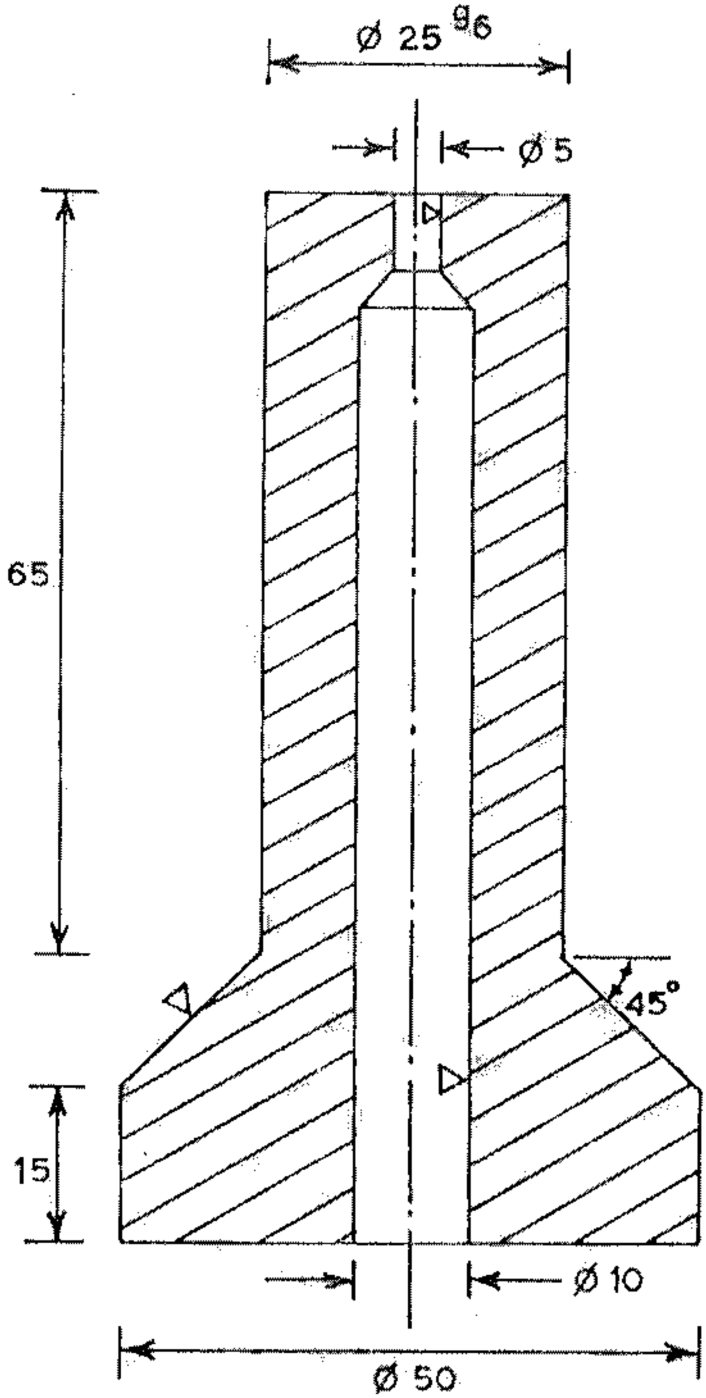


Figure 9: **Static seal plate**



VVV (V)

Figure 11: High pressure piston

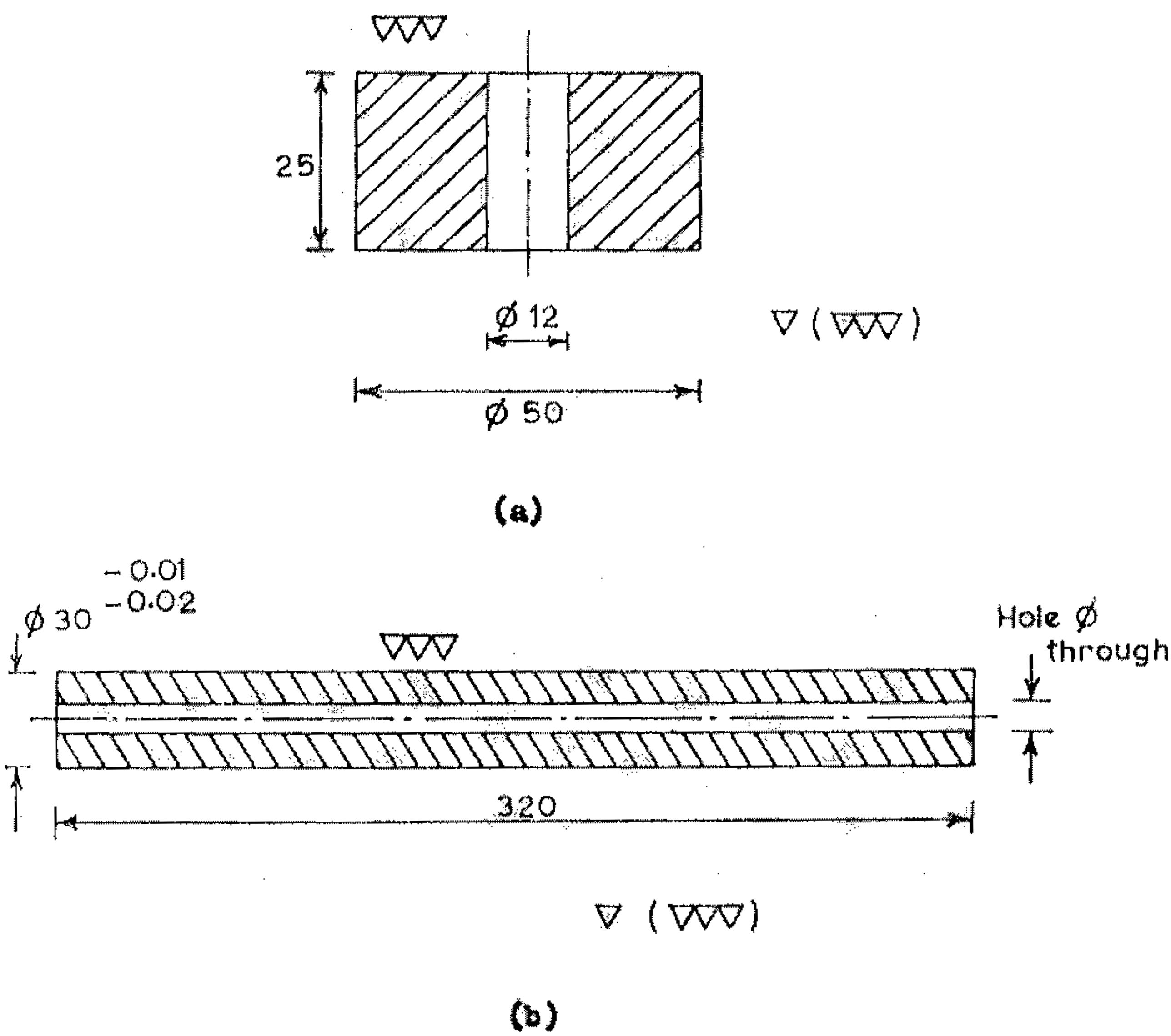
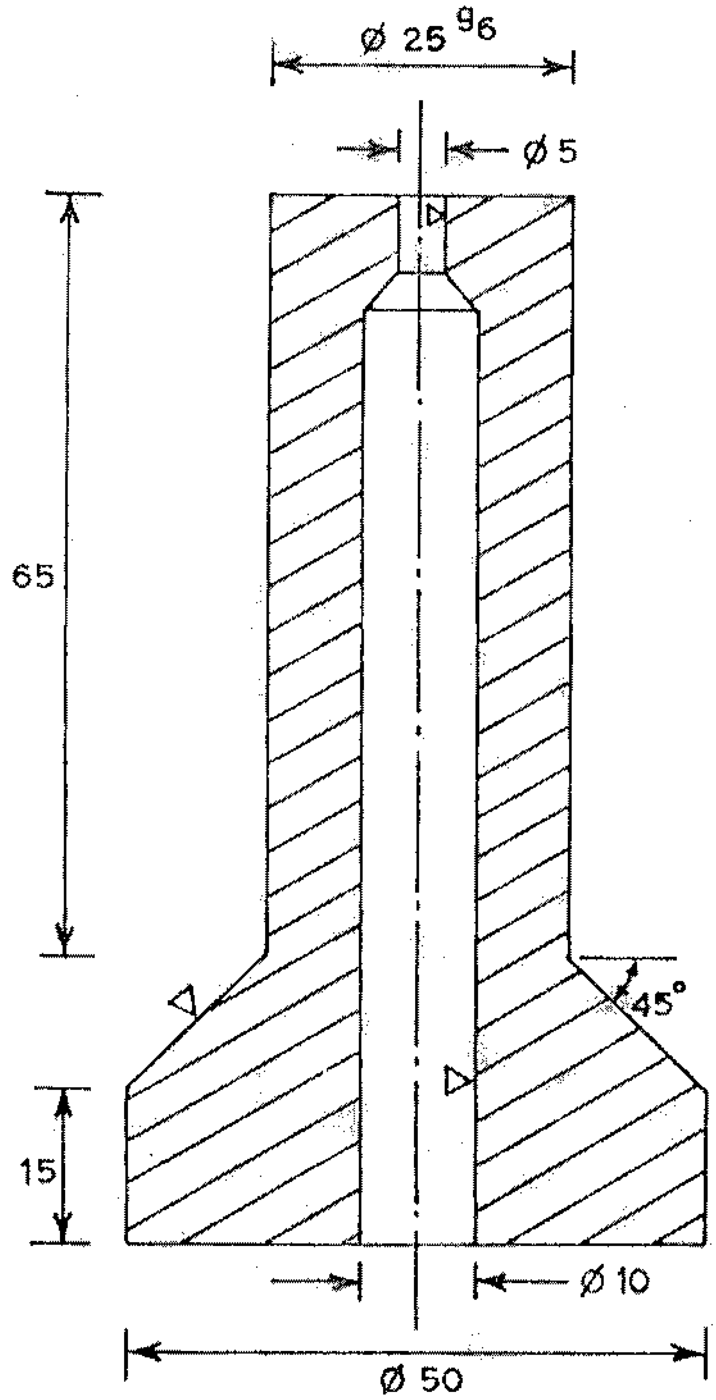


