OPERATOR MANUAL

HIGH-PRESSURE HIGH-TEMPERATURE TESTING MACHINE

Serial No 5

PATERSON INSTRUMENTS PTY LTD

CANBERRA AUSTRALIA

ACN 008 644 273

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# SAFETY INFORMATION

**IMPORTANT NOTICE:** This machine operates with gas under high pressure. While every reasonable precaution has been taken to incorporate conservative safety features in the design and manufacture of the machine, it is the operator's responsibility to observe safe practices, as outlined in this manual. The manufacturers do not accept any responsibility for loss or injury arising out of the use of the machine. The safety features incorporated in the machine are the following:

Pressure vessel design

The main pressure vessel and intensifier bodies are made from Assab Orvar 8407 Supreme steel (Premium AISI H13, W.-Nr 1.2344), chosen for its through-hardening and toughness. They are heat treated to Brinell hardness 380±20. The hardness and freedom from flaws have been checked and certified by a registered testing laboratory. This hardness level has been chosen for optimum balance between maximum ductility and sufficient strength to avoid undue dimensional changes at maximum pressure. These parts and the associated high-pressure plumbing have been tested initially to about 750 MPa, a pressure that leads to some yielding at the inner bores, with consequent auto-frettage to improve the pressure performance.

Safety sleeves

As a second protective measure, the main pressure vessel and intensifier bodies are both fitted with shrunk-on safety sleeves made of mild steel (Assab Hollow Bar Grade 2C). In the unlikely event of a brittle failure of a main body, the safety sleeve should stretch plastically while restraining the fractured parts and allowing the gas to escape without propelling these parts outwards. The safety sleeve also provides some additional support for the pressure, thus reducing the stresses in the main bodies.

## Safety housing

A third level of protection is provided by the steel housing of the machine itself. This housing is constructed of mild steel plate and angles. Its steel walls have a minimum thickness of 15 mm. They consist of three layers for maximal attenuation of any blast waves that might occur in the unlikely event of a major failure. This thickness is also more than adequate to constrain any parts that could be accelerated by escaping gas, which itself is vented to the lower rear of the housing through a labyrinth.

## Door locks

The access doors of the housing are fitted with fail-safe door-bolts. These maintain the doors locked in the event of power failure, and they are controlled to lock whenever the gas pressure exceeds a threshold value, which is set at 30 MPa when the machine is shipped. This level is chosen so that the operation of the gas-booster pump or small plumbing leaks can be conveniently checked on while the pressure is still near to bottle pressure, while preventing risky inspections at higher pressures. It is very important for safety that this setting not be changed. Arrangements have been incorporated which enable leaks at O-ring seals to be detected with bubblers during operation or leaks in the plumbing to be detected after depressurization by the extrusion of grease from escape holes.

## Valve handles

Although the high-pressure valves (NOVA Inc.) are fitted with safety stems that are constrained from ejection by an enlarged head within the body, the stems have, in turn, been fitted with stem extensions having an enlarged part that cannot pass through the holes in the housing through which the valves are operated. Newly added or changed valve extensions (e.g., HIP Inc.) are equipped in a similar fashion.

## Rupture disks

Rupture disks have been fitted to prevent the inadvertent exceeding of safe pressures in the various parts of the high pressure system, as follows:

* Main pressure vessel, intensifier and associated plumbing-700 MPa
* Gas booster pump outlet-200 MPa
* Gas booster pump inlet and plumbing connected to gas bottle-30 MPa
* Oil pump to intensifier-80 MPa (located inside oil tank)

In view of possible variability in rupture disks, care should be taken not to exceed pressures of about 90% of the rupture disk ratings in the respective parts of the pressure system in order to prevent inadvertent failure of the disks.

# SPECIFICATIONS

|  |  |
| --- | --- |
| Confining pressure | 500 MPa in Argon |
| Free volume | 0.21 liters with solid, zirconia-insulated furnace and internal load cell fitted |
| Available energy | 125 kJ in adiabatic expansion from 500 MPa with solid, zirconia-insulated furnace and internal load cell fitted |
| Temperature | 1600 K (+) |
| Axial force | ±100 kN |
| Axial displacement | 30 mm on piston (total range of actuator 50 mm); microswitch limits set at positions 3.0 and 49.4 mm |
| Axial displacement rate | Max: 0.5 mm s-1; min < 0.01 μm s-1, in 0.1 μm steps |
| Pore pressure | 500 MPa |
| Volumometer displacement | 1900 mm3 (7.00 mm diameter x 50 mm stroke); microswitch limits at each end of travel |

Table 1: Paterson Rig Specifications

# GAS PRESSURE SYSTEM

The Paterson apparatus operates using Argon gas as confining pressure medium. The flow chart indicates the high pressure plumbing connections to the valves. The color coding is as follows:

|  |  |
| --- | --- |
| yellow | argon bottle pressure |
| orange | argon gas booster pressure |
| red | argon intensifier/pressure vessel pressure |
| blue | pore fluid system pressure |
| green | intensifier oil pressure |

Table 2: Color Coding Pressure Flow Chart

Before applying pressure, press the **green pushbutton** on the right-hand instrument panel to energize the instruments, having first, if necessary, switched on the **main power supply** using the mains switch box below the desk.

Unless the pore pressure system is to be used (see under Pore Pressure System), the upper "**pore fluid interconnect**" valve should he **closed** throughout the following procedure.

## Flushing the system

After closing the pressure vessel, it is important to flush out the remaining air so as to minimize water condensation and oxidation of furnace windings. For this purpose, the "**dump**" valve should be closed and the other four main gas valves opened *(if the pore fluid system is to be flushed as well, the pore fluid release valve is also closed and two pore fluid interconnect valves opened).* The pressure system is then charged with gas at bottle pressure. After closing the gas bottle, the gas can then be released by opening the "dump" valve. Two or three such **flushings** should normally be sufficient.

An alternative procedure for flushing can be used if there is sufficient Argon gas pressure remaining in the intensifier unit (i.e., 20MPa or higher). In this case, the pressure vessel can be flushed by controlled release of gas in to the vessel via the “**intensifier out**” valve (release valve closed).

**Note:** make sure that the vessel pressure does not exceed 20MPa for flushing.

## First Pumping Stage (0-7bars)

The first stage of pressurization consists of filling the apparatus with gas at bottle pressure. Industrial Argon (300 size bottle) is used and attached via a reduction manifold to the apparatus pressure system.

|  |  |
| --- | --- |
| **Valves** | **Status** |
| gas bottle | open |
| “dump” | closed |
| “inlet” | open |
| “intensifier in” | open |
| “intensifier out” | open |
| “release” | open |

Table 3: Valve Positions First pumping Stage

The gas is introduced simply by opening the gas bottle valve. This valve is then left **open** for further gas supply during the second pumping stage.

## Second Pumping Stage (7-1000bars)

The second stage consists of pre-charging the intensifier and raising the pressure in the main pressure vessel within the limit of the Haskel gas booster pump.

|  |  |
| --- | --- |
| **Valves** | **Status** |
| gas bottle | open |
| “dump” | closed |
| “inlet” | open |
| “intensifier in” | open |
| “intensifier out” | open |
| “release” | closed |

At this stage it is important to check that the back and top doors are closed, as there is an interlock connection between doors and pumps which is effective at pressures above about 30 MPa. Above this pressure, the pumps will not operate unless all doors are closed. However, the pumps can be operated with doors open at lower pressure in the event of special checking or inspection requirements but extreme caution is necessary if use is made of this facility and in no circumstances should any servicing of the pressure system be attempted while pressure is up; if any tightening of joints in the pressure system is required, release all pressure first.

After closing the gas release valve as indicated in the above valve settings, the gas booster pump is operated by switching to "**manual**" or "**automatic**" position of the "**gas booster**" switch. The rate of pumping and the limit of pressure can be regulated by adjustment to the air regulator situated under the desk. In "**manual**" mode, the pump will continue operating until switched off or until the gas pressure is balanced by the air pressure. In the "automatic" mode, assuming that the air pressure is set sufficiently high, the pump will operate up to the level set using the PICS control unit.

At the beginning of this stage, the intensifier piston should be positioned so as to provide sufficient stroke to achieve the desired pressure if this pressure is above the range of the gas booster. The best position will be established in the light of experience. Larger amounts of gas than necessary should not be taken into the system in order to minimize the stored energy in the system and not to waste gas unnecessarily. The procedure for operating the intensifier is set out in the next section.

