QuickPress manual

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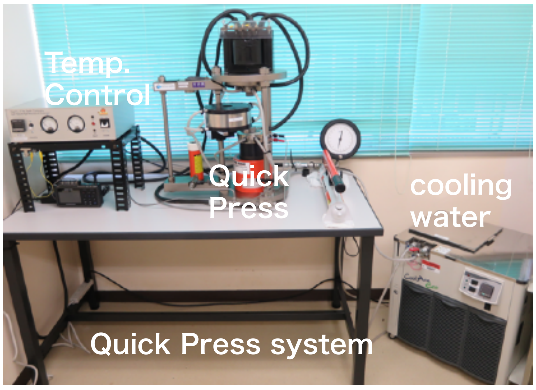
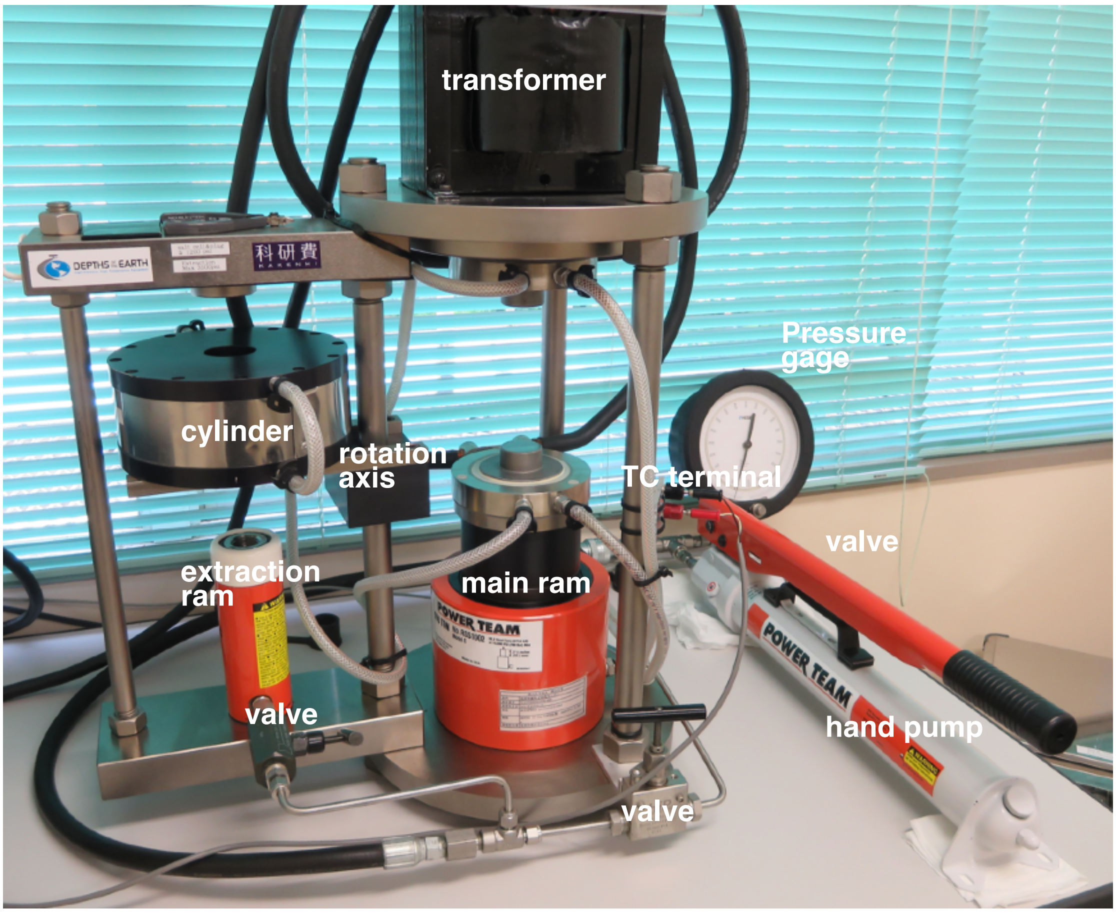
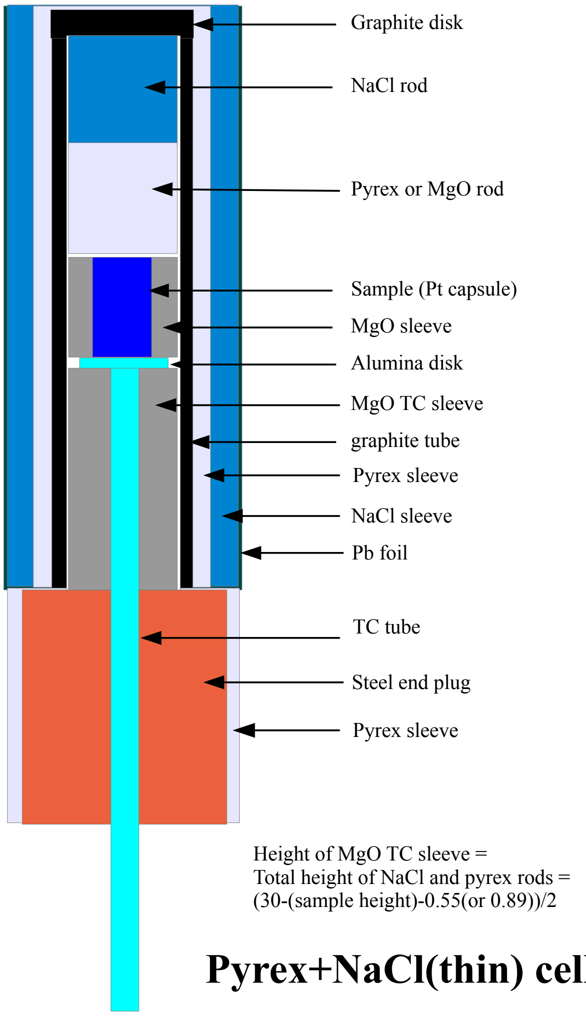


QuickPress is a piston-cylinder high-pressure device manufactured by Depth of the Earth, Inc. It was introduced with a Grant-in-Aid for Scientific Research by Prof. Xue. For more information, see the manual (in English, available in the room) from Depths of the Earth, Inc. The following is not a direct translation of the manual, but is written according to my actual experience with it and our laboratory setup. I also assume a standard configuration using NaCl cells; see the Appendix at the end for information on making NaCl sleeves and rods. We can use a talc cell as well, but we do not have the talc parts here. It used to be located on the 4th floor of the 2nd lab building, but has now been moved to the 3rd floor of the 3rd lab building.