Figure 10: (a) Support steel pad

(b) Piston connecting rod



VVV (∇)

Figure 11: High pressure piston

Movement of the piston (we shall be describing this in more detail while discussing the working of the press). The piston, piston pad as well as the connecting rod have all got a coaxial central hole through which light beam may be passed for optical observations.

The core of the pressure die (fig 12.1) is made out of HC-HC and heat treated to RC60-62. The inside diameter of the die is ground to a very great accuracy to match the outer diameter of the piston to an accuracy of ±2 microns. Since most of the liquid crystalline experiments require to be carried out at high temperature and since at high temperature the strength of the steel is known to diminish, [4] a cooling jacket made out of EN-24 into which the pressure die fits is used. The cooling jacket (fig 12.2) also serves as a binding ring, the outside diameter of the sleeve has a single 1 ½ 'taper, an interference [1] normally given for such components.[10] This sleeve is then fitted into a second binding ring (fig 13a) which has a matching 1 ½ 'taper on the internal diameter, the fitting being done under a load of 5000 psi. This is necessary to prevent the bursting of the binding sleeve at high pressures. The cooling jacket is provided with 25 holes at 65 PCD through which water at a pressure of about 40-45 psi is circulated. The binding ring itself is surrounded by another safety ring (fig 13b) made out of mild steel. We have thus surrounded the pressure die by a series of cylindrical vessels, so that the confining strength

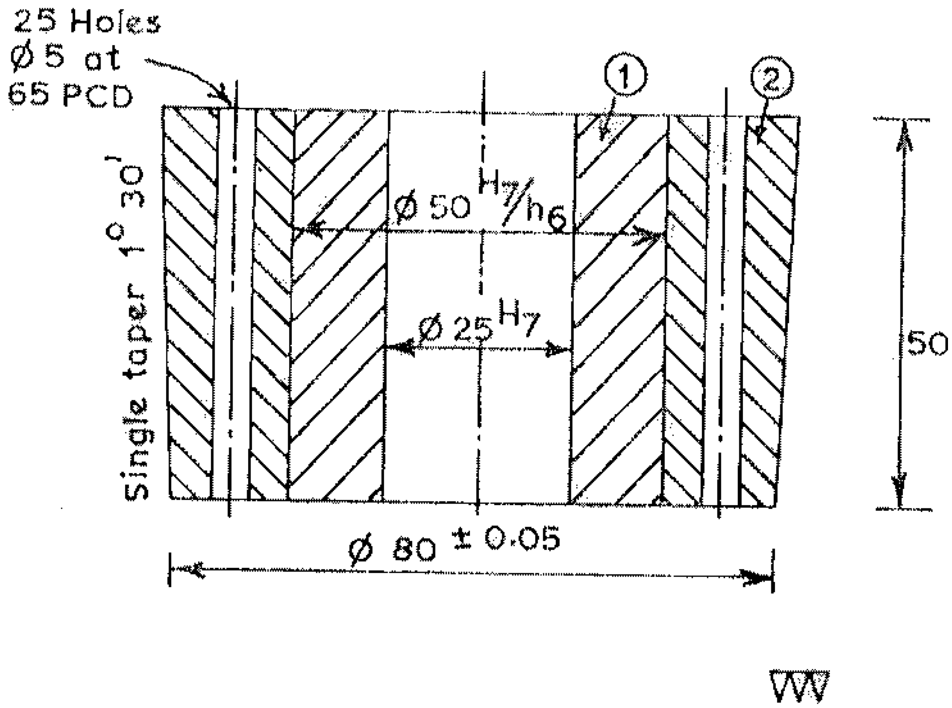
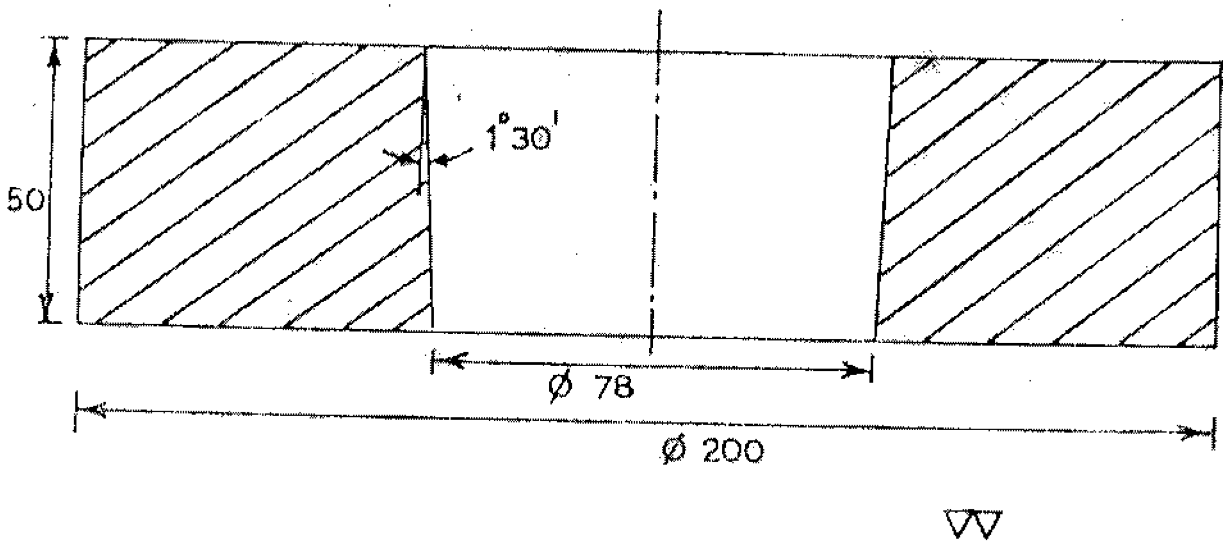


Figure 12: (1) Core of the pressure die
(2) First binding ring with cooling arrangement



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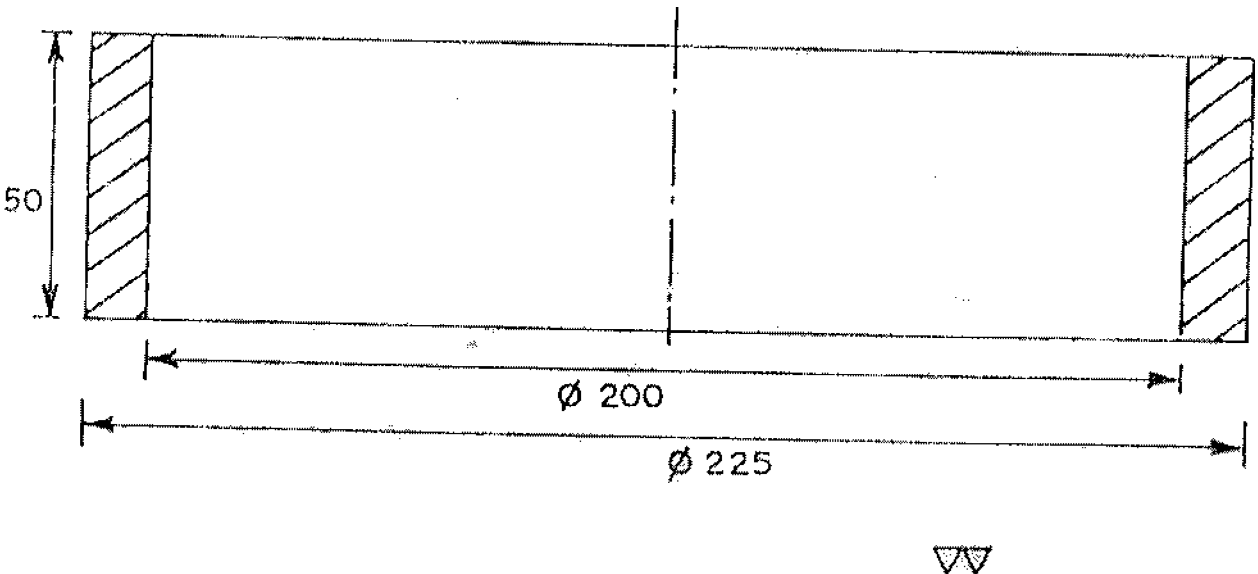


