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Overview

Karma is a high pressure, high temperature isostatic furance that is capable of 1300 C at just over 60 kbar of pressure.

PREPARING YOUR SAMPLE

Your sample in Karma will be contained in a gold capsule (see Appendix for Boron Nitride capsules), which you must prepare. The **SAMPLE SIZE** should be about 0.25 grams, enough powder to adequately fill the gold capsule. The capsule is then placed in an alumina sleeve which in turn sits in a tubular graphite furnace. The assembled furnace sits in a "lava brick" that provides isostatic application of the pressure to the sample.

PREPARE THE GOLD CAPSULE

To prepare the capsule, you'll need a gold tubing sleeve which should be 8 mm in length and 5 mm in diameter with walls of 0.127 mm. You'll also need two gold discs, which are small enough to fit inside the sleeve.

First, make the bottom of the capsule by suspending one of the gold discs about 1 mm from one of the sleeve edges (or 7 mm from the other edge) and folding the end of the gold sleeve (which is malleable) over the top of the disc using a hard edge, like the back end of a pair of forceps. Using the thicker end of the special Karma steel rod, and the designated die, press the disc and the folded edge of the sleeve together (with the disc on the inside of the gold sleeve) so that you now have a hollow gold cylinder with one side open.

Spoon your sample into the gold capsule packing it in densely with the steel rod. You may want to place the steel rod on top of your sample, pack the sample by hand, and then tap the end of the rod with a hammer to pack a little more. Once you've packed your sample into the capsule (leaving about 1 mm of space on top), place the other gold disc on top of the powder. Be sure to blow off your sample powder from the disc and inside of the gold tubing. Again fold the capsule edges over the disc, and press the entire capsule together (this time using the rod that has only one diameter) to make your sealed gold capsule. Inspect your capsule under the microscope to check for rips or holes.

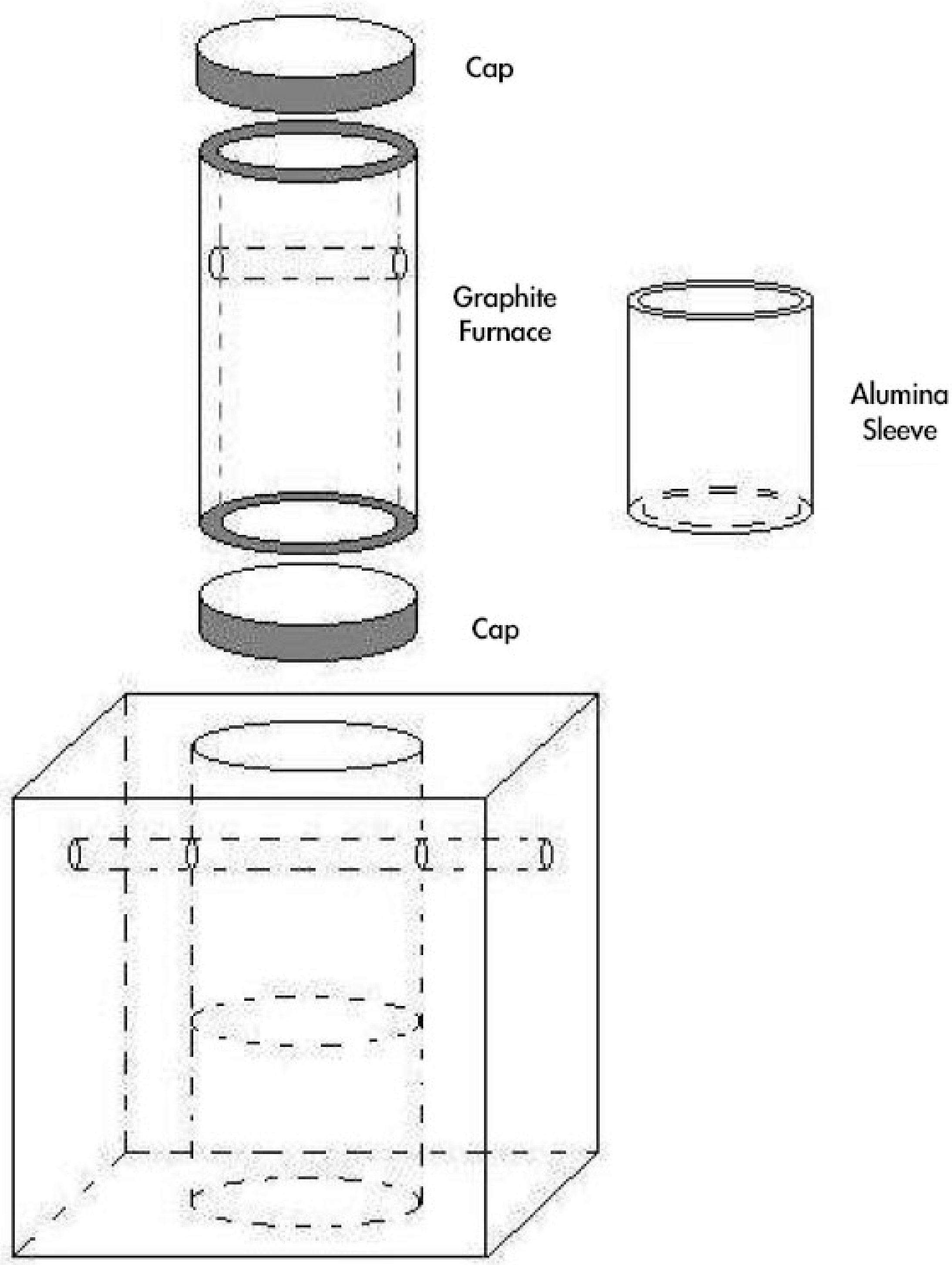
PUTTING YOUR SAMPLE IN THE GRAPHITE FURNACE AND LAVA BRICK

If you examine Figure 1, you'll see that the lava brick has a hole running from top to bottom, and another hole from side to side. The top to bottom hole is centered, while the other hole is not.