The pressure range of the gas booster is approximately **150 times** the air pressure, that is, about **100 MPa** if 700 kPa air pressure is available. Above this pressure, or in the case of fine adjustment of pressure being required, the intensifier is used.

## Third Pumping Stage (1000-5000bars)

The third stage consists of operating the **10:1** **intensifier**, the low-pressure side of which is connected to a **Haskel oil pump** as shown in the green section of the plumbing diagram on the front of the machine. The oil pressure is indicated by the Bourdon gauge at the bottom of the right-hand instrument panel. It is calibrated to read in bars. Since **10 bars = 1 MPa** and the intensifier ratio is 10:1, the readings also correspond to the gas pressure in MPa, within error limits set by intensifier piston friction and gauge inaccuracy. The Bourdon gauge therefore serves as an approximate indicator for the gas pressure in the intensifier when the "intensifier out" valve is closed. It also serves as an independent approximate check on the gas pressure in the main vessel if the "intensifier out" valve is open.

|  |  |
| --- | --- |
| **Valves** | **Status** |
| gas bottle | open |
| “dump” | closed |
| “inlet” | closed |
| “intensifier in” | open |
| “intensifier out” | open |
| “release” | closed |

Before operating the intensifier to pressures above **100 MPa** it is very important to **close** the "**intensifier in**" valve, as indicated in the above settings, in order to protect the gas booster pump from overpressure. In the event of omitting to close this valve, the 200 MPa rupture disk also serves to protect the gas booster pump. The closing of the "**inlet**" valve is not essential but avoids wastage of gas in case any wear in the piston seals of the gas booster leads to leakage of gas from the bottle to atmosphere while the pump is not in use. If it is not expected that a further supply of gas will be required from the bottle, and assuming that the gas suppliers do not wish used gas to be returned to the bottle, the gas bottle valve can be closed at this point and the "**dump**" valve opened.

It is important to leave a free passage open for gas to be released from the apparatus, either to atmosphere or back into the bottle, in case a rapid reduction of pressure is decided upon (for example, if a specimen jacket leak commences and it is thought that this might become dramatic unless the confining pressure is reduced fairly quickly). In the event that gas is inadvertently released from high pressure by opening the "**gas release**" valve when both gas bottle and "**dump**" valves are closed, the **low pressure** lines are protected by a 30 MPa rupture disk.

With the valves set as above, the oil pump can now be operated in either manual or automatic mode by the "**intensifier**" switch on the instrument panel.

In "**manual**" mode, the pump will continue pumping until the oil pressure balances the air pressure in the ratio of approximately **100:1**; this balance is attained either by developing a sufficiently high gas pressure or by the intensifier piston reaching the end of its stroke and being supported by the intensifier body. The air pressure and hence a limit to the gas pressure can be set by means of the appropriate air control valve situated under the desk.

In the "automatic" mode, the same situation applies, with the addition that the pump will be stopped when the gas pressure reaches the **limit** set in the **PICS** unit. The same limit settings apply to both gas booster and intensifier operation. If the pressure drops below the set-point by more than the half the hysteresis setting, the pump will come into operation again.

Note: if the intensifier reaches the end of its stroke before the set point is reached, the control is ineffective. In this case, the air-driven pump will continue to operate until it stalls, the extra oil pressure being supported at the end of the cylinder. In order not to burst the **80 MPa** rupture disk in the oil circuit, it is important never to set the air pressure above **700 kPa** on the air control unit for the intensifier. If the pressure rises above the set-point by more than half the hysteresis setting, the oil bleed valve will open and reduce the pressure to the limit setting. In order to minimize overshoot in the pressure cycling under automatic control or otherwise to provide additional protection, it may be found useful to set the air pressure to the minimum value needed for the pump to operate to the set pressure.

The bleed valve is located behind the right-hand instrument panel and should be set to give a convenient rate of pressure release. The same setting should work over most of the pressure range and so it should not normally be necessary to readjust this valve. It is important that it not be left completely closed.

Since the pressure vessel is protected by a **700 MPa** rupture disk, the gas pressure should not be taken beyond 600 MPa in normal usage. Routine working range is **500 MPa**.

When using a furnace, unnecessary pumping can be avoided by allowing for the increase in pressure due to the heating when doing the initial pumping. Experience will show to what level it is appropriate to raise the pressure before raising the temperature in order that the pressure will finally come to the desired working level. With the intensifier switch in "**automatic**" mode, the bleed function will, in any case, prevent the pressure rising above the **PICS set-point**, provided that the intensifier has enough stroke left in the "**out**" direction. In "**manual**" control, the pressure should be watched carefully during heating and if it becomes excessive it should be reduced by opening the "oil release" valve so as to back off the intensifier (it is important that the "**intensifier out**" valve be kept open for this purpose). If the "**intensifier out**" valve is not open or if the intensifier has been backed off to the end of its stroke, the gas pressure in the pressure vessel must be further reduced by opening the "**gas release**" valve. Thus in "**manual**" control, it is very important not to leave the machine unattended during the heating stage but to monitor the pressure/temperature progression continually. If the pressure should inadvertently rise excessively, the bleed function will automatically release oil from the intensifier when the pressure rises to a limit set under alarm 1 in the confining pressure meter; this limit is set at 600 MPa before shipping.

Once the working conditions have been established, the intensifier can be left connected to the pressure vessel with the "**intensifier out**" valve open and the oil pump switch left on "**automatic**" so that any leaks are automatically compensated for. Alternatively, if the leak rate is low enough for the experiment involved and it is preferred to avoid occasional small steps in the pressure record, the "**intensifier out**" valve can be closed at this stage and the intensifier switch turned to "**off**". If desired, the pressure in the intensifier can then also be reduced by backing it off using the "**oil release**" valve.

The position of the intensifier piston is indicated by the level of the oil in the sight glass to the right of the Bourdon pressure gauge. The extreme positions can be established by pumping to these when the machine is not pressurized. If the intensifier reaches the end of its inward stroke before the desired pressure level is reached, it can be restroked as set out in the next section. With a furnace having solid zirconia insulation, it will be found that approximately half a stroke of the intensifier is needed to raise the pressure from 100 MPa to 300 MPa.

## Restroking the intensifier

In order to re-stroke the intensifier without affecting the pressure in the main pressure vessel, the "**intensifier out**" valve is closed, the intensifier switch turned to "**off**", and the "**oil release**" valve opened until the desired position of the intensifier is reached, whereupon the "**oil release**" valve is closed again. Meanwhile, when the pressure indicated on the Bourdon gauge is below 100 MPa, the "**intensifier in**" and "**inlet**" valves are opened and, if necessary, the "**dump**" valve is closed and the gas bottle valve opened. Then the gas booster is operated until the intensifier is again fully charged to about 100 MPa. After closing the "intensifier in" valve and, if wished, the "inlet" valve, the oil pump is next operated via the manual switch to raise the gas pressure in the intensifier. This pressure is monitored with the Bourdon pressure gauge and, when the pressure level (reading bars as MPa) becomes equal to that indicated by the "confining pressure" digital gauge, the "intensifier out" valve is re-opened and pumping of the pressure vessel continued to its desired level. Finally reset the gas bottle and "dump" valves, if desired, as set out in the previous section, and also turn the intensifier switch back to "automatic", if required.

## Reducing Pressure

Assuming that the "intensifier out" valve is already open or, if not, opening it carefully so as to equalize the pressure in the intensifier and the pressure vessel, the pressure can now be reduced by opening the "oil release" valve and backing off the intensifier, stopping at the position anticipated to be needed in the next run. Finally, assuming that the gas is not to be returned to the bottle and that the "dump" valve is already open and the bottle valve closed, or, if not, setting the valves thus, the pressure is now reduced to atmospheric by opening the "gas release" valve. Care should be taken to open this valve gradually so that excessive pressure does not build up in the low pressure lines and burst the 30 MPa rupture disk. If the furnace has been in use, the final pressure release is best done when the temperature has cooled to around 400 to 450 K in order to make some compensation for the very marked adiabatic cooling that tends to occur in the depressurizing gas. However, unless a particular p-T path is to be followed down, it is advisable to cool to the above temperature prior to any gas release in order to avoid the excessively slow cooling that occurs at room pressure. Finally, open all gas valves so as to avoid leaving some sections of the gas system under pressure inadvertently.