(b)

Figure 13: (a) Second binding ring

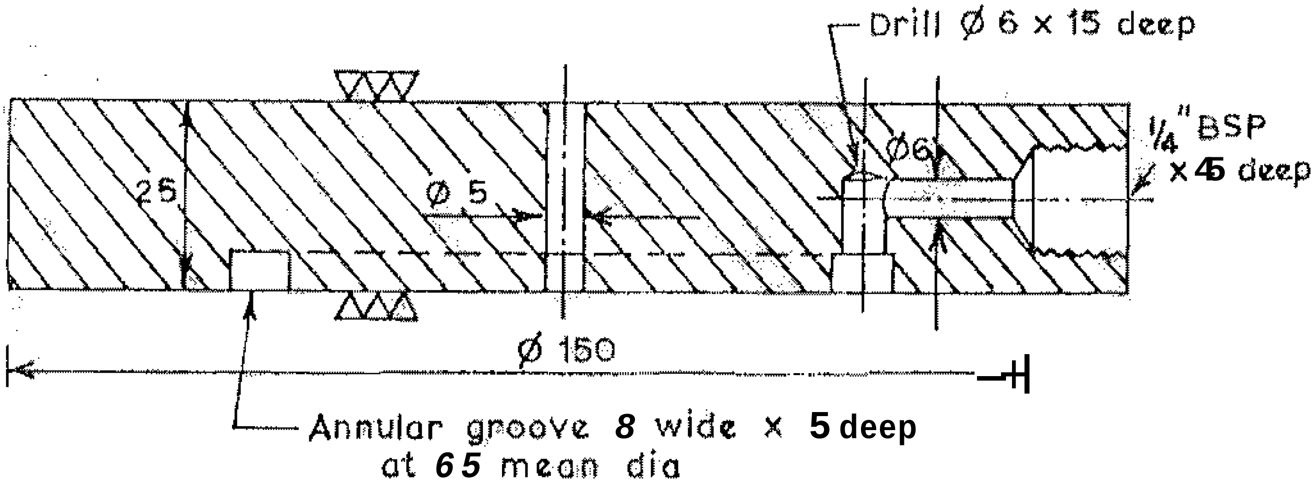
(b) Binding sleeve

e) Support pads

The Space between the pressure plate and the top platen is filled up with a series of pads. In direct contact with the pressure plate, there is a pad with an annular groove (fig 14a), which serves for the passage of water. At one point of the pad, the groove is made to join a hole drilled radially from the outer surface. A ¼" BSP threading done to the extreme end of the hole near the outer surface facilitates fixing of a nozzle to the groove pad which serves as the inlet for water circulation. This pad is insulated from the pressure plate using a thin layer of mica. Two more identical pads (fig 14b) rest on the groove pad. In addition, there is a pad affixed to the top platen (fig 15a). A feature of affixing this pad to the platen is that by using a combination of bakelite washer and nylon sleeves (fig 15b), it is insulated electrically from the main body of the press and therefore is a convenient point from which electrical connections can be taken easily through a threaded groove on one side.

3. Valves and Pressure Connections :-

A high pressure system generally consists of a variety of parts such as valves for control of pressure, tubing, seals, connectors and glands, etc. Since the line pressures



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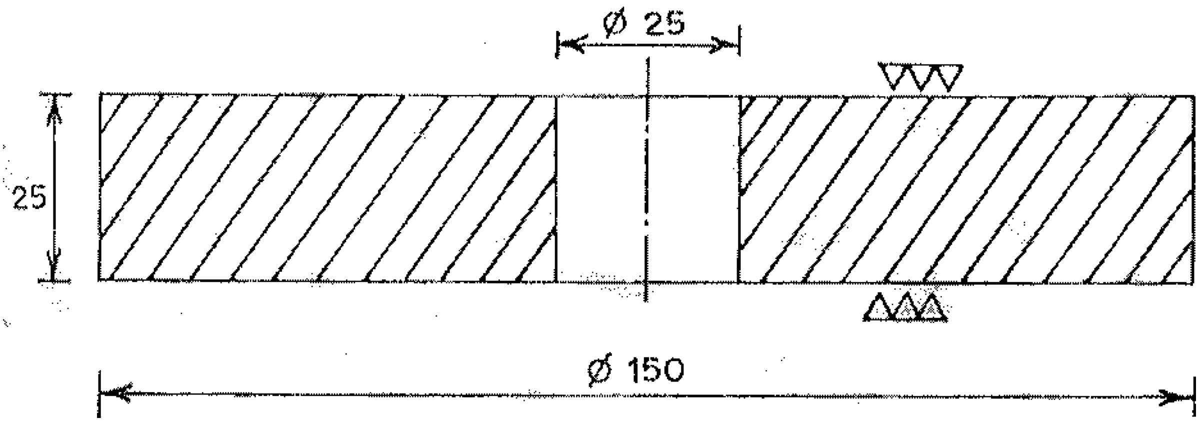
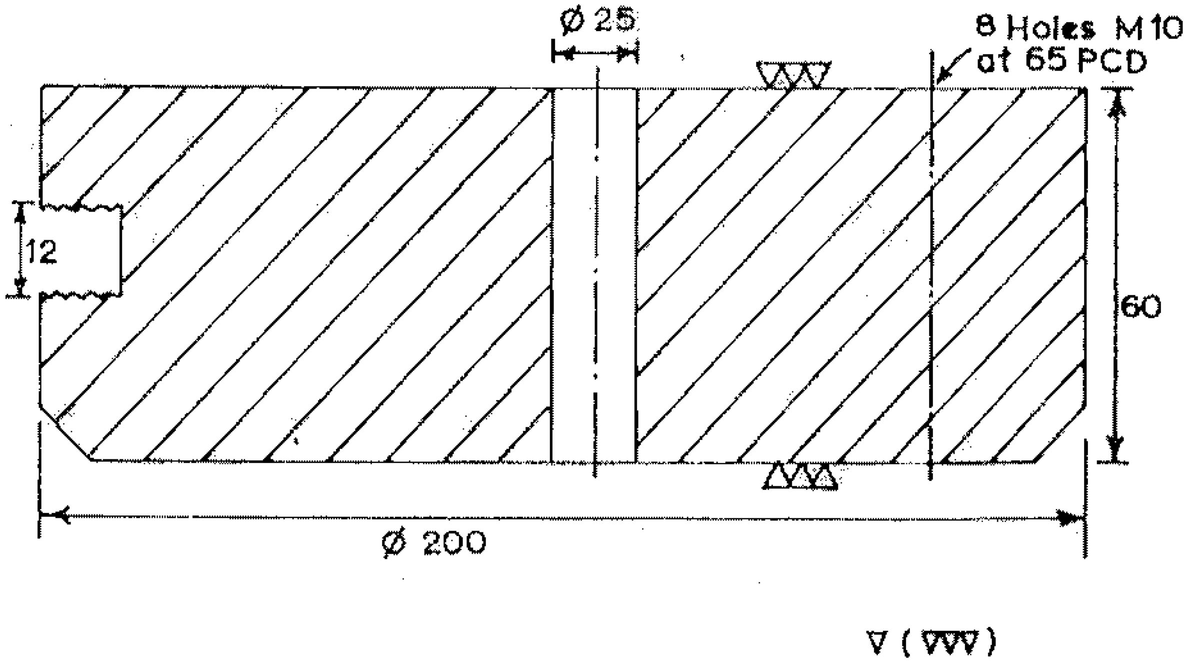


(b)

Figure 14: (a) Insulated groove pad

(b) Steel pads



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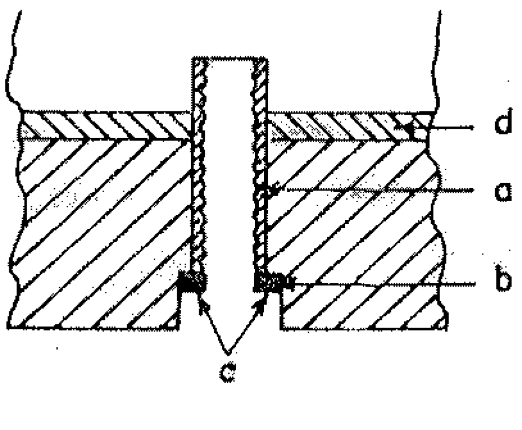


Figure 15: (a) Top platen pad
(b) Schematic diagram showing affixing of the pad to the top platen.