Use for joint research and joint use is also possible.





Pyrex+NaCl(thin) cell

Preparation of high pressure cell parts

The entire cell is shown above. Some of the component parts are ready-made and can be used as is, but some parts need to be prepared by the user. The NaCl sleeve, NaCl rod, MgO sample sleeve, etc. The thermocouple wire and insulation tube are also necessary.

First, the NaCl sleeves and rods should be made before the experiment. Two types of sleeves are used depending on the experimental temperature: a thick NaCl sleeve is used for temperatures below 1000 ºC, and a thin NaCl sleeve (combined with a Pyrex glass sleeve) is used for temperatures above 1000 ºC. For temperatures below 1 GPa and close to 1000 ºC, it is better to use a thin NaCl sleeve because the melting point of NaCl is close, making the heating unstable. The cell figure above shows the case of a high-temperature cell using a thin NaCl sleeve. In the low-temperature cell, there is no Pyrex glass sleeve, and the NaCl sleeve is thicker by that amount, but there is no other difference. If NaCl rods are used as spacers, they should be prepared in advance. Already made NaCl parts are in the oven and may be used. The NaCl sleeves and rods need to be baked at 450 ºC for several hours beforehand, so they should be prepared first.

Many parts can be used without modification, but the length of MgO parts around the sample must be adjusted for each sample. In our laboratory, we usually use samples sealed in platinum tubes for experiments. The diameter and length of the platinum tube after sealing are measured with calipers (and recorded in your notebook). The diameter is determined by the jig, which is about 0.2 mm larger than the original Pt tube diameter. It is necessary to make a MgO sleeve for around the sample that matches it. If you can use the default MgO sleeve (for a sample diameter of 3.2 mm), you can shave it with a file to the same length as the platinum tube. A jig (brass cylinder with a 6 mm diameter hole) is available for grinding. If you do not have one of the appropriate size, you can make it yourself on a lathe. There are two types of crushable MgO rods, one of which already has a hole for a thermocouple insulation tube. Either one can be used. Cut it to the appropriate length with a thread saw and adjust the length with a sandpaper. A jig (brass with a 6 mm hole) is provided.

Prepare the MgO sleeve for the thermocouple and the MgO rod according to the length of the sample. The latter is adjusted in length depending on whether or not you want to use NaCl rods. The distribution of lengths depends on whether you want to place the sample in the center of the cell or the thermocouple in the center of the cell, etc., so design according to what suits your experiment. If the sample is placed in the center of the heater, the length of the MgO sleeve for the thermocouple is calculated in the figure above. The alumina spacer placed near the tip of the thermocouple comes in two thicknesses, 0.55 and 0.89 mm. If you are concerned about deformation of the sample by the thermocouple, use the thicker one. If you do not have a ready-made MgO sleeve or rod for the thermocouple, you can make the MgO rod on the lathe, cut it to the appropriate length using a thread saw, and adjust the length with a sandpaper. Adjust each so that the combined height of the sample and all sleeves and spacers is 30 mm.

I don't usually use it, but Pyrex glass rod is also available. However, this is a long rod and cannot be machined on a lathe, but must be cut with a diamond cutter.

Note that for samples containing iron, Au or PtPd alloys can be used since iron is absorbed by the Pt capsule, but note that Au has a low melting point. Graphite or hBN (boron nitride) can be used if sealing is not required and reaction with sample is negligible. The hBN sold at Nilaco has an outer diameter of just 6 mm, which matches the inner diameter of the graphite heater. hBN is an insulator, so there is no problem if it comes in contact with the heater. Kanzaki has some in stock. In the case of graphite capsule, if the outer diameter is set to 6 mm, the graphite heater will be in contact with the hBN, causing the current to flow through it and changing the temperature distribution (although this would make better temperature distribution). If this is a problem, a thin MgO sleeve between two graphite layer might be used.

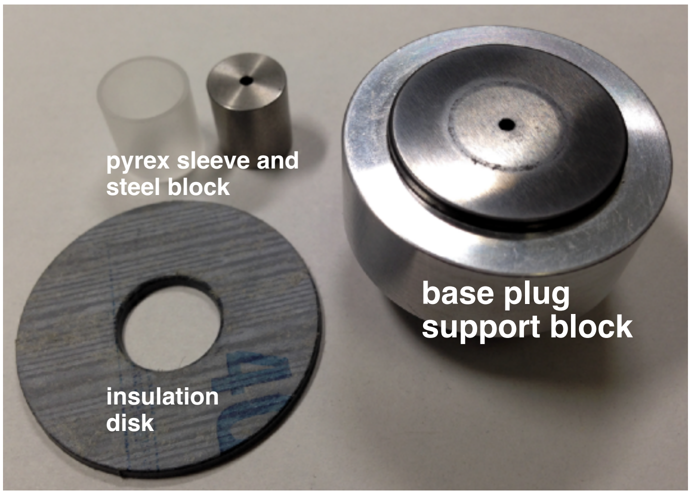
As for temperature distribution, ready-made graphite heaters are straight, so there is a relatively large temperature distribution (high at the center). If the temperature distribution needs to be reduced, the simplest solution is to make the sample smaller, but if this is not possible, a flat temperature distribution can be achieved by making the heater step-like (thicker in the center). A simple solution is to use an off-the-shelf graphite heater as is and add another graphite sleeve in contact with the graphite heater around the sample area. In this case, the usable space for the sample is smaller (in the radial direction). Another option is to use a high-melting-point metal foil, in which case the sample diameter is hardly affected. In both cases, additional processing such as MgO is required.