The computer program can be used for ramping the pressure up or down through control of the confining pressure set-point at any stage in the operating program. In order for this to be effective, it is necessary that the intensifier switch be set on "automatic" and to ensure that the intensifier piston is positioned so that there is sufficient stroke available in whichever direction it will be necessary for the intensifier piston to move for achieving the programmed pressure changes.

## Bubblers and Leak Detection

Tubes have been inserted at various places in the pressure vessel and intensifier in order to collect any leaked gas from 0-ring seals and direct it to a bubbler so that a leak can be located. The bubblers are viewed through the opening in the lower left part of the right-hand instrument panel. The connections are as follows:

1. Specimen jacket seal, via thermocouple hole in top piston
2. Top piston seal in closure plug
3. Top closure plug main seal plus six electrical feed-through seals
4. Bottom plug main seal plus the upper two seals on the loading/compensating piston assemblage
5. Bottom seal on loading/compensating piston assemblage
6. Internal load cell seals, including feedthroughs, via bore of loading/compensating piston assemblage
7. Intensifier gas piston
8. Intensifier exit plug seal
9. Volumometer piston seal

Bubbles in the upper chamber represent "**blowing**" and in the lower chamber "**sucking**". In the case of bubblers 4 and 7, bubbles observed during piston motion may only consist of air being displaced by this motion. In other bubblers, a few bubbles may be seen during pumping, which arise from settling down of 0-rings or collapse of the specimen jacket.

The fluid in the bubblers may need to be replenished from time to time, especially after a severe leak. **Tellus C10** oil is used.

Most other seals can be checked by inserting grease in the vent holes, for example, in the valve bodies and connector pieces. Often there is a vent hole on both sides of the part; one can be covered with tape so as to direct the leaked gas through the other hole. In the case of the connection into the pressure vessel, a cap is press-fitted into the vessel sleeve and sealed by an 0-ring around the tube; leakage can be detected by extrusion of grease placed in the small hole drilled in this cap.

## Pore pressure connection

There is a connecting port on the high-pressure side of the "intensifier in" valve, which is used for connection of the intensifier to the pore pressure system, through the "pore fluid interconnect" valve. This port can also be used for connecting a secondary standard pressure gauge in order to check the calibration of the "confining pressure" gauge, or for connection to other experiments, for example, for loading a diamond anvil cell with argon.

## Argon gas supply

The pumping rate in the machine is, of course, at a maximum when the argon bottle is full, usually at a pressure of around 15-20 MPa. If it is desired to retain this speed after substantial argon usage and to avoid wastage of argon by rejecting partially full bottles because of low pumping speed, it may be found worthwhile to set up a two-bottle arrangement, with one bottle being kept continually full by means of a gas booster pump linking the two. Gas can then always be drawn from a full bottle and the other bottle used down to a point of negligible wastage before exchanging for a new bottle. The second, continually full bottle should be exchanged from time to time with the replacement bottle in order to prevent undue accumulation of contamination. However, such an arrangement should only be used with appropriate filters and with the gas supplier's agreement.

## Replacing failed rupture disks

Spare rupture disks should be held in reserve. If it is necessary to change one, it will be found easiest to remove the body to the bench, insert the new disk according to the maker's instructions and re-fit the body, tightening the connection sufficiently to establish a seal. Overtightening will tend to distort the mating parts and make future sealing more difficult to achieve. Check the success of the attachment with grease as described under "Bubblers and leak detection".

## Changing 0-ring-gas seal on piston in intensifier

Position the intensifier at about half-way in its stroke (if it is initially further in, bottle gas pressure will be needed to move the piston out to this position). Ensure that all gas pressure is released (that is, all gas valves open). Remove the gas line connection from the top of the intensifier to the first connector block. Check that the intensifier gas cylinder turns freely in its threaded connection to the oil cylinder, using the main cylinder nut-extracting tool to loosen it if necessary. Screw the coupling on the hoist full length into the gas line connector thread in the top of the intensifier cylinder. Operate the hoist to take the weight of the cylinder while unscrewing it. Be careful to steady it near the lower end as it comes out of the oil cylinder so as to prevent damage as it swings freely.

Raise the cylinder carefully until the gas piston is well clear of the top of the oil cylinder. Cover the oil cylinder to prevent ingress of grit or dust. Remove the gas piston by hand (should it be tight due to excessive extrusion of the mitre ring or swelling of the 0-ring, it will be necessary to make up a split clamp from brass to clamp firmly on the piston close to the cylinder and then lever the piston out using the cylinder as fulcrum, taking care not to scratch the piston). Extract the 0-ring and mitre ring. if the mitre ring is OK, replace after removing any extrusion lip on it by rubbing on a piece of fine abrasive paper supported on a flat surface. Replace 0-ring, very slightly smeared with silicone grease (a hard Disogrin polyurethane 0-ring is used). Put a slight smear of silicone grease on the gas piston and slide back into the cylinder, taking care to support it by hand in case it drops out again under the effect of its own weight. Lower the gas cylinder gently meanwhile and when near the oil cylinder, position the gas piston so that it locates properly in the top of the oil piston. Continue lowering the gas cylinder carefully until the threads are again meshed, and then screw it into the gas cylinder until the threads are fully engaged. Near full engagement, proceed slowly so as not to cause the threads to tighten too firmly under the influence of the inertia of the cylinder; only bring it gently into the end position, otherwise difficulty will be encountered in trying to loosen the thread next time. Finally, replace the gas line connection to the top of the intensifier, using only moderate tightening torque in order to avoid excessive distortion of the coned end on the tubing.

## Oil in intensifier

The oil used in the intensifier oil cylinder is Shell Tellus C10. Topping up should only be required if there has been a failure of the oil piston seal or oil plumbing.

# HIGH TEMPERATURE FACILITY

The furnace has three windings and three furnace winding thermocouples, which are connected to the furnace power and control circuits. A specimen thermocouple can also been installed in the bore of the specimen assembly. The furnace controls are mounted in the left-hand instrument panel and the specimen thermocouple traverse mechanism is situated to the left of this panel.

The larger **Eurotherm** unit is the temperature controller, which is connected to the control thermocouple by the selection buttons situated below the controller. The thermocouple connections initially fitted are as follows:

1. Specimen, located in the bore of the upper loading piston; in case of a two-junction calibrating thermocouple, it is connected to the lower junction.
2. Upper junction of calibrating thermocouple, if fitted.
3. Top winding; junction situated near the bottom end of top winding.
4. Centre winding; junction situated near the middle of the centre winding.
5. Bottom winding; junction situated near the upper end of the bottom winding.

The temperature is both controlled and indicated from the thermocouple that is connected to the unit by pressing the selection button on the switchbox below the controller. It is important not to change the selection while the furnace is under control in order to avoid undue temperature excursions.

The smaller Eurotherm unit, adjacent and to the right of the temperature controller, indicates the temperature at the thermocouple selected by the button in the box below it. The two selection boxes are wired in parallel and the buttons represent the same thermocouples in both boxes. The right-hand unit enables the temperatures at all thermocouple junctions to be monitored in turn without affecting the control function determined by the thermocouple selected on the left-hand unit. The right hand unit therefore allows temperature profiles to be explored with a moving thermocouple connected to channels 1 and/or 2 while the furnace remains under control with a winding thermocouple 5, 6, or 7. It should be noted that the readings from channels 1 and 2 should represent true temperatures because the circuits are completed entirely with compensating wires, whereas the readings from channels 5, 6, and 7 represent only relative temperatures because of parasitic emfs introduced by the use of a steel feedthrough in one side of the circuit and an earth return in the other side; the readings on channels 5, 6 and 7 also differ substantially from the specimen temperature because they represent temperatures somewhat down-gradient from the winding temperatures themselves. For this and other reasons associated with possible drift in furnace behavior, it is important to calibrate the control thermocouples against a standard specimen thermocouple at regular intervals in order to keep account of the appropriate furnace control settings so long as a furnace thermocouple is used for control. For the same reason, it is always desirable to use, if possible, a specimen thermocouple inserted in the hollow upper piston to monitor the actual specimen temperature.