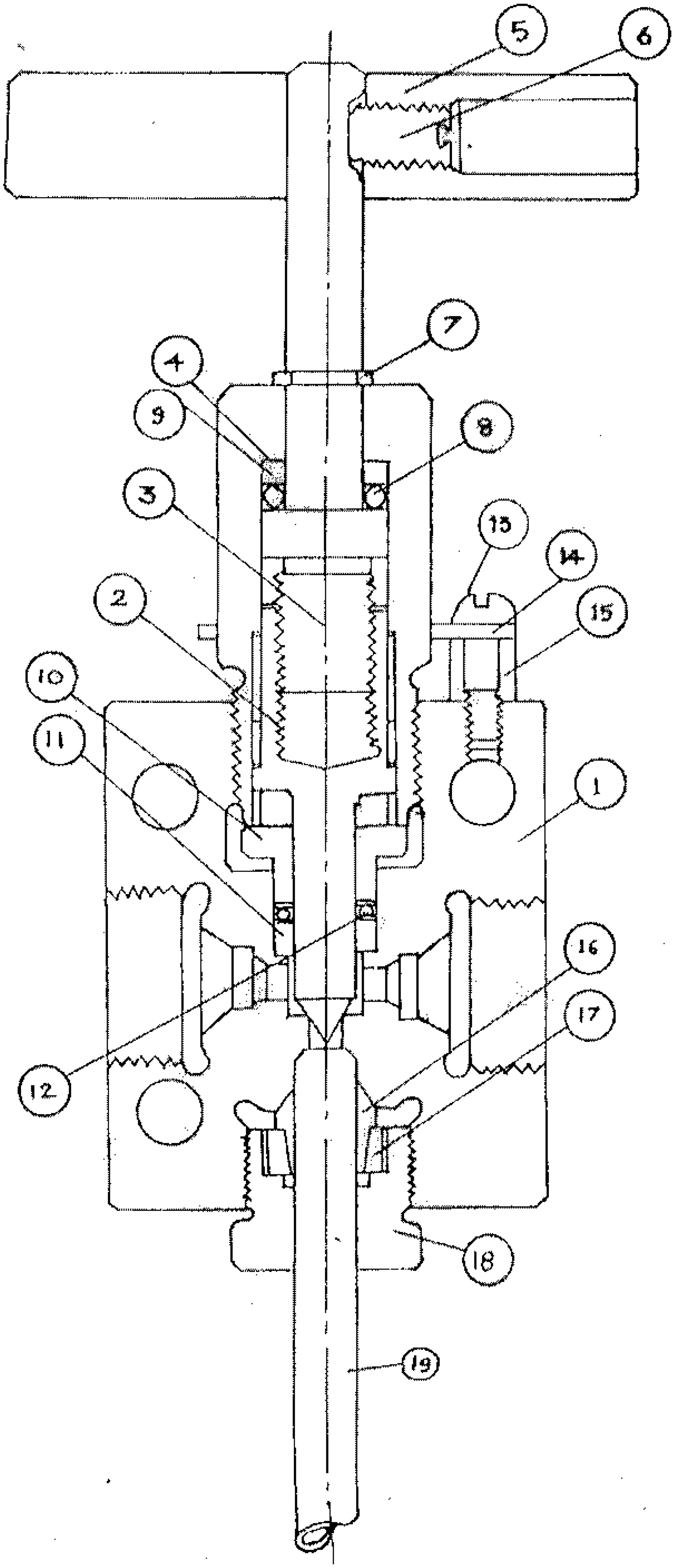
a Nylon sleeve b Bakelite washer
c Allen screw d Hylam sheet

Involved in our studies were rather low (not exceeding 10,000 psi – there being an intensification of about 64 due to the ratio of the piston surface diameter of the master ram), it was decided to design and fabricate our own pressure connections.

a) *Regulator* ***valve*** :- The function of the regulator valve is to control the outlet pressure at a predetermined level when the input pressure is varied. Since the requirement of a valve in the present set up involves low flow capacity, a needle valve [12] is used. The basic operation of this valve is the movement of the needle in the valve seat.. Therefore the design of the valve stem is extremely important. The most simple stem design is the 'solid stem'. However, it has a main disadvantage in that the valve seating gets easily damaged as the needle rotates inside the seating while closing and may therefore grind and gall.[12] To avoid this problem and a valve has been designed which is equipped with two pieces stem wherein the bottom part of the stem slides up and down in the packing without rotating, consequently the tip of the valve does not rotate on the seat, thus avoiding the grinding and galling of the stem and the seal parts. The schematic diagram of the 3-way (one connection on the pressure side) valve is shown in fig 16. The details of the valve body are shown in fig 17 which shows taper seal seatings on three sides with an M20 holes for the stem. The surfaces shown in the drawing

Fig 16: Schematic diagram of the 3-way valve.

1. Body
1. Stem
2. Screw
3. Cap
4. Handle
5. Grub Screw
6. Circlip
7. Balls
8. Ring
9. Gland
10. Gland ring
11. **'O'** ring
12. Round head screw
13. Lock plate
14. Spacer
15. Taper seal
16. Back-up ring
17. Nut cap
18. Tube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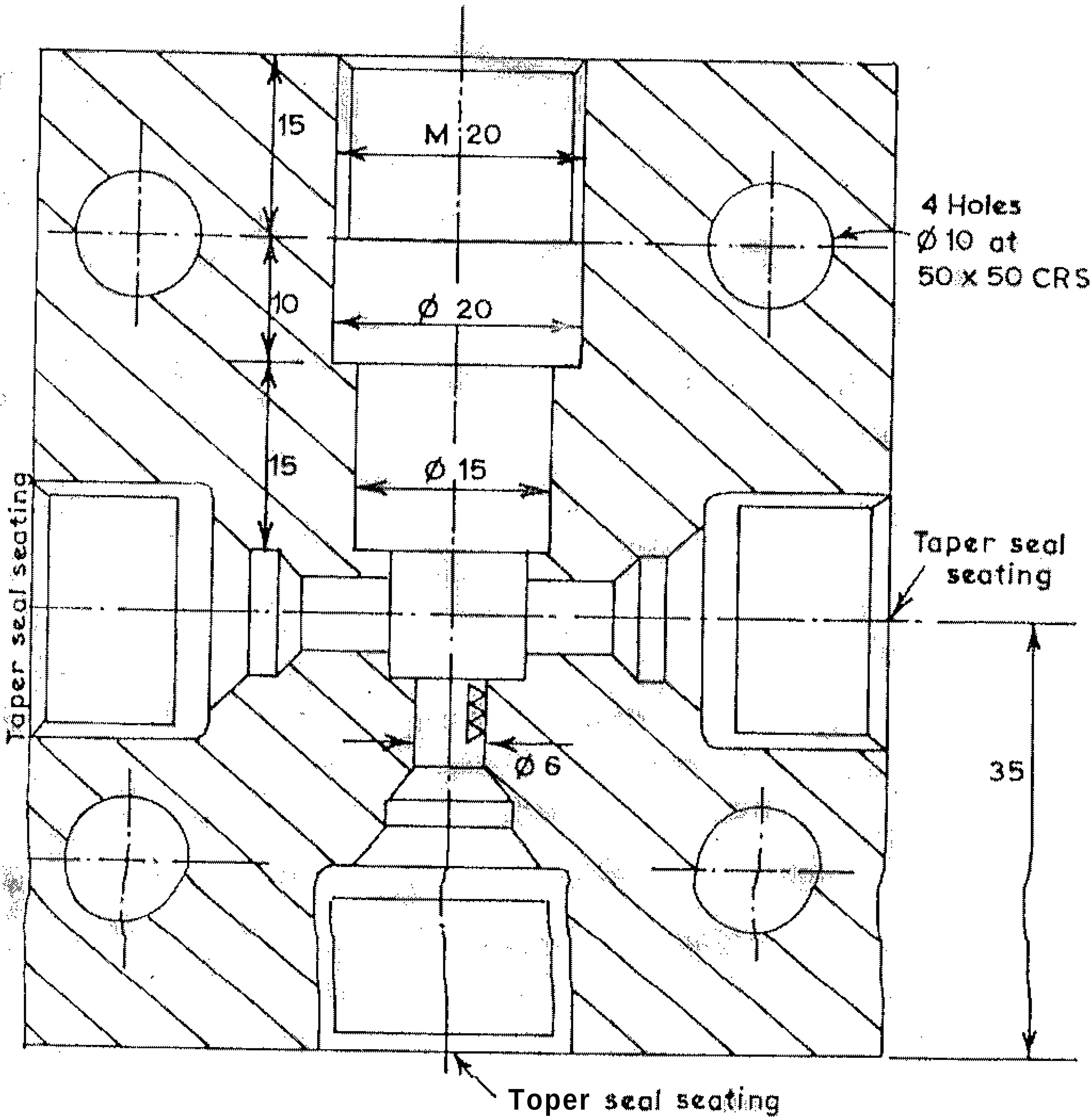
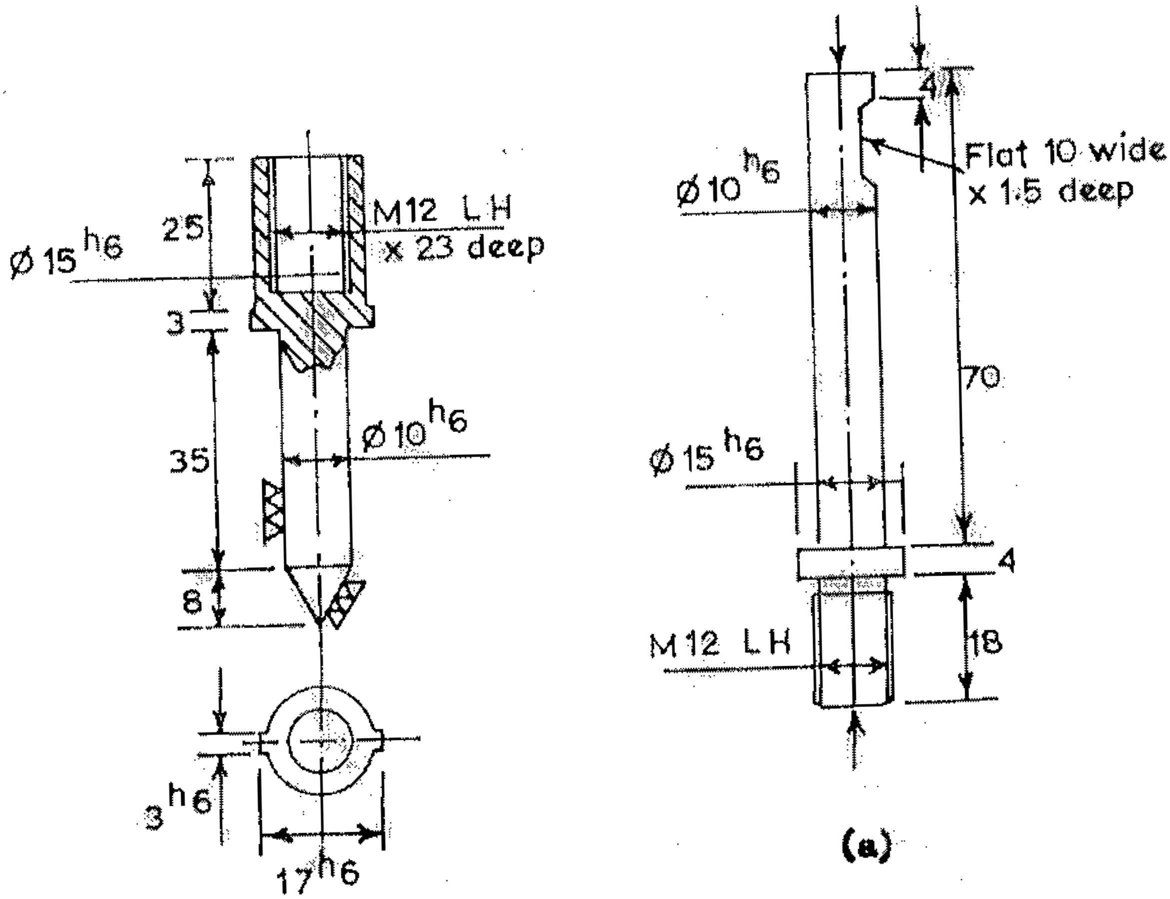