Due to the use of graphite heaters, the area near the sample is in a reducing environment. In the case of a talc cell, the talc seems to maintain a relatively oxidative environment, whereas in the case of a NaCl sleeve, it becomes reductive. If an oxidizing environment is required, hematite (Fe_2O_3) rods are used near the sample, and the length of the MgO and NaCl rods is adjusted by shortening them. Hematite rod is made using a "cell extrusion ram" in the same way as NaCl rods. A length of about 3 mm could be shaped without the need to add any particular additives; bake at 500 ºC for a few hours in air and store in a 150 ºC oven.

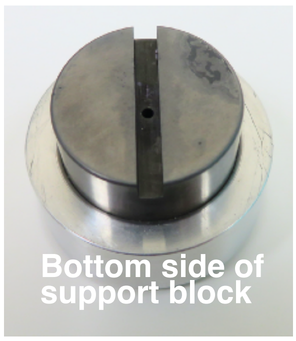
After adjusting the length, MgO parts are baked in an electric furnace at 1000 ºC for 1 hour (if you are concerned about adsorbed water, etc.) or heated in a gas burner until red hot (if you are in a hurry) to remove water. After cooling, these parts can be used immediately or stored in a 150 ºC oven.

The thermocouple insulation tube (4 holes) is available and needs to be cut about the length of the MgO sleeve for the thermocouple tube + 35 mm, but it should be a few mm shorter than that length to prevent the thermocouple from breaking during pressurization. Scratch it with a diamond file to get the required length, and then break it by hand. Flatten both ends with a diamond file while also adjusting the length.

For thermocouples for temperature measurement, we usually use 0.2 mm diameter W_5Re_{95}-$W_{26}Re_{74}$ (available from Nilaco). The temperature controller we use does not support W_3Re_{97}-$W_{25}Re_{75}$, so we cannot use W_3Re_{97}-$W_{25}Re_{75}$, but make no mistake, as we use W_3Re_{97}-$W_{25}Re_{75}$ in our multi-anvil experiments and those wires are also stored in another laboratory. Platinum-rhodium thermocouples can also be used, but in that case the temperature controller settings need to be changed. If a data logger is used, the settings for the thermocouple type must also be changed. Since the thermocouple wires are reused, the heavily deformed part of the thermocouple junction side from the previous experiment should be cut off, and the insulation tube for the thermocouple wire should be cut appropriately to make the exposed part necessary for insertion into the thermocouple insulation tube. The heavily deformed part is unusable, so cut it. If it is too short, order it to Nilaco or ask Kanzaki.



W_5Re_{95}-$W_{26}Re_{74}$ is not easy to weld, so the thermocouple junction is mechanically contacted by crossing the wires. For this purpose, a thermocouple insulation tube with four holes is used, the tip of the wire is made into a J-shape, and the straight part and the bent tip are inserted into two opposite holes of the four holes. The same is done with the remaining two holes, so that the two wires cross at the tip of the insulation tube and contact is made. This method has long been used by Kanzaki for multi-anvil experiments. In the case of platinum-rhodium thermocouples, this procedure is not necessary, the junction can be made with a laboratory welder. Note that the quality of WRe wire varies considerably from batch to batch. If the wire is badly wound or breaks easily when bent with pliers, avoid using it and use a new one or order a new one to Nilaco. The

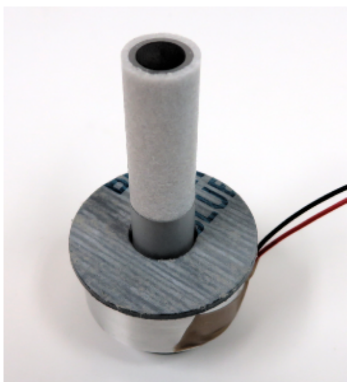


exposed part of the wire should be covered with a special red and black insulating tube (about 1 cm of the end should be exposed for contact). However, this tube is too thick and stiff to insulate the wires at the base plug where they are bent, so you can insert a thin, soft tube (used in multi-anvil experiments) between the alumina thermocouple sleeve and this insulating tube. However, I have never had a short circuit without doing this. Note that $W_{26}Re_{74}$ is red (+ side) and can be distinguished from the W_5Re_{95} wire under a binocular because it appears more brilliant.

Assembly of high-pressure cell

Clean the base plug support block (hereafter referred to as "support block") with acetone since it is dirty from the previous experiment. Stains that cannot be removed with acetone are removed with a razor or a sandpaper. Insert the thermocouple insulation tube (the one that already has the thermocouple ready) into the hole in the support block, run the wire in the groove, and fix the thermocouple cable with tape next to the support block with the required length sticking out at the top. Depth's manual says to fix with clay, but the insulation tube is hard, so it is impossible to fix it without tape. To avoid a short circuit at the exit of the hole, the insulated cable should be slightly inside the hole (the thermocouple insulation tube should be shortened). As mentioned earlier, the insulation cable supplied by Depth is hard and thick, so you can use a softer insulating tube for this part only (although there is no problem without it). Also, make sure that the fixing tape is not applied to the underside of the support block where the load is applied. Insert the base plug (steel plug) into the thermocouple protection tube placed in the support block. Cover the base plug with a Pyrex glass sleeve (the short one). This serves as insulation, and forgetting to do so will result in a short circuit, making heating impossible (there was actually a case in which a student forgot to do this and the heating failed). Cover the Pyrex sleeve with a disk with a hole in the center for insulating the base plug. Be careful not to put the sleeve and base plug directly on the insulating disk. If you forget to do this, you will not be able to heat. It is easy to forget to install the disk and glass sleeve, so make sure they are properly installed when assembling. The photo on the right shows the properly assembled condition.