**Note:** Since the PICS control system is connected to the Eurotherm controller only**, the temperature recorded by the data logging system is that indicated by the thermocouple which has been selected for the control and is not necessarily the specimen temperature.** Thus, if the furnace is being controlled from one of the winding thermocouples, the temperature that will be logged is that for this control thermocouple, and the actual specimen temperature will have to be deduced from these readings using the calibration data from a recent temperature calibration, unless the reading on the other Eurotherm indicator, connected to a specimen thermocouple, has been noted at intervals during the experiment. If the specimen thermocouple is used for control, then the specimen temperature will be recorded directly; however, in this case, since there is a considerable time delay between application of power and response at the thermocouple, the Eurotherm controller will need to be re-tuned if it has been previously controlling through a winding thermocouple. Moreover, when controlling from the specimen thermocouple, it may be found to be difficult to tune the controller sufficiently finely to avoid obvious force fluctuations on the specimen resulting from thermal expansion fluctuations associated with small temperature fluctuations; when controlling from a winding thermocouple, temperature fluctuations at the windings are small and are attenuated before reaching the specimen, ensuring very steady temperatures at the specimen.

## Installation of furnace

The furnace itself is a self-contained unit assembled within a stainless steel sleeve of 62 mm outside diameter. It has three molybdenum windings, a central one covering 50 mm length, and two fringing ones covering 20 mm each. The winding connections, as well as connections to the three thermocouples adjacent to the windings, are wired to a 12-pin socket forming one end of the furnace, which plugs on to the 12 pins on the vessel closure plug. Six of these pins are earthed and the other six are connected to electrical feedthroughs.

To connect the furnace, simply plug it on to the vessel closure plug, making sure that the azimuth is such that the locating pin enters the corresponding locating hole. Then the mild steel retaining cylinder, with spring plate and spacer ring in the bottom of it, is pushed over the furnace and secured in place with three M3 x 6 mm long socket head cap screws. The spacer ring should be of such length as to give a moderate pre-loading to the furnace and to allow for the thermal expansion.

With the furnace attached, the plug and furnace assemblage is then inserted into the pressure vessel after checking that the 0-ring and mitre ring are correctly in place and putting a very slight smear of silicone grease on the 15' lead on the plug. The azimuth of the plug should be such that the feed-through slot marked with a nick (indent) on the right hand side as viewed from the access platform, that is, facing towards the desk at the front of the machine. The connections are then made according to the diagram, tightening up the brass rods on the feed-through pins and then attaching the leads to the brass rods. The rods pass outwards through close-fitting plastic plugs in the cooling sleeve, which serve to prevent leaked gas going to atmosphere and to direct it to bubbler 3.

## Operation

For operation of the temperature con of and monitoring units, see the Eurotherm instruction books. Since the windings are of molybdenum, it is essential that the furnace only be operated while the pressure vessel is filled with argon, and normally at high pressure.

To operate the furnace from the Eurotherm unit, proceed as follows:

1. Turn on the cooling water flow.
2. Checking first that the output power (OP) from the controller is zero, switch on the furnace power with the green button. To ensure that the OP value is zero, the temperature set-point has to be below the indicated temperature. If the OP value is initially high, a high current surge is likely to be trip the circuit breaker for the lower winding, the setting of which is designed to give maximum protection to the electrical feed-throughs in case of short circuits.
3. With the RAMP function on HOLD, achieved by pushing the RUN/HOLD key twice, go to SP1 in the operator menu and set the desired running temperature. Return to the short scroll list and again press the RUN/HOLD key to start the heating ramp. The ramp rate has been set at 20K per minute before delivery and this rate should not be exceeded in order to minimize thermal stress in the furnace during heating. If it is desired to change the set-point upwards at a subsequent time, the same procedure of ramping to the new setpoint should be observed.
4. At the end of the run, it is sufficient to switch off the power by pushing the red supply button. Owing to the effectiveness of the furnace insulation, the temperature will not fall very rapidly and there is no risk of furnace damage due to thermal shock. Alternatively a controlled rate of cooling can be achieved by ramping or programming.

## Calibration of the furnace

Each of the three furnace windings is supplied by a separate thyristor, situated behind the instrument panel. The thyristors are fitted with current limitation and settings have been made to avoid currents in excess of about 15 A during heating; otherwise very large currents would be drawn when applying full voltage to cold molybdenum windings.

The output power from the Eurotherm control unit is connected in parallel to all three thyristors but is attenuated individually with the rheostats situated to the left of the ammeters. These rheostats enable the relative power to be adjusted in the three windings during calibration so as to achieve a desired temperature distribution. The rheostat for the winding taking maximum current, usually the bottom winding, should be set to its maximum. If a uniform temperature is required, this should be achievable over a length of about 50 mm in the central part of the furnace, with a deviation of as small as a kelvin or two.

The selection knob next to the voltmeter enables the voltage to be monitored from the transformer output (V) and on each of the windings (B, C, T for bottom, centre and top, respectively). Since moving iron meters are used for both voltage and current, the readings are approximately rms values and so multiplying them in pairs gives approximately the individual power dissipations in each of the windings. It will generally be found that the power in the bottom winding will be the largest, and that in the centre winding the least, for a uniform temperature distribution in the hot zone.

For calibration, a dummy specimen assembly is needed, with a 2 mm bore extending over the full length of the temperature to be explored. A suitable and convenient assembly consists of a 30 mm length of 15 mm diameter PSZ piston at each end and three 42 mm lengths of 15 mm diameter alumina between. This assembly is mounted on upper and lower anvil pistons, as set out in the later section on Specimen Assembly and Deformation Procedures. The calibration thermocouple has two junctions 100 mm apart. It should be set up on the thermocouple traversing arm so that it reaches to the top of the lower anvil piston closing the specimen assembly when the traversing control is set at zero at the front of the machine. Using this traverse control, temperature readings can be taken at, say, 10 mm intervals; at each point two readings can be taken, using the positions 1 and 2 on the switchbox attached to the right-hand meter, so that a 20 mm temperature profile can be obtained with only 10 mm traverse of the thermocouple. Then adjustments to the power partitioning rheostats are made so that the desired temperature profile is obtained. At the same time, the temperature reading for the furnace control thermocouple, as displayed on the temperature controller, is recorded for future setting. It will probably be found that control on the top winding thermocouple will be the most satisfactory.

If a good quality alumina dummy specimen material is used, it should be possible to carry out such a temperature calibration to about 1500-1600 K at 300 MPa pressure. However, at higher temperatures and/or higher pressures, collapse of the bore of the alumina may cause trapping and loss of the thermocouple. For these conditions, it may be necessary to depend on calibration at a series of lower temperatures, and extrapolation of the profiles and settings made to the higher conditions, or make up an arrangement with a thermocouple trapped in the piston.

# DEFORMATION FACILITY

The specimen is deformed by advancing the pressure-compensated loading piston fitted in the bottom closure of the pressure vessel. Pressure compensation is effected by applying the gas pressure to the reverse side of an annular boss on the main piston This annulus has the same area as the cross-section of the main piston, so as to counterbalance the force on the piston due to the confining pressure alone. The force needed to advance the piston is therefore solely that due to the stress difference ("differential stress") in the specimen plus jacket, with the addition of the friction on the piston.

The loading piston is moved by the actuator attached below the pressure vessel support. However, fitted between the actuator and loading piston is an external load cell and a stirrup or yoke, which gives access to the end of the piston for the introduction of electrical leads and pore pressure fittings.

## Actuator

The actuator is based on a ball screw and nut, with a 10 mm lead, supported between thrust ball bearings, and driven by a Printed Motors servo-motor through reduction gearing. The speed reduction consists of a 5:1 ratio belt drive followed by a 200:1 Harmonic Drive reduction gear. The maximum speed of the motor is 3000 rpm. The load rating of the actuator is 100 kN in both extension and compression. Its total displacement range is nominally 50 mm but there are limit switches, set near the end positions, which open circuit the motor power if tripped. In order to restore motor operation after tripping the "hard" limits, it is necessary to wind back the actuator by turning the motor pulley by hand. Only the upper 30 mm of the range of the actuator is used for the movement of the deformation piston, and there is no microswitch limit on the bottom of this range; it is therefore important always to set appropriate position and load limits in the PICS unit, through which the motor is operated.