Figure 17: Body of the valve

(▽▽▽) are finished to a very high degree of smoothness to facilitate the sliding of the pressure tubing into the valve seating without any friction. The details of the stem is given in fig 18a. Only the tip of the stem is heat treated to RC 60 after grinding. **A 3** mm slot is cut near the top of the stem as shown in fig 18a by spark erosion technique. Into this slot are placed a metal ring and balls along with a Teflon 'O' ring (see fig 16). The top of the stem has a 23mm hole with left handed M12 threads into which a SS screw (fig 18b) can be screwed. The screw has a small slot at the top for facilititation of fixing aluminium handle (see fig 16). The stem along with the screw is held rigidly onto the body by means of stainless steel cap (fig 19) which also has two slots to match the slot in the stem. The entire assembly of the stem, screw and the cap, therefore serve the purpose of the stem moving translationally up and down for a rotatory movement of the handle. A lock up plate attached to the body of the valve (see fig 16) prevents the rotation of the cap with respect to the valve body.

b) ***Pressure*** connections :- The most widely used high pressure tubing connector is the union or the standard connection with a cone type, metal to metal seal. [12] Essentially any pressure connection consists of the following parts, the tubing, the sleeve, ferrule or collar, the gland nut and a mating part for the tubing. Considering that the



(b)

(a)

Figure 18: (a) Valve stem

(b) Details of the valve seating showing the tip and the slot.

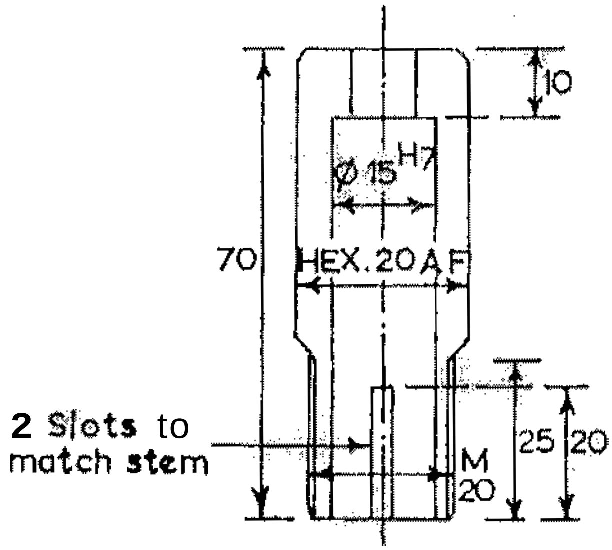


Figure 19: Steel cap for the valve stem

line pressure in our experiments are seldom expected to exceed 10,000 psi, what is normally called as 'bite' fitting or 'taper seal' fitting is used. This fitting works on the principle that the deformation of the ferrule or taper seal takes place under the force of the gland nut so that the seal bites into the tubing thus forming the sealing and holding the fitting in place.

The taper seals (fig 20) are made from SS 316 seamless tubes 15mm outer diameter and 10mm inner diameter. **A** 1° outer single taper is provided for the male part (fig 20b(1)) with a corresponding matching taper also provided in the mating female part (fig 20b(2)). In addition the tip of the taper seal is given a 73° (included) taper which serves to grip the tubing more efficiently on tightening of the gland. The gland itself (fig 20a) is made of SS 304. It also has hexagonal heads on the top to facilitate tightening. The taper seals as well as glands are deburred ultrasonically after machining. The details of the taper seal seating on the body of the valves are shown in fig 20c. It is seen that the body has a 74° included taper nearer the 20mm opening at the surface in addition to a 45° single taper to facilitate proper sealing of the taper seal on tightening of the gland as shown in fig 21a. Rings of two different sizes (fig 21b) are used for glands and thrust bearing purposes. A taper seal to ¼ " BSP adapter (fig 22) is used to connect the pressure tubing to both