Insert the MgO sleeve for the thermocouple into the exposed portion of the thermocouple insulation tube inserted in the support block. If the thermocouple protrudes from the MgO sleeve, adjust the position of the thermocouple protection tube so that the tip is just at the tip of the MgO sleeve. Place a thin alumina disk (available in two thicknesses) on top of it. Place the MgO sleeve with the sample on it. If there is a gap between the sample and the MgO sleeve, fill the gap with MgO or alumina powder (not necessary if the sample is shaped). Put a graphite heater over them so that they are enclosed (you can do this in any way that is easy to do). Insert the MgO and NaCl rods into the graphite heater. If the length is made correctly, the end of the NaCl rod and the end of the heater should



be about the same height. Cover this with a thick NaCl sleeve (for experiments below 1000 ºC) or a thin NaCl sleeve + Pyrex glass sleeve. The pyrex glass and graphite heater is quite tight, but should normally fit without breaking. Finally, a graphite disk is placed at the top edge. It may not be thick enough, but in reality it is fine. The figure of previous page shows how it looks so far (this example is a thin NaCl sleeve and pyrex glass), before wrapping the lead foil.

Wrap lead foil cut to about 39 mm wide (some are already cut) around the NaCl sleeve. Avoid overlapping. If there is an overlap, cut a little to adjust. The lead foil should protrude 1~2 mm from the top edge of the NaCl sleeve, and the protruding part should be carefully folded into the top side (and make sure no protrusion). Otherwise, the lead foil will be deformed and cannot be inserted into the cylinder. This completes the preparation of the high pressure cell.

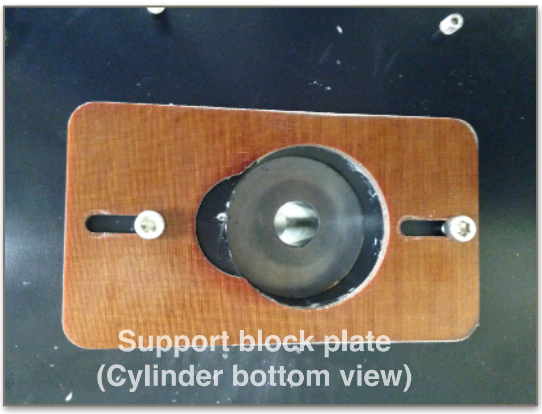
High-temperature high-pressure run

The maximum working pressure is 2 GPa (generally use below this pressure). Note that the supplied hydraulic gauge is in psi unit (pound square inch).

First, close (turn clockwise) the valve on the ram for extraction. There is no need to tighten it hard. Next, open the valve on the main ram side (counter-clockwise).

Rotate the cylinder to a position midway between the main ram side and the extaction ram for cell insertion, etc. Unscrew the support block plate on the underside of the cylinder, but position it

on the side where the block enters (see photo). Normally, it should be pulled toward the front. If you forget to do this, you will not be able to insert the base plug support block portion of the high-pressure cell properly. Apply a thin coat of Molykote to the inside surface of the cylinder hole with a cotton swab. If lead foil is not used, apply a thicker coat. Apply a thin coat of Molykote to the sides of the WC piston (a photo in next page).

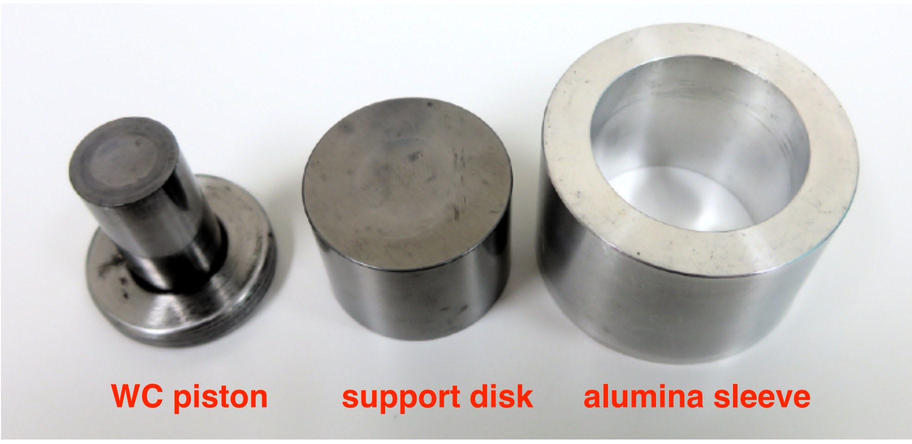


Molykote

Use a mirror to look into the lower part of the cylinder and check if there are any insulating disks left over from the previous use. If there is, remove it. It may be easier to turn the cylinder upside down. Note that if you do the experiment with this disc still in place, the steel plug part will stick out more, resulting in a larger deformation of the steel plug, which will make it difficult to remove the cell after the run (by normal procedure).

Next, insert the high-pressure cell into the cylinder. While holding the support block with one hand and the upper side of the lead foil of the cell with the other hand, slowly and carefully insert the tip of the cell into the cylinder hole from the lower side of the cylinder. The direction of the thermocouple wire should also be considered at that time. If force is applied in the horizontal direction, the thermocouple insulation tube will be broken, so always apply force vertically. If there is resistance or the lead foil shifts downward, stop insertion and pull out. The lead foil is now deformed, so use a new lead foil or, if the deformation is minimal, turn the top and bottom over and reuse the foil.