## Internal load cell

At the inner end of the loading piston is attached the internal load cell, which also incorporates the anvil through which the force is applied to the specimen assembly itself. The internal load cell has a rating of 100 kN in both extension and compression. It consists of an elastic element, to each end of which are attached the plates of a capacitance displacement transducer. The transducer consists of three circular plates, the outer pair being connected to the top end of the elastic element, while the central plate is connected to the bottom end and is equidistant between the outer plates. The two capacitances thus form a half-bridge. The plates are initially positioned so that the two variable capacitances are as nearly equal as possible. The "other half-bridge" is formed by an externally-mounted pair of resistors connected through a balancing potentiometer, the wiper of which is connected to ground (see circuit diagram)

An excitation voltage from the Radio Spares (Schlumberger) 646-583 oscillator/demodulator card, of about 5V and frequency 10 kHz, is applied to the outer plates. The voltage difference between the inner plate and ground is then a measure of the axial force on the load cell. This voltage is applied to a buffer amplifier, the first stage of which is mounted immediately on the atmospheric-pressure side of the load cell body, from which connection is made along the load cell stem to a connector within the stirrup and thence to the main part of the amplifier mounted in a box within the machine housing. The low impedance output of the buffer amplifier is, in turn, connected to the demodulator unit, also in the same box. In initial setting up, the bridge is balanced by minimizing its AC output, monitored from the two sockets on the front of the preamplifier box inside the machine housing, by adjustment of the potentiometer that is also accessible from the front of the preamplifier box.

By means of the adjustment knob adjacent to the internal force meter on the front panel, the signal from the demodulator unit to the display and PICS retransmission can be offset to indicate zero under no-load conditions if this zero has been displaced during the application of the confining pressure or has drifted. Initially, this potentiometer should be set at midpoint and the meter reading zeroed by adjustment of the Z adjustment on the preamplifier box (this is attached to the RS card). There is also an offset potentiometer mounted on the distribution board on the side of the machine under the platform, but this is redundant.

The internal load cell output is adjusted with the S potentiometer on the front of the preamplifier box. It can, in principle, be calibrated by comparing the readings with those of the external load cell. However, at pressures above room pressure, this comparison has to be made using the mean of readings with "friction in" and "friction out", which is necessary because the calibration of the load cell may be different under pressure than at atmospheric pressure. Since friction is notoriously variable and non-repeatable, a more precise calibration can be made using a spring mounted in place of the specimen assembly, the spring constant of which has been determined independently. Such a calibrating spring is supplied as an accessory. When calibrating with the spring, displacement versus force readings are made both for the spring and for a steel blank, the displacement readings for the latter then being subtracted from the former so as to remove the apparatus distortion component and yield the true amount of shortening of the spring. From this shortening, the force can be calculated using the known spring constant (corrected for change of elastic modulus with pressure, if desired).

In practice, care has to be taken to avoid introducing conducting particles that can short-circuit the pairs of capacitance plates. Experience has shown that electrical shorts between plates are the most common source of problems with the internal load cell.

In the base of the internal load cell, there is an axially located feed-through which is primarily for a pore fluid connection. In the absence of the pore fluid connection, this opening is blanked off by a sealing block that serves to fill the central space in the load cell. However, the block can also be substituted by an electrical feedthrough in the event that an additional one is needed; it is even possible to fit a multiple electrical feed-through in this space.

The top of the internal load cell is provided with a screwed-in anvil which can be used for simple compression testing, or for extension testing if the bayonet-ended anvil piston is fitted to the specimen assembly. The screwed-in anvil is removed when the down-stream pore fluid connection is made. This change-over can be done from the top of the pressure vessel if the furnace is first removed and then the cap over the bottom plug section that houses the internal load cell.

## Installation of internal load cell and bottom piston

**Caution:** When installing the internal load cell, great care must be taken to avoid damage to the electrical connector attached to the stem connecting the two parts of the bottom piston assembly. The nut on this stem, which also serves as a protective sleeve, should always be attached during assembly or servicing operations.

First screw the inner or load cell piston into the seat of the internal load cell, fitted with its 0-ring, until it is firmly seated. Next push the load cell and piston assembly into the body of the bottom closure plug after checking the 0-ring and mitre ring and slightly greasing the leading edge of the piston with silicone grease. Then screw on the cover of the bottom closure plug. Next insert the compensating piston with its outer 0-ring seal in place, taking similar care of the 0-rings and mitre rings, and ensuring that the locating pins enter the holes in the bottom of the load cell piston. Finally, screw the smooth nut on to the projecting stem that holds together the inner and outer portions of the loading piston assembly.

With the actuator fully retracted, the external load cell and its connecting pieces removed, and the LVDT connector detached from the top of the actuator, place the bottom closure and its assembly below the pressure vessel and support them while the long assembly rod is inserted through the pressure vessel from the top and screwed into the cover of the bottom closure. Then the assembly rod and attached closure can be raised into position, having previously checked that mitre ring and 0-ring in the pressure vessel are in good condition and in place, and having put a slight smear of silicone grease on the leading edge of the sealing section on the closure plug. When the closure is in place, a cross bar can be inserted in the assembly rod to hold it temporarily in place while screwing in the bottom nut.

With the piston assembly in its bottom position, attach the stirrup to it and hold in place temporarily with the hexagon nut while the external load cell and its attached spacer piece are put in place on the top of the actuator piston. By undoing the hexagonal nut the stirrup can then be lowered carefully on to the external load cell. Now insert and tighten the screws holding the external load cell to the stirrup, rotating the latter as needed for easy access. Next, the screws that hold the load cell spacer to the actuator piston can be inserted and tightened. Re-attach the actuator LVDT and external load cell connections. The actuator can now be operated to bring the stirrup up to contact with the shoulder of the piston; holding the piston with the 27 mm AF open-ended spanner, re-attach and tighten hexagonal nut using the 32 mm AF open-ended spanner. Attach the buffer amplifier lead to the connector at the bottom end of the piston, taking care to mesh the electrical connection in the correct azimuth. The assembly should now be functional after moving the actuator and piston into the desired operating position.

For dis-assembly, the above procedure is reversed.. First move the actuator to the bottom position of the piston so as to be able readily to unscrew the hexagonal nut and free the stirrup. Next move the actuator to its bottom position or sufficiently low to allow space for removing the stirrup, the external load cell assembly, and the bottom closure. Use the long assembly rod to support the closure while screwing out the bottom nut. Take great care at the moment the nut becomes fully disengaged to ensure that its weight is well supported lest it fall and hurt a finger. A soft pad placed over the actuator piston is a good precaution.

# SPECIMEN ASSEMBLY AND TESTING PROCEDURES

The details of specimen assemblies can be expected to vary widely according to the requirements of particular experiments. Thus, the specimen arrangement is more a matter of the experimental design than a feature of the machine itself. Nevertheless, it must be compatible with the machine. The following description refers to a standard arrangement that can be widely applied in the compressive triaxial testing of a variety of materials, using a cylindrical specimen of 10 mm diameter.

## Parts required

1. Specimen, 10 mm diameter x 20 mm length
2. Two pistons of 80 mm length; alternatives for the pistons are
	* full length high-speed tool steel up to 800 K
	* full length partially stabilized zirconia (PSZ) up to 1200 K
	* 30 mm PSZ + 50 mm Al203 adjacent to specimen above 1200 K
3. Two spacers, 10 mm diameter x 3 mm length of similar material to that of pistons (the spacer serves to protect the piston from damage in case of failure and to prevent the specimen being extruded up the thermocouple hole)
4. Upper steel anvil piston and bottom steel anvil piston Approx. 210 mm length iron jacket
5. Two 0-rings, one size 014 and one 114

## Preparing the jacket

1. Assemble the former of the jacket spinning jig by screwing together the two dummy pistons and the 26 mm spacer or dummy specimen (the length and diameter of this spacer can be varied to suit particular experimental requirements).
2. Push the assembly into the jacket so that the end of the jacket coincides with the end of the hollow dummy piston housing the long nut.
3. Push the collet over this end of the assembly so that the hollow dummy piston and jacket are supported in the counterbore of the collet and hold the collet in the lathe, preferably within another, 19 mm, collet and tighten. Alternatively, the jacket assembly can be clamped directly in a chuck.
4. Support the other end of the assembly on a "live" centre with a small end load.
5. With the spinning wheel mounted in the toolholder normal to the jacket, and the jacket assembly turning in the lathe, push the wheel against the jacket in the section to be reduced and traverse it back and forth, gradually moving inwards and feeling the limits by the resistance on the cross-feed handle until the jacket confirms to the dummy piston and specimen assembly within it.
6. Disassemble the jig.
7. Machine the ends of the jacket to the appropriate lengths, the top end to be within about 0.5 mm of the shoulder on the top anvil piston ( in the case of pore pressure experiments, a gap of about 0.5 mm should be allowed here), and the bottom end extending about lmm beyond the 0-ring.
8. Polish the OD near the end of the longer parallel portion of the jacket and the ID near the end of the shorter portion, to ensure good 0-ring seals.