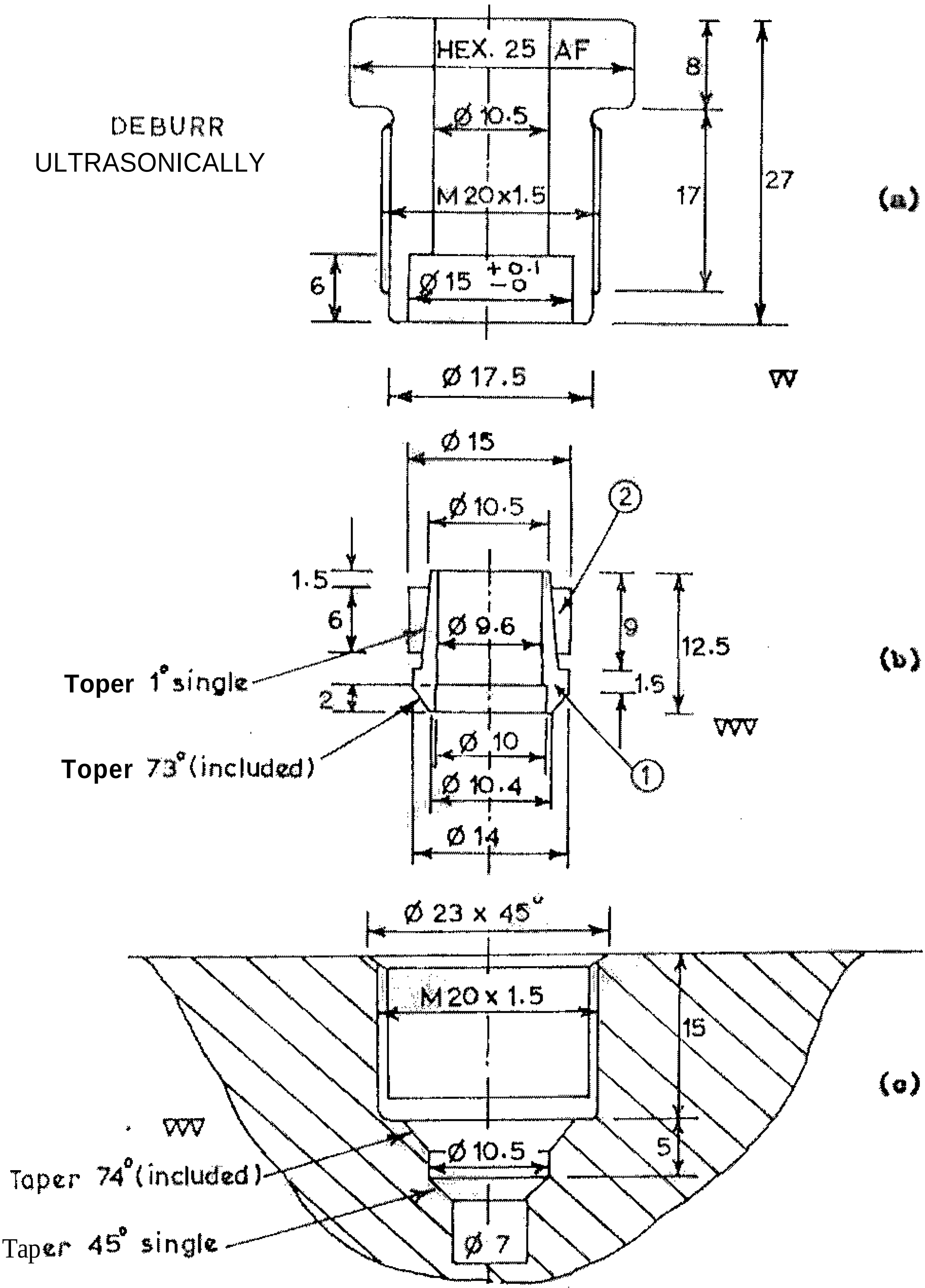
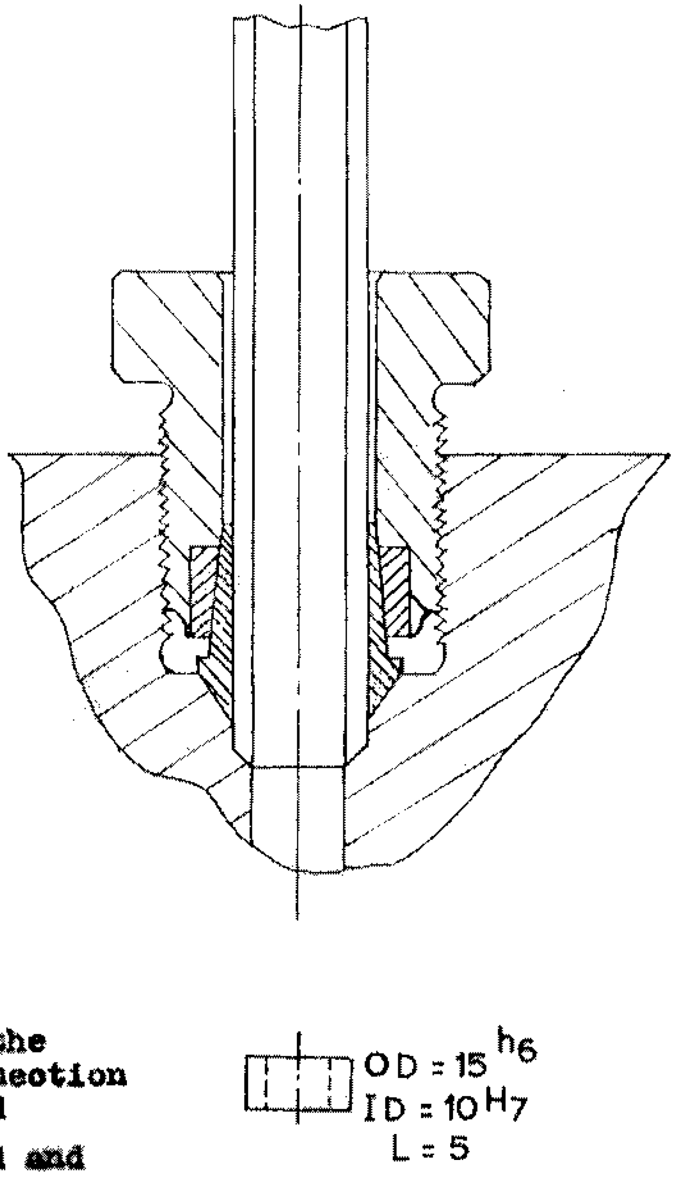


Figure 20: Details of the taper seal connections. (a) Gland
(b) Mating parts of the taper seal system
(c) Enlarged view of the section of the valve body.

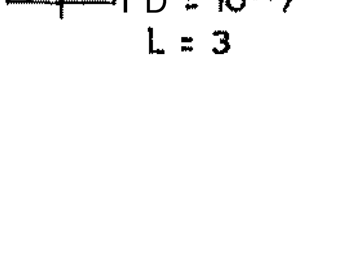


(a)

Figure 21

(a) Tightening of the taper seal connection using the gland

(b) Rings for gland and thrust bearing



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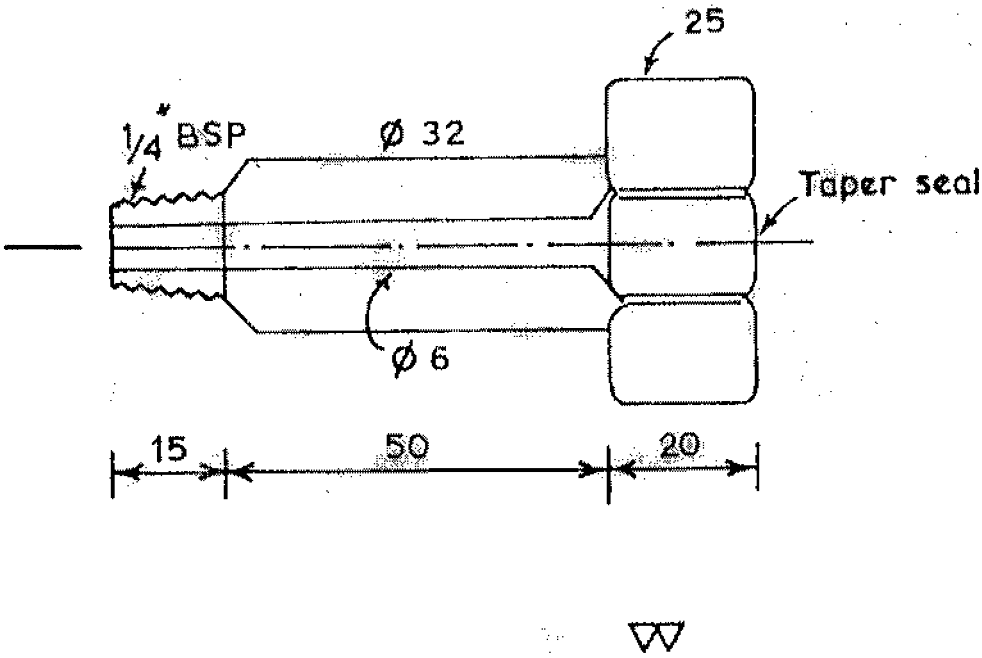

Figure 22: Taper seal to 1/4" BSP adapter

4. Working of the Press

Keeping in mind the various individual parts of the high pressure system and their desired function, a typical high pressure connection system whose schematic diagram is shown in fig 23 is conceived. V_1 to V_{12} in this diagram are three way valves of the type described earlier. T_1 to T_3 are the three way joints with taper seal seating for each of the pressure side. The basic design of the joints is exactly identical to that of the body of the valve (fig 17) and will not be discussed here. Two motor pumps MP_1 and MP_2 (WA Whitney : Model 700 – 690, USA) are used to pump hydraulic fluid into the master ram and end load ram respectively. The valves V_3 and V_4 control the pumping of the fluid to these chambers. A special feature of the hydraulic system (shown in fig 23) is that the pressure in both end load and master ram is varied and held at any desired value by finely controllable relief valves RV_1 and RV_2. This is achieved in the following manner. Initially with valves V_3 and V_4 open and V_{11} and V_{12} closed, the hydraulic oil is pumped from MP_2 through V_4 into the end load ram. The relief valve RV_1 is manipulated such that the end load pressure builds up to any desired value typically about 30 bars. This is necessary since the pressure plate should always be maintained under compression when the interior of the pressure plate is