Insert the cell slowly and push it in until it cannot go up any further. Slide the support block plate on the underside of the cylinder and support the underside of the support block with this plate. If the place cannot slide, it is because the cell assembly is not fully inserted. Once fully inserted, tighten the screws on the support block plate to secure it in place. Look at the top of the cell from the upper side of the cylinder to confirm that the lead foil has not shifted out of place. If the lead foil has shifted, start over, but pull out carefully so as not to break the thermocouple insulation tube.



After successfully inserting the cell, insert the WC piston from the upper side of the cylinder. The piston support block (made of WC) and an aluminum sleeve for it are placed on top of the piston. The cylinder is rotated to bring it toward the main ram, but the ram must be lowered so that the pressure dispersion plate at the end of the main ram does not hit the cylinder support in the rotating section (it usually does during the previous experiment). Also, the cylinder needs to be shifted to the side during rotation. Care should also be taken in routing the water cooling hose during rotation (if it gets stuck, it will not turn). Move the cylinder so that the piston support block is roughly aligned with the center of the pressure dispersion plate that receives the upper block (find adjust later).

Route the thermocouple wires so that they do not hang over the top of the main ram and connect them to the red and black terminals (they should be the same color as the cable). Tighten the wires through the holes in the terminals. Note that if the tip of the banana plug of the data logger is too far into the terminal, it will block the hole in the terminal. Turn on the switch next to the temperature controller and check that the temperature reading is about room temperature (this controller seems to show a bit low room temperature). In the case of W/Re thermocouple, the junction is a mechanical contact, so sometimes the connection at the junction is not properly made at this point. Thus the thermocouple is not necessarily broken. In many cases, pressure will make contact; if the junction is welded, as in the case of a Pt/Rh thermocouple, failure to show room temperature at this point indicates a trouble, such as incomplete connection. Check the connections at the terminals. The temperature controller is usually set for the $W_5Re_{95}/W_{74}Re_{26}$ thermocouple, so change the setting if a different type of thermocouple is used.

valve of hand pump

Look at the lower part of the cylinder and confirm that the thermocouple is not resting on the support block part that is pushed by the main ram.

Confirm that the valve on the ram side for cell extrusion is closed and the main ram side is open. Close the valve on the hand pump. Raise the main ram with the hand pump until just before the piston support block contacts the upper board. Readjust the cylinder position here so that the piston support block is centered.

At this point, a defensive polycarbonate board is placed beside the QuickPress. This is to protect workers from flying debris when the piston is broken. The state of the polycarbonate plate when installed is shown in photo. The polycarbonate plate is left in place until the pressure is relieved after the experiment.

When using the “hot piston out” technique, first slowly pressurize to a hydraulic pressure equivalent to the planned experimental pressure +10% (see Table below). By using NaCl cell and hot piston out technique, it is believed that no friction correction is necessary. Since the pressure drops gradually, check the pressure value from time to time and keep it as constant as possible.

For experiments at 0.5~0.7 GPa, raise the pressure once to 1.5 GPa and then lower it, in order to get a good contact with the heater assembly.

P (GPa)	oil (psi)	+10% (psi)
1.0	1,450	1,600
1.5	2,175	2,400
2.0	2,900	3,200

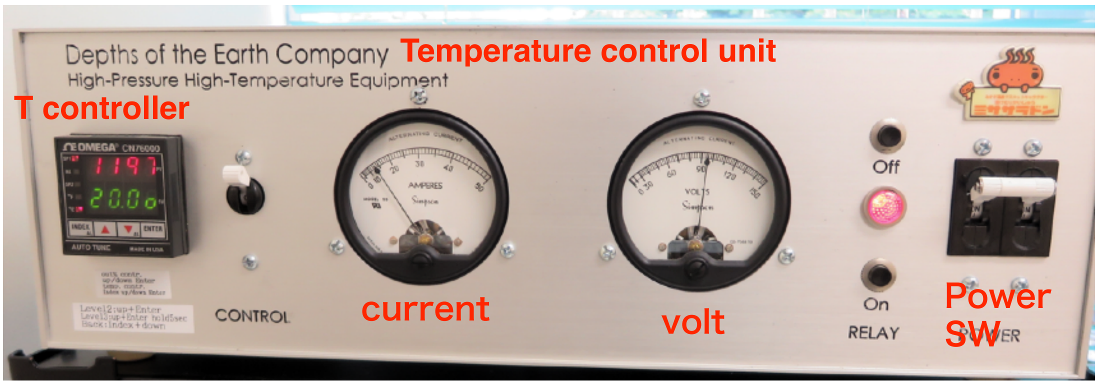
relation of pressure and oil pressure

Turn on the main power switch of the cooling water circulator (it is located in a position that is difficult to see from above). Then press the pump switch to turn it on. Finally press the cooling switch to turn it on. Do not touch the valve (red lever) to the piston cylinder side. If it is fully closed while it is in operation (do not put the lever at right angle to the line), as this will damage the cooling water circulation system. The temperature should be set at 20 degrees Celsius (default). If the water level is low, add tap water. Raise the temperature when there is a lot of dew on the cylinder (this may happen in June and July). Turn on the room cooler except in the middle of winter.

[To use the data logger, turn on the data logger. 1ch is set to measure temperature with $W_5Re_{95}/W_{74}Re_{26}$. The temperature can be monitored on the LCD screen as it is, but it is not recorded (free running state). To record data, press the Start switch. To stop recording, press the Start switch again].

Start heating. First, turn on the main power SW on the right end of the temperature control unit (to the upper side). Turn on the SW next to the temperature controller as well. In this state, heating is not yet possible. The green number on the underside of the temperature controller must be less than 5.0. If larger than 5.0, lower this below 5.0 by pushing down arrow key, and then press ENTER key.