## Assembly and mounting

1. Insert one of the pistons into the jacket.
2. Insert successively one spacer, the specimen, and the second spacer into the section of reduced diameter.
3. Insert the second piston.
4. Insert the bottom anvil piston, with 0-ring fitted, into the end that is polished on the ID, being careful to avoid scratching the polished section and to tuck in the 0-ring without damage (a blunt brass pencil-like tool is useful for this purpose, or a fingernail).
5. Slide the assemblage on to the top anvil piston, with the 0-ring and compressing angle ring, and tighten down the compressing nut by hand, ensuring that the gap between the nut and the body of the piston is substantially less than 1 mm in order not to catch on the mitre ring in the plug.
6. Attach the furnace sealing sleeve by wrapping 2 or 3 layers of Zircar alumina paper APA2 around the jacket (3 layers = 172 x 60 mm, 2 layers = 115 x 60 mm) and compressing it with the three segments of the furnace inner sleeve; slip the lips of the segments under the retaining groove on the 0-ring compressing nut, and slide the steel ring on the other end (this will be pushed up by the furnace core but should slide down to more or less the same position on removing the specimen assembly from the furnace again).
7. With a suitable tube attached to a small vacuum cleaner, remove any dust or particles from the anvil seat on the internal load cell.
8. Very thinly smear the 15° lead on the top piston with silicone grease.
9. Using the threaded insertion tool, slide the assembly through the top plug with a slightly rotary motion, feeling it pass through the various close fits and finally contact the anvil seat on the internal load cell; then draw back a little to allow for thermal expansion of the specimen and some "run-in" prior to contact in a deformation experiment.. The anvil contact has a distinctive firm feel, learnt by experience, which needs to be distinguished from "hanging up" on other impediments on the way in.
10. Finally, screw the small top anvil piston support nut into the large plug-supporting nut until contact is made with the large nut. At this point, the anvil piston support nut should be contacting the anvil piston and there should be a small clearance between the bottom anvil piston and the load cell. The actuator and bottom loading piston have to have been positioned by actuator movement beforehand in order to achieve these relative positions.

## Deformation testing

The following notes give a broad guidance to the procedure for carrying out a deformation test, using mainly manual control. To carry out a test under the CDAQ computer control system, see the control system manual and the supplementary notes below. In practice, the detailed procedures will be determined by the aims of the particular experiment. In the case of manual control, it is important to set the limits on position, external force and internal force using the touchscreen on the PICS unit before operating the actuator.

We shall assume that the specimen assembly has been inserted, as described elsewhere, and that the actuator has been positioned so as to allow perhaps 0.5 mm free movement of the bottom piston before contact with the specimen. The contact point can be checked by advancing the piston very carefully with the actuator until the first registration of load by the internal load cell is observed, and then the actuator is backed off by a suitable amount to allow for thermal expansion during heating.

With this preliminary positioning, the pressure and temperature can now be raised to the desired level. The behavior of the specimen during pressure and temperature increase can be monitored if desired by occasionally advancing the piston to the "touch point", taking care to limit the force to a small value relative to the strength of the specimen; alternatively, the CDAQ program can be used in internal force mode to maintain a small force on the specimen and monitor the displacement. Some calibration of temperature and pressure effects on the apparatus dimensions will be needed for interpretation of such observations.

After the pressure and temperature conditions have been established, advance the piston to the touch-point one or more times in order to establish the zero for subsequent displacement measurements during deformation. Then the PICS unit can be operated through the touchscreen to ramp the position at the desired displacement rate for the test and deform the specimen. Upon reaching the desired deformation, stop the actuator and reverse it to remove the load, backing off a short amount. Then advance again as at the beginning of the test to establish anew the touch-point. The difference between the original and new touch-points gives the total change of length of the specimen achieved during the deformation. This can be compared with the change of length measured at atmospheric pressure in order to determine whether there are other length changes introduced during going up to or coming back from the pressure-temperature conditions of the test.

# PORE PRESSURE SYSTEM

The pore pressure system consists of the following parts:

1. The volumometer, which serves to generate and to control pore fluid pressure. The pressure cylinder and piston are made from **Assab Stavax** stainless steel for resistance to corrosion in case of the use of water as pore fluid.
2. The connections of the volumometer to the upper end of the specimen assembly.
3. The connections of the down-stream pressure transducer to the lower end of the specimen assembly.
4. Up-stream and down-stream pressure transducers.

There is also a bracket for alternative connection of the volumometer, or of a second volumometer, to the lower end of the specimen assembly.

## The Volumometer

The actuator part of the volumometer should not normally need servicing, except possibly to re-grease it after several years. The gears, bearings and ball screw are initially packed with Shell Lithiplex L grease. However, the 0-ring in the piston seal may need to be renewed from time to time. For access to this seal, the retaining nut 10 is unscrewed and the main cylinder 1 withdrawn, leaving the piston 9 in place. After unscrewing the retaining nut 6, the seal sleeve 5 can be removed. There is an 012 0-ring located in the body of the main cylinder and an 010 (or better, an 803) 0-ring located in the bore of the seal sleeve 5, backed up by a beryllium-copper mitre ring. Replace both 0-rings and, if necessary, the mitre ring, making sure to place the latter so that it mates to the chamfer in the seal sleeve 5. Put a slight smear of silicone grease on the piston and re-assemble in the reverse order.

The volumometer is fitted with limit switches that interrupt the power supply to the motor near each end of the travel of the piston. If a microswitch is tripped, it will be necessary to wind back the motor by hand to close the switch again; this can be done by turning the small wheel attached to the outer end of the motor shaft.

## Experiments with pore fluid pressure.

The schematic arrangement for a pore fluid experiment is shown in drawing 5009. A tee-piece above the pressure vessel serves as a connection between the tube coming from the volumometer and the tube that goes down through the top piston to the specimen assembly. The thermocouple (a metal-sheathed ceramic-insulated one, silver-soldered into a blank cone) passes down through the vertical tube to reach to just above the specimen. The vertical tube is sealed to a separate spigot, serving as the top anvil piston of the specimen assembly, in place of the integral spigot on the normal top anvil piston. The jacket should be made about 0.5 mm shorter than required to reach to the top of the spigot in order that the 0-ring can serve to seal the confining pressure fluid both from entering the pore fluid system and from escaping past the spigot to atmosphere. The hollow bottom anvil piston is inserted in the other end of the jacket in the normal way.

Before introducing the assembly into the pressure vessel, ensure that there is an 014 0-ring in the groove in the pore fluid anvil piece that is located in the top of the load cell in place of the normal anvil piece. This anvil is spring loaded by the coiled small-bore pressure tubing that connects it to the tube leading to the down-stream pressure gauge. Apply a small load to the assembly so that when the confining pressure is applied the seal will be effected between the bottom of the specimen assembly and the anvil. After the seal is made, the specimen assembly can be raised slightly (by a maximum of about 2.5 mm) so as to allow the "touch point" to be established.

When argon is used as pore fluid, connection to the argon confining pressure system is made by closing the "pore fluid release" valve and opening the upper "pore fluid interconnect" valve. The latter valve is left open during the raising of the confining pressure until this pressure reaches the value required for the pore pressure. At this point the "pore fluid interconnect" valve is closed and the confining pressure taken up further to its operating level. From this stage on, the pore fluid pressure is controlled by advancing or retracting the piston of the volumometer under the control of the PICS system. When there is also a downstream pressure transducer and it is connected to the lower "pore fluid interconnect" valve, this valve should be left open to ensure filling of the specimen from both ends. For a permeability experiment, this valve is closed before running.

When other pore fluids are to be used, they can be introduced through the "pore fluid release" valve. In the case of a liquid such as water, the pore fluid system can be evacuated and the pore fluid introduced by "back-filling" under the effect of atmospheric pressure. If the volumometer piston is initially in its outermost position, it can then be advanced to raise the pore fluid pressure to the desired operating level. Where the pore fluid is too compressible for this mode of operating, an auxiliary external pressurizing system will need to be attached.