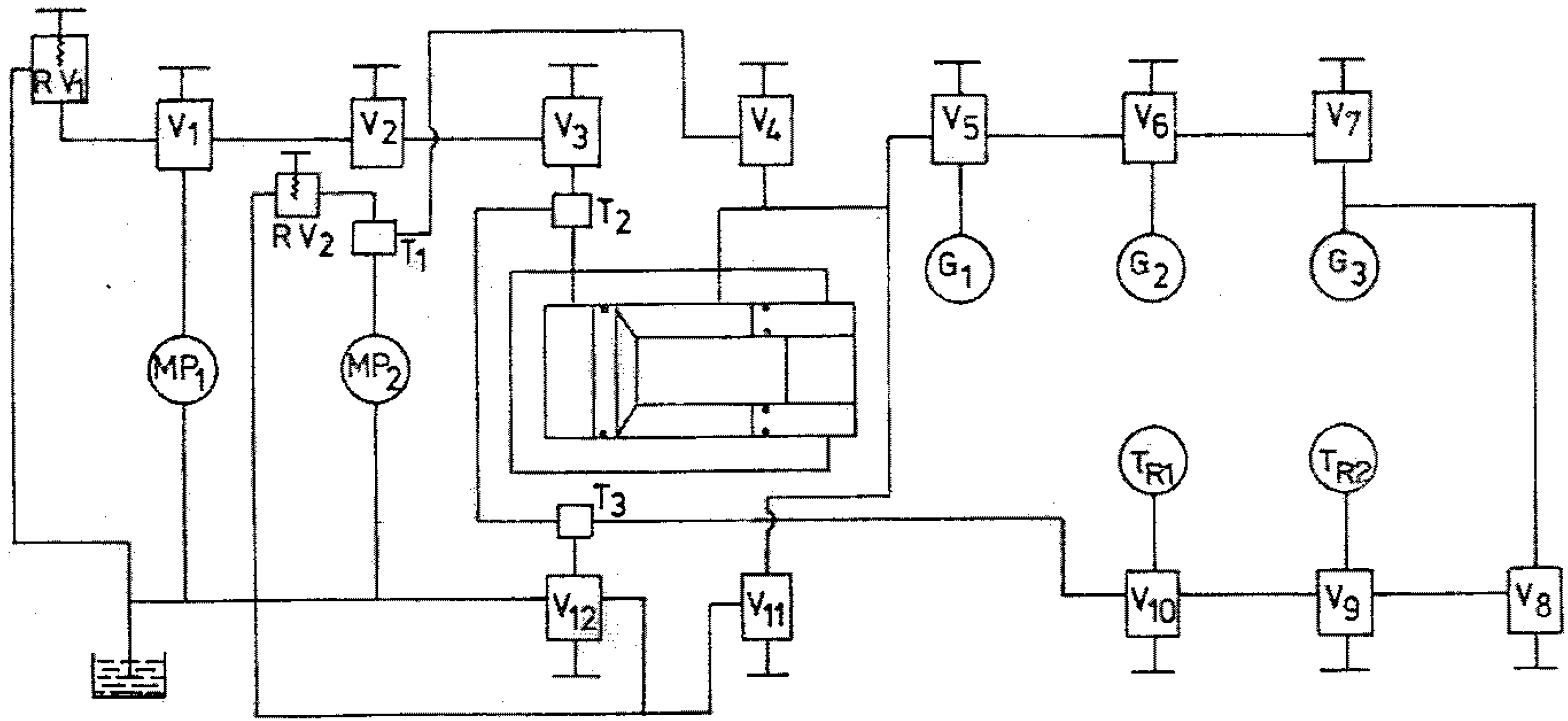

Figure 23: Schematic diagram of the high pressure plumbing system
MP_1 & MP_2 – Motor pumps, T_1, T_2 & T_3 – T joints
V_1 to V_{12} – non-rotating stem valve, RV_1–RV_2 – Relief valves
G_1, G_2 & G_3 – Gauges, TR_1 – TR_2 – Transducers

subjected to very high pressures. Keeping RV_1 locked in this position, MP_1 is now switched on, thereby injecting the fluid into the master ram. RV_2 is now operated so that the master ram starts moving up and presses the cell kept inside the pressure chamber. The end load pressure can be read by the gauges G_1 and G_2 whilst the master ram pressure by the two transducers TR_1 and TR_2. All the gauges as well as the transducers are provided with independent valves to isolate them in case the line pressure exceeds the full scale reading of the measuring instrument. The guage G_3 is situated such that it can measure either the master ram pressure or the end load pressure depending upon whether V_8 open. V_7 closed or V_7 open, V_8 closed respectively. In most of the experiments G_3 which is a CMM type Heise guage with a twin range of 0-1500 psi and 1500-3000 psi with a resolution of 2 psi was used to measure the master ram pressure. The master ram pressure is also read by the membrane type transducers TR_1 and TR_2 made by ISRO (type PT21UA 100 HQ S3 and 200 HQ S3) with a reading accuracy of 0.25% and linearity better than 0.2%. After fixing the required end load and master ram pressures all the valves are locked and the pumps were switched off. It was possible to retain the pressure without any appreciable drop during the course of experiments. Fine incremental changes of pressure could in this way be achieved by relief valves without recourse to constant use of a hand pump as is normally done. Thus, we have used the relief valves, which are normally thought to be useful only for controlling pressures under dynamic conditions, with great degree of success to maintain static pressures As fat as we are aware this is the only case wherein relief valves are being used for such a purpose .

5. Pressure Calibration

The pressure experienced by the cell when it is subjected to the force exerted by 1" diameter cell can be obtained by knowledge of the line pressures as shown by the gauges and transducers along with the ratio of the master ram area to the piston area. The possibility of frictional losses which could be different at different positions of the piston was also considered. A manganin resistance guage was used (details of the calibration will be given in the next section) to ensure that the movement of the piston was perfectly linear during its entire traverse in the pressure die. The absolute pressure as experienced by the sample in the cell which is naturally different from the line pressure, is dependent on the components used in the cell and the compression of the compression media. This true pressure was calibrated by using a suitable secondary standard as we shall describe in the next section.

6. Special Features of the Press

As already described, the working area of the pressure chamber has 1" diameter. This area is sufficient to insert a high pressure DTA cell specially designed for detecting liquid crystalline transitions (see part II of this chapter). A groove made on the top surface of the groove pad (fig 14a) facilitates to take out the thermocouple leads from the interior high pressure area to the zero pressure area without any pressure gradient in the manner described by Jayaraman et al. As already described, since the piston as well as the supporting pads have all got concentric holes it is possible by using a sutiable cell provided with sapphire windows (see chapter VII) to observe phase transitions by optical transmission technique. A third experiment which can be done using the same set up is the compressibility and volumetric studies by the 'piston displacement method'. This is done in the following way. Two steel tubes are welded on either side of the base of the connecting rod (fig 10b). The other ends of the tubes hold two steel platforms. As the piston moves within the pressure die these platforms press against the tip of the inner cylinder of a long stroke displacement meter. The outer cylinder of this instrument is anchored to the top platen so that the piston movement displaces the internal coaxial cylinder with respect to the outer one causing a change in the capacitance of the system which in turn is measured as a voltage digitally. Using this displacement meter, piston movement can be monitored to an accuracy of 10 microns out of a full scale reading of 5mm. Therefore, by having a typical Teflon cell containing the liquid crystal in the pressure chamber its volume change can be measured as a function of pressure. Thus, the 200 ton press described above incorporating simple piston cylinder device can be used for conducting a variety of high pressure experiments on liquid crystalline materials.

In the next section we shall describe a high pressure differential thermal analysis (DTA) cell using which most of the data presented in this thesis have been collected.

PART – II

Coaxial DTA cell for the study of liquid crystalline transitions at high pressure

As already stated, the design of the experimental set up for high pressure investigations of liquid crystals poses certain special problems which are not normally encountered in the study of solid state under pressure. DTA is a simple and convenient method of detecting first order phase transitions. It has been employed as a probe for the study of phase transitions in solids by Kennedy and Newton and also by Jayaraman et al. However the design of the cell used, in these experiments is not well suited for the study of liquid crystals for the following reasons (i) liquid crystalline transitions are characterized by extremely small heats of transition of the order 0.42 kJ/mol. It is therefore necessary to impress the thermocouple junction the sample because otherwise the weak signal will be undetectable; (ii) liquid crystalline compounds generally react with materials that are commonly used as sample containers.

The first high pressure DTA cell described in literature due to Tikhomirova et al who used it only to detect a solid-nematic transition. However, in their experiments the pressure transmitting fluid which was in direct contact with the sample contaminated the latter. The first accurate high pressure DTA experiments are due to Chandrashekhar et al who used a coaxial DTA cell designed by Reshamwala and Shashidhar. The author has used basically the same cell except that some modifications in dimensions have been made to suit the dimensions of the pressure plate used in this case. In the following we shall describe the cell in detail and give its performance. The importance of the working of the cell need not be stressed further than to say that all the data (except for the C-A transition in chapter V) were obtained by using this cell only.