Then, press the On switch at the RELAY on the left side of the main power SW. The lamp should light up. The lamp will not light unless the water hydraulic relay is on (means cooling water is not running). If the light does not come on, check to see if the coolant circulator pump is running. The



green lamp (upper one) of cooling water circulator should be lit, or if not, press the switch for cool (upper one). Now the heating is possible.

If the temperature controller is controlled by % output (that is default, read the original manual), use the up arrow key to increase the output to 5%, then press the ENTER key. Below 5%, the actual output for heating is zero. Above 5%, the temperature should increase. The output change is not reflected unless the ENTER key is pressed. If the temperature drops at the beginning of heating, the polarity of the thermocouple is reversed, so reconnect it (this should not normally happen when using recycled wires). As the temperature rises, the load will drop at first, so keep the hand pump at the experimental pressure +10%. While increasing the output %, record the time, oil pressure, temperature, current, voltage, output %, and water temperature (displayed on the front panel of the cooling water circulator) in the log notebook at appropriate intervals. Note that if the water temperature does not drop to the set temperature, no cooling is taking place. Check if the chiller is running. If the green lights (upper and lower) is off, press the pump and cooler switches. Also, referring to the results of previous experiments, check whether the relationship between heating power and temperature is the same as in previous experiments (with same NaCl sleeve type). If the temperature is significantly low, there may be a short circuit in the thermocouple wire taking out area (in the support block). If the temperature is abnormally high and dizzying, it is also likely that the thermocouple has broken. In this case, the experiment can be continued by estimating the temperature using past power-temperature relation, and short-circuit both poles of the terminal to which the thermocouple is connected with banana plug wire (available in the area).

When approaching the experimental temperature, adjust the output to bring it closer to the experimental temperature. Once the experimental temperature is reached, open the valve on the hand pump very slowly to lower the excess 10% to reach the experimental pressure. This is “the hot piston out” technique. This is expected to cause the piston to move outward, which is thought to reduce friction.

During the experiment, the resistance changes due to deformation of the graphite heater, etc. The output % is changed so that the temperature is adjusted to be close to the experimental temperature. Also, the pressure usually increases a little bit with time, so it should be adjusted. In case of prolonged holding, the oil tends to leak a little and the pressure drops a little. In that case, use a hand press to restore the pressure.

As for the stability of the temperature, the graphite heater used may be a characteristic of the graphite heater, but its resistance changes considerably over time at temperatures above 1000 ºC. Therefore, it is necessary to watch it from time to time and adjust it.

When the holding time has been reached, we normally quench the run by pressing the Relay Off switch or set the output to 0% and press the Enter key. Since the pressure drops during quenching, if you want to quench under isobaric pressure condition (when relaxation due to pressure drop becomes a problem, such as when synthesizing hydrous glass), press the hand pump immediately after quenching and keep the pressure for a while.

If you take it out immediately, the base plug, etc. will still be hot, so it is better to wait a few minutes after quenching while circulating cooling water. Open the valve of the hand pump and slowly reduce the pressure. When the pressure reaches zero, the ram will start to drop. The screws protruding from the bottom of the cylinder will be displaced and rest on the cylinder support fork. The screw protruding from the bottom of the cylinder rides misaligned on the cylinder support fork, so shift it so that it rides properly. When the top of the ram's pressure distribution plate is lower than the rotating support fork, close the hand pump valve.

Reduce the output to 5% or less for next time if not done yet. Turn off the temperature controller. For the cooling water circulator, turn off the cooling switch, turn off the pump, and turn off the main unit's power switch. Note that during the summer, if the cooling water circulation system is not turned off for a while, dew will form on the cylinders and other parts of the device.

Extracting the cell

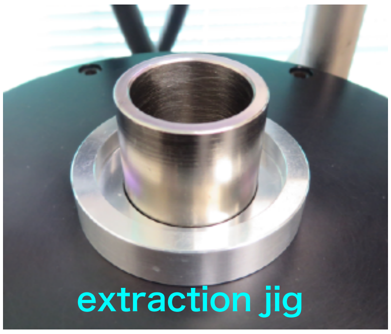
First, remove the thermocouple wires from the terminals.

Cover the top of the ram for cell extrusion with plastic wrap as shown in the photo on the right to prevent NaCl and other substances from falling into the ram. Attach the extrusion piston to it. Note that there is also a thin sleeve on the piston. Some wrap does not stretch well, so use proper wrap.

Rotate the cylinder part and bring it to the front side of the device. Loosen the screws on the support block plate on the underside of the cylinder, shift the plate, and remove the support block. Note that it may still be hot. To remove the support block, it is necessary to cut the thermocouple wire, so use the nipper to cut the thermocouple wire as close to the base of the base plug as possible. It may be easier to do this by turning the cylinder halfway vertically.

Completely shift the support block plate (to the front side), and secure it with screws. If this is not done properly, the plate will be broken during cell extraction.

Remove the piston support block and aluminum sleeve from the top of the cylinder; the WC piston itself cannot be removed by hand. Fully close the valve on the main ram. Fully open the valve on the ram for cell extraction. If the ram for cell extrusion is not fully down, open the valve on the hand pump. If it is still up, the piston for extrusion will collide with the rotary fork.



Attach the pipe of the cell extrusion jig to the top of the cylinder and the jig of the aluminum ring that fits into the top (the larger aluminum one is oriented so that the concave side is up). See the photo on the right. Rotate the cylinder section to move it directly above the extrusion ram.

Raise the extrusion piston with a hand pump and stop it just before it contacts the cylinder. While looking in the mirror provided, fine-tune the cylinder position so that the tip of the jig is centered on the base plug that is visible from the cylinder.