If a downstream connection to the pore fluid system is not required, the normal blind bottom closure piston of the specimen assembly is used, together with the normal anvil in the top of the load cell.

For permeability and storage capacity measurements, the set-up with upsteam and down-stream connections, described above, is used. The pore fluid pressure can then be given a sinusoidal fluctuation by the PICS unit controlling the volumometer, and the downstream pressure fluctuation monitored. When a low permeability specimen is used, it may be found that the initial time needed for the filling of the downstream part of the system is excessive without direct connection being made through the lower fluid interconnect valve. For maximum sensitivity in measuring small permeabilities, it is essential to minimize the downstream volume, which can be assisted by filling all tubing bores with stainless steel wire. For further information on procedure and data processing, see the control system manual.

# INSTALLATION

For shipping, the stairs and the access platform, with the control unit, are removed from the left-hand side of the machine and crated separately. The machine itself is then laid horizontally on its right-hand side and attached by means of brackets to a cradle. A wooden crate is attached to the cradle; with overall horizontal dimensions of approximately 1.2 x 2.6 m (the machine itself, on its side, has horizontal dimensions of approximately 1.0 x 2.1 m).

For handling the crate it is recommended that a forklift truck be used, lifting from under the cradle. If a crane is to be used, the crating should first be removed from top and sides and the eyebolts fitted to the ends of the machine used for lifting. Slinging the whole crate should be avoided because of the tendency to crush the crating between the slings, unless spreaders are placed between the sling ropes on top of the crate.

The principal installation steps are, first, to remove the cradle from under the machine and, second, to raise the machine to its normal upright position. These steps can be handled with an overhead travelling crane of capacity 2.5 tons or more, using slings attached to the eyebolts fitted at top and bottom of the machine (that is, at each end in the horizontal position). However, if such a facility and/or suitable headroom in the laboratory is lacking, it is suggested that the maneuvering of the machine be done using jacks of the Enerpac/Portopower type with appropriate extension tubes and attachment brackets, for example, a set of four hydraulic cylinders and pumps (with pressure gauges for force monitoring) of minimum 2 ton capacity each, with clevis eye mountings at each end and a sufficient set of extension tubes to permit the following maneuvers. For attachment of the jacks to the machine, channel or angle section beams can be fitted to the machine by the bolt holes at either end, and the clevis eye mountings attached to the beams, which should be projecting beyond the width of the machine. An angle section of about 50 x 50 x 5 mm or a channel section of 100 x 50 mm is suggested. With a beam attached at each end of the machine and with suitable footings on the jacks to prevent tilting, the machine can be lifted for removing the cradle, and then lowered on the floor. At this stage, the machine is still lying on its side. It now has to be put in a vertical position. With both beams attached to the top end of the machine, one pair of jacks can be used to lift this end and the second pair used to stabilize the machine as it approaches the vertical position. While up-ending the machine, it is necessary to retain the bracing piece that has been fitted between the two rear legs in order to avoid possible bending of the rear right-hand leg, and it may also be necessary to provide bracing to prevent the right-hand legs from sliding on the floor during the tilting. Particular care is necessary with the second set of jacks at the stage when the diagonal line between the top-left edge and bottom right feet approaches the vertical. These jacks need to be braced against a wall or other suitable fixture to take the force as the machine approaches the vertical position. The mass distribution is approximately but not exactly neutral as the above diagonal line passes through the vertical. The operation will have to be carried out in relatively small steps, the machine being supported between each step while the extension tubes are changed to suit the next step. If the operation of standing the machine upright is done with a crane, similar care is necessary as the machine moves through the neutral position.

Once in the vertical position, it may again be necessary to use the crane or the jacks to support the weight of the machine while its position on the floor is adjusted to what is required. If the machine has to be moved an appreciable distance to the installation site after standing up, castor wheels can be mounted to the four feet, using the 24 mm diameter holes in the feet for fixing. When in place, holding-down bolts can be used to fix the machine to the floor if desired but this is not normally necessary.

After the machine is in place, the access platform with control unit can be attached to the left hand side with the screws provided (these have been re-inserted in their tapped holes during the packing). Then the stairs are attached to the rear of the platform. It is recommended that the machine be placed with its back facing a wall, ideally at a distance of about 1 meter, and the stairs can be located in this space. The other attachments to be made are the operator's desk and the shelf above the desk, for which brackets and screws are provided. The bracing bracket on the top of the pressure vessel, and the three supporting rods need to be removed from the top of the pressure vessel, as well as the wooden support strut between the pressure vessel and the right-hand wall of the cabinet. The hand-operated hoist should be fitted to the bracket above the intensifier for future servicing of the latter.

Once the machine is in position, compressed air, cooling water and cooling water return pipes can be fitted, using the mating half of the "1/2 inch" Yorkshire fittings provided. These fittings are designed for 12.7 mm OD tubing; if 12 mm tubing is used, it will be necessary to expand it slightly or insert a filler piece before soldering. The argon gas supply is attached by a standard high pressure fitting.

Electrical connection to 220/240V 50/60 cycle supply is made using the cable left attached to the junction box on the rear of the lower front panel. The live and neutral wires are brown and blue, respectively, and the earth green with yellow stripe.

The PICS control unit and the actuator switch panel are attached to the front of the access platform and the connections made to the cables exiting from the left-hand side of the main cabinet according to the markings on the cables or the appropriate connectors. The computer and recorder are housed in the additional hinged unit that has to be attached to the left-hand side of the access platform, and the connections made to the PICS unit.

# Add-ons for MIT Paterson

|  |  |
| --- | --- |
| Furnace tube:  | OD=27mm; ID=20.8 (21)mm |
| Position of Furnace insert: |  dent is at 3pm – make sure furnace and thermocouple connectors are aligned. Connectors (clockwise from 3pm location: Middle Furnace – Middle TC – Bottom Furnace – Bottom TC – Top Furnace – Top TC (3pm) |
| Top for furnace:  | Location of pin is at 1pm. If not – make sure that furnace is inserted properly. Also check if bottom cover for load cell is loose. |
| Top for sample assembly: |  Location at 11pm. If not lower RAM – sample might be too long. |

## Flushing the System

An alternative procedure for flushing is to attach a rotary vacuum pump to the outlet of the "dump" valve and pump down the whole system with this valve open. When evacuated, the system is charged with argon at bottle pressure after closing the "dump" valve. However, this procedure may disturb the seating of the 0-rings by placing a one-atmosphere reverse pressure on them and so possibly increase the chance of a subsequent leak.

## Acqusition System

Dell Optiplex GXM 5120

Pentium 80MB Ram; 1GB HD (MS-DOS-622)

SanCarlos; TCP/IP 18.83.1.45

255.255.0.0 – 18.83.0.1 (GW) – 18.71.0.151; 18.70.0.160; 18.72.0.3

WIN 7

National Instrument Board: PXI-DAC-16X (PXI; 16bit ADC; 1-100gain; 5 Counter, 2 16bit AO)

AI Channels (8 differential)

0-internal Force

1-Displacement 2.5mm

2-Displacement 50mm

3-Position ?

4-Pore pressure (up)

5-Pore pressure (down)

6-External Force

7-Confining pressure

8-temperature

AO Channels (2 differential?)

0-axial server motor voltage [-10-+10V]

1-pore fluid motor [-10-+10V]

Serial Port write/read (computer board)

Eurotherm 903 Control-write ramp, set point, rate)

## Sample Preparation

Fe- Jacket removal:

Use 12th Floor hood. Close plastic container while using. Mix 1 part HNO3 and 3 parts HCl and add sample. Wait ~1 day. Dispose spent acid and check if bottles need to be refilled. Rinse treated pieces extensively in sink. Always use latex gloves and glasses.

Sample Preparation

Furnace calibration (high temperature)

Cut steel tubing to ~220mm pieces on lathe.

Assemble Al rods with internal bore to fit in Fe jacket (some might be too tight). Pick two pieces for one side.

Use mandrill (10mm diameter; 11.75mm is for HIP) and fit Fe jacket. Measure and mark center location (remember that jacket is not symmetrical, the bottom part is 6mm shorter). Indent jacket to 10mm using the lath.

Disassemble jacket, mandrill unit (punch out center piece, if necessary). Mount Al rods (2) for bottom section. Measure gap between end of Al rod and jacket end and cut it to 10mm. Polish inside jacket (O-ring seat).

Turn pieces top section and turn gap down to 16mm. Polish outside for O-Ring seat.