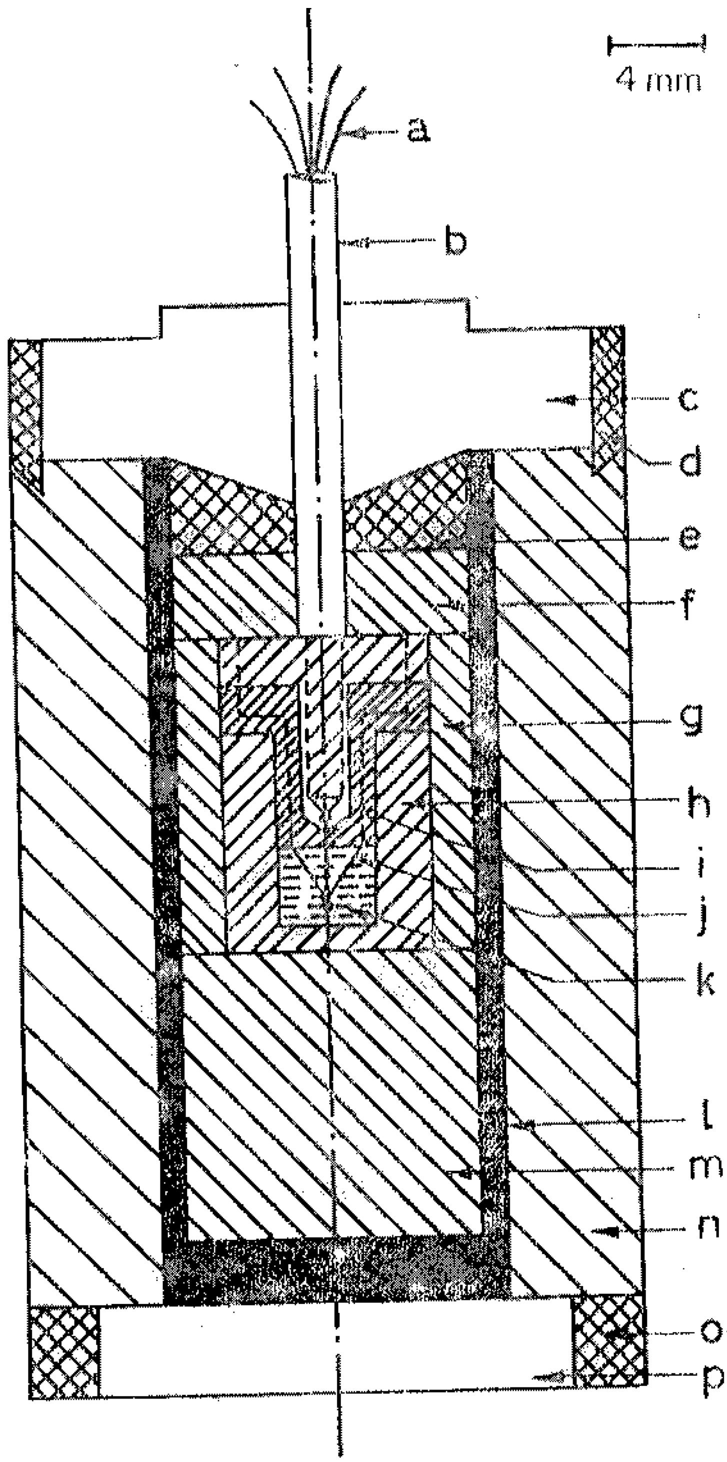

Figure 24: Schematic diagram of the DTA cell

The diagram of the DTA cell is shown in fig 24. The sample is contained in a capsule made of PTFE of normal grade (hereafter referred to as Teflon). The advantages of using Teflon are that it does not react with any of the liquid crystalline materials and that it can be pressure sealed easily. The capsule is provided with a Teflon cap (fig *25)* which is press fitted into the capsule after filling it with the sample in an inert atmosphere in the isotropic phase. Chrome1 and alumel wires are pierced through the cap and a junction is made so that this junction sits amidst the sample when the cap is fitted into the sample capsule. The Teflon cap has a central bore into which another Teflon plug can be fitted. The reference junction, also made from chromel-alumel wires, is pierced through this plug and positioned so that it is separated from the sample by about'lmm of Teflon. By this we have achieved two important things. (i) since the sample thermocouple junction is immersed in the substance, we can detect even faint first order transitions, and (ii) the reference and sample junctions, both being surrounded by Teflon, are heated almost to the same extent during the experiment. Owing to the difference in thermal conductivities of the sample and Teflon, however, there is always a difference in temperature of about 2°C between the sample and reference junctions. This difference changes with increase of temperature, it does cause a problem in that a sharp transition might be missed during the off setting of the recorder pen. This drift was minimized as follows. A relatively fast DTA run (heating rate 8-12°C/mt) was taken

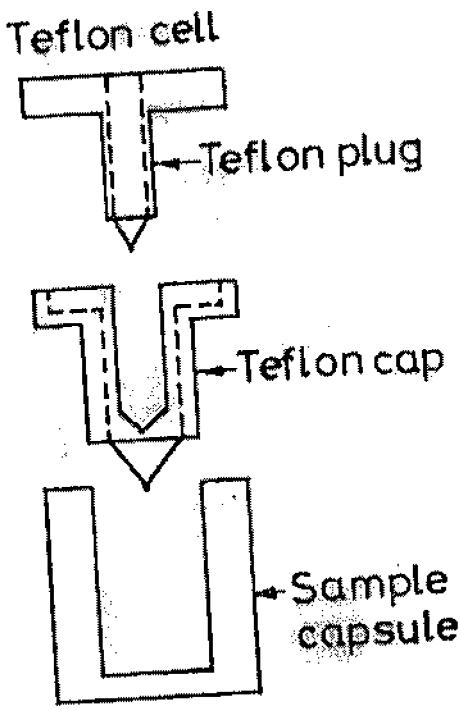

Figure 25: Details of the sample capsule

to roughly locate the transition temperature. After cooling of the sample, the temperature was stabilized at a value just below the transition temperature. **A** second run, this time at a much slower heating rate (0.5 to 2^{o}C/mt) recorded the transition more accurately, the baseline now obtained being almost horizontal.

Another problem to be borne in mind is the possibility that the phase transition in Teflon may affect the observation. Teflon is known to exhibit two polymorphic transitions at atmospheric pressure, at 20oC and 30oC. A third crystalline transition for Teflon was first observed by Bridgman (22) at high pressure at room temperature. This has been subsequently confirmed and studied in greater detail by Weir (23) and also by Bee Croft and Swenson. (24) All these transitions occur at temperature lower than and pressure higher than those involved in our studies and therefore could not affected the results discussed in this thesis. The crystalline transitions in Teflon are also pressure dependant but a knowledge of their phase diagram (23) helps to preclude their interference with the liquid crystalline transitions.

Considering that the use of a liquid transmitting media in impracticable, (18) the obvious pressure transmitting media is talc which is known to be hydrostatic to 85-90%. The sample capsule is suitably surrounded on all sides by talc as shown in figure 24, so that the pressure gradients are minimized. The entire unit slides into a cylindrical sleeve of graphite which acts as the heater. The temperature profile in such a graphite heater used in conjunction with a piston cylinder device will, in general, have a non uniform temperature zone near the ends and a constant temperature zone in the region of the geometrical centre of the heater. (25) By keeping a number of probes along the axis of the graphite cylinder, it was about 8mm long, the temperature variation within this zone being less than 0.25C. The length of the zone diminished for temperatures above 250C. The heights of the top talc disc and the bottom talc pad are now so adjusted that the sample is well inside the constant temperature zone. The thermocouple wires are brought out from the high pressure area to the atmospheric pressure region as described earlier (13). Briefly, the thermocouple leads are threaded through a four hole ceramic tube and connected to the appropriate wires coming out of the cap of the Teflon cell by spot welding.

The heater assembly is then inserted inside a thick talc body. The outer diameter of this talc cylinder is machined so that it can just slide inside the 25m hole of the pressure plate. A pyrophyllite cone kept on the talc disc has a cone angle exactly matching the cone angle of the stainless steel piece kept on the top of it (see parts c and e in figure 24). When pressure is applied the stainless steel cone which is touching the top of the graphite heater comes into contact with the groove pad (figure14a), whereas the bottom of the graphite heater is connected through a bottom stainless steel (also enclosed by a pryophyllite ring) to the pressure plate. The groove pad and the pressure plate being insulated from each other, server as convenient ends through which current can be passed through the graphite heater. The heater is powered by a high current low voltage transformer which has a maximum output capacity of 200 Amperes at 20 volts. The input voltage on the primary side of the transformer can be varied continuously with the help of a variable speed gear box which is coupled to the autotransformer. This permits to select any desired rate of heating.

The difference in temperature between the sample and reference (T) was fed to a Keithley nanovolt amplifier (model 140) whose output was displayed on the Y axis of a Ricksen-Denshi X-Y recorder (model F-43). The sample temperature was fed to the X axis of the recorder and also was simultaneously read on a digital mill voltmeter. An amplification of 1000 was used to record most of the liquid crystalline transitions. The sensitivity of the Y axis was suitably reduced to record the solid-liquid crystal transition. In series with the differential T, a zero suppression unit was used to suppress the thermocouple signal with a resolution of 1 microvolt. The variation of thermo e.m.f with pressure is negligible in the pressure ranges of our interest. (26)

Pressure calibration and measurement

The linearity of the pressure measurements was ensured by measuring the resistance variation with pressure of a standard managanin gauge (27, 13) immersed in silicone oil filling the sample capsule. It was observed that in the pressure range up to about 8 kbars the variation was linear.

The line pressures were measured by a calibrated transducer (type PT 21 UA made by ISRO) reading to an accuracy of 0.2%. However, this is not the true pressure experienced by the sample. The true pressure is less than the line pressure, the correction being dependant upon the thickness and the grade of the Teflon used and also in the inside geometry of the cell. The phase diagram of freshly distilled CC14 was obtained and on comparison with that of Bridgman's (1) it was noted that our data agreed with those of the Bridgman's within 2 to 3% over the entire range of pressures investigated. Also to confirm the accuracy of the true pressures, the phase diagram of PAA (used as a secondary standard) was studied and this was in agreement with those of others (28, 29, 17) (see chapter III). Thus, we can take the accuracy of our absolute pressure measurement to be of the order of 2% over the entire range of our study.

Table I

Components of the press with the materials used and their hardness

Component	Material	Hardness
1. Platens (upper and lower)	Mild Steel	-----
2. Master Cylinder	EN – 24	55 – 60 RC
3. Tie rods	EN – 24	55 – 60 RC
4. Nuts for the tie rods	EN – 24	55 – 60 RC
5. End load ram	EN – 24	55 – 55 RC
6. End load support plate	EN – 24	50 – 55 RC
7. Master ram	EN – 24	55 – 60 RC
8. Static seal plate	EN – 24	55 – 60 RC
9. Support pads	EN – 24	55 – 60 RC
10. High pressure piston	HC – HC	60 – 65 RC
11. Pressure die	HC – HC	58 – 60 RC
12. Binding rings and sleeves	EN – 24	55 – 60 RC
13. Groove pad	EN – 24	50 – 55 RC
14. Valve body	Stainless steel	-----
15. Valve stem	EN – 24	45 – 50 RC
16. Taper seals	Stainless steel	-----

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