The cylinder is further raised with a hand pump and adjusted by supporting it by hand so that the ring of the jig fixture fits into the protrusion on the top surface of the extraction. When the pipe reaches the top, check the piston position with a mirror. If there is any misalignment, adjust the piston position. Place a polycarbonate plate for protection. Pressurize and push out the pipe, but do so at no more than 3,500 psi. Normally, the cell and piston are pushed out a little at a time at less than 3,500 psi.

When pushing the hand pump produces little or no hydraulic pressure, the cell should be pushed out, so open the hand pump valve and turn the ram down all the way. Conversely, if the hydraulic pressure starts to increase, that means the piston and cell are bridging and being pushed. When you want to take out the cell without deforming it too much, stop once the oil pressure stops falling, retrieve only the piston, and push out the remaining part again. Place a box to catch what falls down from the cylinder, and move the cylinder toward you. If it is not completely pushed out, repeat the operation.

Once completely extruded, remove the extrusion piston etc. At this time, the jig attached to the ram for extrusion should be removed with its wrap. Collect the piston and cell. At this time, identify the fragment which contains the sample and be careful not to discard it as waste.

If the extrusion ram cannot extrude even at 3,500 psi, contact Kanzaki. To extract, use a special short piston and short pipe with main ram. Turn the cylinder over and place a jig (a black polyacetal disk with a hole) on top of the cylinder to center the piston. Place the piston in the center hole of this shaft. Insert a short extrusion pipe from under the cylinder and rotate it toward the ram while holding it down. Raise the ram and stop it before the tip of the piston contacts the upper pressure dispersion plate. Align the center and apply pressure to push the piston out, but place a polycarbonate plate for protection since the piston may break. After some amount of extraction, return to the ram for extrusion and extract it completely.

Note that current cylinder (#2) is slightly off-center, so if a jig is used to place the piston on center, the piston will be off-center and seem to contact the cylinder. To avoid this, do not use the jig mentioned above, but fix the piston to the center with tape and do the above work. In fact, a piston has been destroyed during this operation.

Sometimes the disk for base plug insulation remains stuck to the underside of the cylinder. Turn the cylinder over and remove the disk with a flat-blade screwdriver or similar tool. Wipe the cylinder with acetone. Clean the inside surface. Clean the inside surface with a file if necessary.

Collect as much lead foil as possible and place it in the glass bottle provided. Thermocouples should also be collected. The stainless steel base plugs should also be collected. Other items (including NaCl) may be thrown in the trash.

Since NaCl and other substances will be scattered around during the work, try to keep them as small as possible. After run, clean the equipments and desk well. In particular, remove any NaCl powder. A small vacuum cleaner is placed under the desk.

Wipe the used jigs, pistons, and cylinders with acetone. The jigs should be dried and, if necessary, sprayed with rust inhibitor to prevent rusting. The piston and cylinder should be observed carefully to make sure there are no cracks, etc., and the surfaces should be cleaned by rubbing with a sandpaper. Store them in a desiccator.

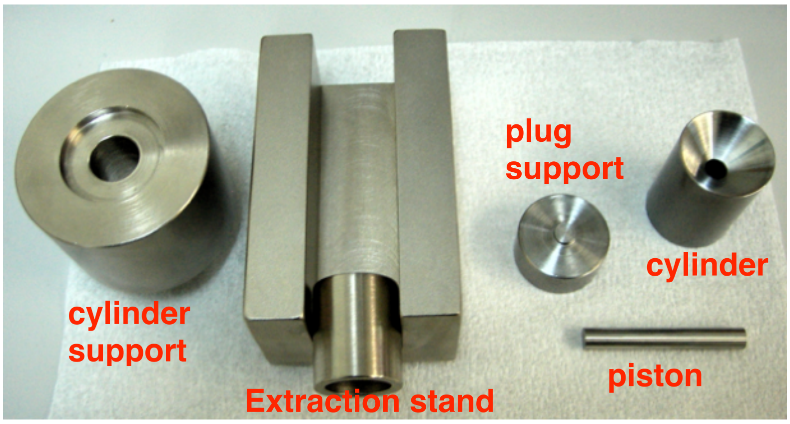
Keep the thermocouple wires as they will be reused. Turn off the air conditioner and lights.

Appendix 1

Procedure for making NaCl plugs (rods)

1. This is required only when used as a spacer for high pressure cells. Diameter is 6 mm. Length can be adjusted by weight.
2. Dry NaCl at 150 ºC for 1 day before weighing. Dried product is in the oven.
3. Weigh one plug of NaCl and place it in an aluminum dish. Approximate weight: 0.367 g for 6 mm length; 0.50 g for 8 mm length.
4. Cover the extruding ram with plastic wrap to protect it from NaCl.
5. Find the tools for making. The necessary tools are shown in the picture below. The cylinder support (far left) and the extrusion stand (center) are also used for making sleeves. These are stored in a desiccator.
6. Kimwipe (or equivalent) is tightly packed in the holes of the cylinder support (to prevent the piston and the ejected plug from falling out).
7. Apply a light coat of Molycote (white) to the piston and the inner surface of the cylinder for making plugs.
8. Set the cylinder on the plug support on the desk.
9. Pour NaCl from an aluminum dish into the cylinder and lightly tap the sides to drop the NaCl powder down.
10. Insert the piston into the cylinder hole.
11. Set the extrusion stand on the extrusion ram. Place the cylinder support on it. Check that they are centered on the ram.
12. Set the cylinder etc. on the cylinder support (see photo).
13. Close the valve of the hand pump. Close the valve of the main ram. Open the valve of the ram for extrusion. Using the hand pump, raise the ram until the piston hits the upper plate.
14. It is important that the piston is vertical (especially when making long plugs). Do not apply force to the piston in an odd position. It may break and fly off. Install a polycarbonate plate for protection.
15. Raise the ram until the piston enters 0.5 cm. Now open the valve of the hand pump to release the pressure and lower the ram slightly.
16. Close the hand pump valve again and pump up to 1,000 psi while watching the pressure gauge. Hold at this pressure for 1 minute. Do not exceed 1,200 psi or the piston may rupture and splatter.
17. Open the valve on the hand pump and lower the ram slightly.
18. Remove the plug support and the cylinder, but return the cylinder only, and adjust its position.
19. Close the valve on the hand pump and raise the ram. You will hear a noise as the piston is pushed down. Raise the ram until the plug is completely out of the cylinder. The plug may not drop down and stick to the piston.
20. Measure the length of the plug and adjust the weight to be weighed if it is different from the expected length. Repeat this process to make the required number of plugs.