Disassemble, clean and dry.

## O-Ring List

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | Paterson O-Ring List | # | ID | OD |  |  |
| 3 | Pressure vessel Bore, both ends | 335 | 67 |  | 1.5 |  |
| 6 | Bubbler Ends | 10 |  | 9.5 |  |  |
| 1 | Lower Unit | 217 | 30 |  |  |  |
| 9 | Bubbler Bodies | 014 or 112 | 13 | 16 |  |  |
| 2 | Hydraulic Actuator Parts | 619 | 74.6 |  |  | 337 |
| 4 | Hydraulic Actuator Parts | 621 | 89.7 |  | 1.5 | 342 |
| 1 | Intensifier End cap | 426 | 118.5 | 130 | 1.5 |  |
| 1 | Intensifier Oil Piston | 426 | 118.5 | 130 | 1.5 |  |
| 1 | Intensifier gas Piston | 223 | 41 |  | 1 |  |
| 10 | Temp Plug feed | 10 |  |  |  |  |
| 10 |  | 7 |  |  |  |  |
| 1 | Piston/Jacket | 114 |  |  |  |  |
| 1 | Piston/Jacket-lower | 14 |  |  |  |  |
| 3 | Bottom/Top Piston | 217 | 30 |  | 1 |  |

Table 4: List of O-Rings

## High-resolution DCDT for Axial Displacement Measurement and Control

### Installation

Mount the switch panel on the platform frame immediately above the actuator switch panel and PICS. Mount the relay and connector board in an appropriate place for making the connections to the transducers. In the case of the Potsdam machine, this place will be on the inside wall of the instrument compartment, on the platform side, at the same level as the meters. In the case of the MIT machine, it will be adjacent to the Analog Devices transducer conditioning board, possibly most conveniently on the inside of the back panel underneath the platform.

Mount the unit on the side of the yoke using two M6 screws and remove the retaining pin in the core extension to allow the core to register on the bottom side of the pressure vessel.

Then attach the flexible drives to the two shafts projecting on the LH side of the unit. The lower drive serves for remote positioning of the unit and the upper one for locking it in position. The other ends of the flexible drives are to be mounted with suitable clamps on the side of the housing just below the level of the desk so that they can be manipulated while observing the positioning lights on the switch panel. It will be necessary to cut a slot in the top of the panel enclosing the lower side of the machine in order to accommodate the flexible drives. The flexible cables will enter the high pressure enclosure through the vent between the intensifier and the wall, and curve around the intensifier.

The leads that at present go from the actuator DCDT to the POSITION meter (in the Potsdam case) or to the Analog Devices board (in the MIT case) have to be redirected to the connector board and new leads run from the connector board to the meter or AD board, respectively, according to the wiring diagram supplied. The other leads from the connector board go to the switch panel. A 24 V ac supply line are shown in the diagram being taken from the existing actuator switch panel to the new switch panel but this can be taken from anywhere convenient.

It is very important in connecting up to ensure that the polarity of the supply to the DCDT's be preserved as at present since reversal of the supply to DCDT tends to destroy it (there seems to be no inbuilt protection in the Schaevitz units).

Note that the Schaevitz 050 DC-E DCDT (range ±1.25 mm) is mounted with its leads emerging from the top end in order to give the same output polarity as the output from the actuator DCDT. The mounting unit has been dimensioned so that the next larger sized Schaevitz, the 125 DC-E DCDT with ±3.0 mm range, can alternatively be used if a 6 mm range is desired instead of the 2.5 mm range that the 050 DC-E gives, with corresponding reduction in resolution.

### Operation

The HR DCDT unit is designed to enable the operation of the testing machine in axial displacement (AD) mode with a step size of axial displacement advance of 1/20 of that obtained using the actuator DCDT output as feedback, that is, 0.6 µm instead of 12ym. Finer resolution data logging is then also provided. Further, since the measurement is effectively made where the loading piston emerges from the pressure vessel, the apparatus distortion corrections will be reduced in magnitude. The piston travel available with control from the HR DCDT is 2.5 mm, corresponding to about 12% strain. The experiment is therefore normally limited to this amount of displacement. If more is required, it will be necessary to interrupt the experiment, reposition the HR DCDT unit and repeat the operation, or install a DCDT of 6 mm range. However, it might be expected that with larger-strain experiments the actuator step size will be less critical and that therefore the normal actuator DCDT will be used for feedback and data logging.

In using the HR DCDT facility, the following procedures should be followed and precautions observed:

1. Position the actuator where the experiment is to start. This is preferably done with the confining pressure and temperature conditions for the experiment already established and the piston position at or near the touchpoint. Note the absolute actuator position if required.
2. Position the HR DCDT unit so that its core is near the end of its range. This can be done using the flexible drives, first unclamping the unit with the upper drive and then moving it to the required position with the lower drive and finally clamping it again with the upper. The positioning lights on the switch panel, actuated by microswitches on the unit, are used for guidance. The microswitches must be set so that the respective lights are turned on when the core of the HR DCDT is at its upper and lower measuring limits (this setting will need to be done in the first set-up but, once done, will remain valid for other positions of the DCDT). In a compression test, the unit is moved down until the LOW light comes on and then moved back until the light is just extinguished again, ensuring that the whole of the linear range of 2.5 mm is available for the experiment.
3. With the actuator motor switched off (very important):
	1. Turn the change-over switch on the switch panel to the HR DCDT position.
	2. On the PICS AD status screen, select the CALIBRATION option and set the DCDT calibration to 2.5 mm.
	3. Set the axial displacement limits as required; probably, 0 and 2.5 will normally be appropriate.
4. Now the actuator can be switched on and the experiment carried out in the normal way.
5. After switching back to the normal DCDT, the calibration setting in the PICS must be reset to 50 mm and new limits set.

Note that, with the HR DCDT switched in, the POSITION digital meter on the main panel will be reading in units of 1/20 mm, that is, the readings have to be divided by 20 to obtain the relative position in millimeters; this position will be relative to the new zero established by the positioning of the HR DCDT unit.

## Temperature Calibration

Assemble Calibration Column (jacket and Al Rod with 2mm bore). Total length in mm is: 10 (Gap bottom)+80 (Al-column)+26 (Zr cylinder)+80(Al-column)+16 (Gap top).

Inspect surfaces (O-ring seats) for scratches, and bore for Thermocouple. Mount bottom plug (no bore) and top plug (open). Add jacket sleeve (top) to prevent convection.

Mount Calibration Column. Check O-ring seat on bottom. If necessary re-adjust with long contraption. Check furnace and thermocouple connections. Close top plug (note plug positions, don’t tighten to hard). Mount thermocouple in Holder and insert into column (check if it goes all the way to the bottom). Connect TC and leak tubes and close lid.

Flush air out of confining pressure system. Make sure, Pore/Confining Pressure Interconnect Valve is closed. Use gas from intensifier and open to vessel (Booster Valve closed). Bleed off to Release and Dump. Repeat three times to flush air out.

Find touch point of axial load. Select safe soft settings on the computer, so motor does not jump. Switch on actuator. Ramp up slowly and monitor internal and external load cells for steep increase. In case of calibration column, the material strength is high and the increase will be sharp. Stop actuator at touch point (or at a slight load).

Pressure up confining pressure system: Close all doors (otherwise system will stop at 30MPa). Open Argon gas bottle. Open inlet booster valve and start pump. At 200MPa, stop pump, close booster outlet valve and note pressure change. There is the typical adiabatic decrease in Pressure, but it should level off rapidly (30mins). Note the temperature before and after pumping.

If confining pressure system holds (check leak indicators) increase pressure using the intensifier. Check oil levels before use! Start intensifier pump until ~300MPa. Intensifier system should hold pressure. If not, close intensifier valve. Wait to make sure system is not leaking.

Increasing the temperature will cause a substantial pressure increase. If possible, keep the intensifier valve open (to add additional volume and buffer the effect). Stay with the machine and check on the confining pressure frequently.

Turn on water cooling system. Switch on Eurotherm controller (manually) and set ramp, set point. Slowly increase power settings of the furnace on top, center, and bottom (check values in Log Book).

After temperature equilibration, measure the temperature at the corresponding thermocouple location. The sample position in the column is between 80 and 106mm. Log profile and check maximum temperature and plateau. Adjust final controller setting to desires temperature. Calibrate thermocouples frequently!