21. The plugs are baked in a muffle furnace at 450 ºC for 1 to 4 hours to recrystallize them and evaporate the lubricant.
22. Keep the baked plugs in the oven at 150 ºC until they are used in the experiment.
23. When finished, carefully wipe all tools with acetone. Spray the tools with rust inhibitor spray and wipe off any excess oil.
24. Clean up the NaCl-sprayed area well, including the area around the extrusion ram. I keep a mini vacuum cleaner under the QuickPress table.
25. Do the same when making hematite disks, up to 3 mm thick, which can be shaped without additives and with little or no cracking.
26. There is a power outage once a year due to security inspections, and in advance of the power outage, I evacuate NaCl parts to a vacuum oven or a desiccator that can be evacuated to a vacuum. After the power outage, the parts are returned to the oven at 150 ºC.



Appendix 2

Procedure for making NaCl sleeves

1. Dry NaCl at 150 ºC for 1 day before weighing. The dried product is in the oven (maybe).
2. Weigh 1 sleeve of NaCl and place in an aluminum dish. Approximate weight: 2.45 g for high temperature; 4.40 g for low temperature.
3. Cover the extrusion ram with plastic wrap to protect it from NaCl.
4. Find the tools for making the sleeve. They are stored in a desiccator. The necessary tools are shown in the picture. This is for a sleeve for high temperature. Note that different sets of mandrels, rings, and guide rings are used depending on the thickness of the sleeve, but the procedure is identical.
5. Pack the kimwipe (or equivalent) tightly into the hole in the cylinder support and set it on the upper side. This is to prevent the mandrill from falling out.
6. Apply a light coat of Molycoat (white) to the core, piston, and inner surface of the cylinder. Do not let the Molycote get on the end of the core rod (the side that is filled with NaCl) and the funnel.
7. Place the mandrill guide ring on the cylinder support on the desk, and place the mandrill inside the guide ring. Place the cylinder over it. If the mandrill is too low, push up the Kimwipe from under the cylinder support.
8. Place the funnel on top of the cylinder.
9. Pour NaCl from an aluminum dish into the cylinder slowly and tap the sides lightly to drop the NaCl down. The thick sleeve does not have much problem pouring, but the thin one does not get the NaCl into the gap well, so you need to use a thin stick or something to fill the gap with NaCl. If this is not done properly, the finished product will break.
10. Remove the funnel.
11. Insert the ring from the top of the cylinder, and confirm that the mandrill enters the ring. Insert the piston into the cylinder from above.
12. Place the complete cylinder set on the extruding ram (see photo at right). Lower the ram if necessary. At this time, the extraction stand is not necessary.
13. Close the valve of the hand pump. Close the valve on the main ram. Open the valve of the ram for extrusion. Using the hand pump, raise the ram until the piston hits the upper plate.
14. It is important that the piston is vertical. Do not apply force to the piston in an odd position. The piston may break and fly apart.
15. Raise the ram until the piston enters 0.5 cm. Now open the valve on the hand pump to release the pressure and lower the ram slightly. Install the polycarbonate plate for protection.
16. Close the valve on the hand pump and hand pump up to 1,200 psi while watching the pressure gauge. The piston will stick&slip and the pressure will drop, but raise to 1,200 psi regardless. Hold at this pressure for 1 minute, but do not exceed 1,200 psi.
17. Open the valve on the hand pump and lower the ram.

18. Remove the entire set from the extrusion ram, and remove the cylinder support and mandrill guide ring. If the guide ring cannot be removed, use a monkey wrench to rotate the guide ring and remove it.
19. Place the extrusion stand on the ram. Place the cylinder etc. on it. Adjust it so that it is centered.
20. Close the valve of the hand pump, raise the ram until the piston hits the upper plate, and advance the ram further. A loud noise is heard. Raise ram until sleeve is fully visible. Lower the ram, remove the mandrill from the cylinder, and take out the sleeve.
21. Measure the length of the sleeve and adjust the weight if it is different from the expected length (approx. 31.8 mm).
22. Clean the piston, cylinder, etc. with acetone and apply Molycote for the next sleeve. Repeat this process to make as many sleeves as needed.
23. Bake the finished sleeves in a muffle furnace at 450 ºC for 1 to 4 hours to recrystallize them and to evaporate the lubricant. For high-temperature sleeves, pyrex glass sleeves are inserted into the NaCl sleeves before baking. This is to prevent the thin sleeve from being deformed by heating. For thicker low-temperature sleeves, the sleeve can be baked as is.
24. Place the sleeve in a glass bottle and store in a 150 ºC oven.
25. When finished, carefully wipe all tools with acetone. Place the tools in a desiccator.
26. Clean up the NaCl-sprayed area well, including the area around the extrusion ram; a mini vacuum cleaner is placed under the QuickPress table.
27. Other than pure NaCl: I have tried in the past to make NaCl mixed with hematite and $Mg(OH)_2$ (which made thin sleeves with no problem) and CaF_2 sleeves (only managed to make the thicker ones).