The alumina sleeve will fit inside the graphite furnace, but due to variation, the first thing you should do is to check that you have an alumina sleeve that easily slides into your graphite furnace.

Apply a minimal amount of adhesive around the inside of the large hole at the bottom of the lava brick. Place the bottom cap inside the hole at the bottom of the brick. It should stick in place. Slide the graphite furnace into place, making sure that the side holes in the graphite furnace line up with the holes in the brick and that it contacts the bottom cap. Avoid lifting the graphite furnace during the remainder of the sample preparation steps, to keep contact between it and the cap. Remember that the graphite must conduct electricity to heat, and does so through the end caps.

Figure 1: Schematic of Lava Brick Holder for High Pressure Samples



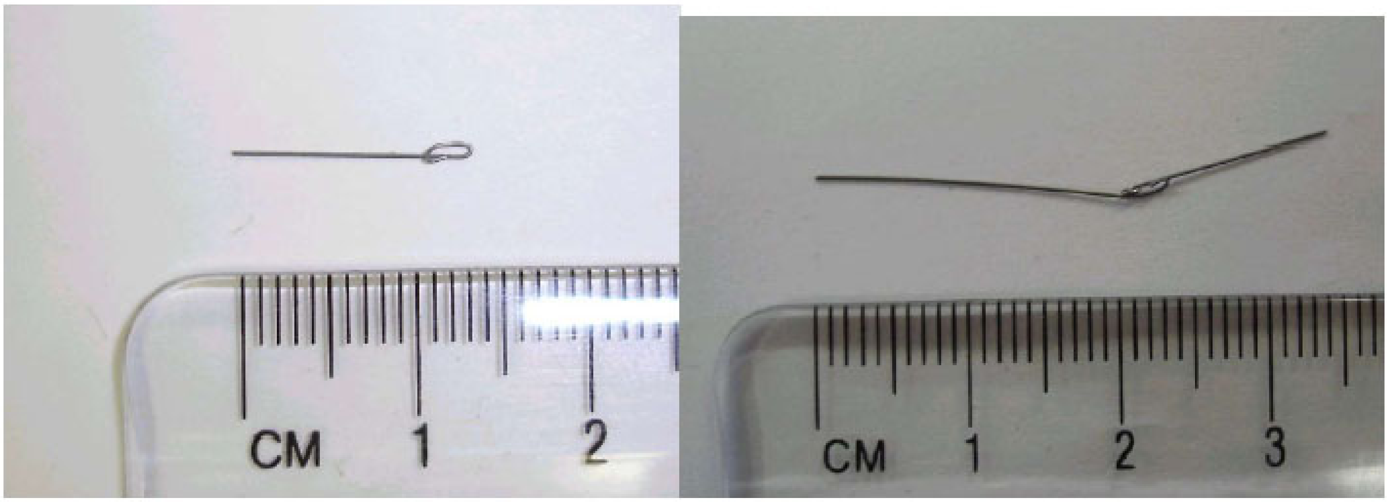
Next, you must put an MgO layer in the bottom of the graphite furnace. The MgO layer is actually 3 layers, from bottom to top: a layer of MgO powder, a solid MgO disc, and a layer of MgO powder. The height of this MgO packing needs to be such that when you put the Alumina sleeve on top of it (next step), the top of the Alumina sleeve will be just below the side to side hole. You should also pack the MgO powder in, perhaps with a steel rod.

Next, place the gold capsule inside the Alumina sleeve, and then slide both into the graphite tube, on top of the MgO layer, making sure that there is room between the side to side hole and the top of the Alumina sleeve.

After that comes the **THERMOCOUPLE**. The thermocouple is made of two tungsten wires designated red and blue. The red wire is an alloy with 3% Re, and the blue wire with 25% Re. The direction is important, so remember which is which. There should be some wire already clipped to the appropriate length. Take one of the wires, and form a small loop (Figure 2, left) using a pair of small pliers. Slide the other wire in through the first loop, and then make a loop in the second wire so that the two wires are looped together (Figure 2, right).

It is important for the loops to be small and the connection to be tight so that electricity can flow through the wire. Small loops help prevent MgO powder from disrupting the connection.

Figure 2: Thermocouple



On the left is one of the wires looped (a little too big actually). On the right are both wires looped together.

Slide the thermocouple into the side to side hole, making sure that the loop is centered over the sample. You'll want to test the connection of the thermocouple with the Ohm-meter. Slide two insulating wire covers (Figure 9) over the wire on both ends so that they insulate the thermocouple from the graphite tube.

Once the thermocouple is in place and insulated, you'll need to make another MgO layer. This time, scoop powder almost all the way to the top of the graphite furnace (again packing it down) so that you can place another solid MgO disc on top of the powder and flush with the graphite tube. Remove all MgO powder from the top surface of the graphite tube, and place the top graphite cap on the tube so that there is contact between all three graphite pieces.

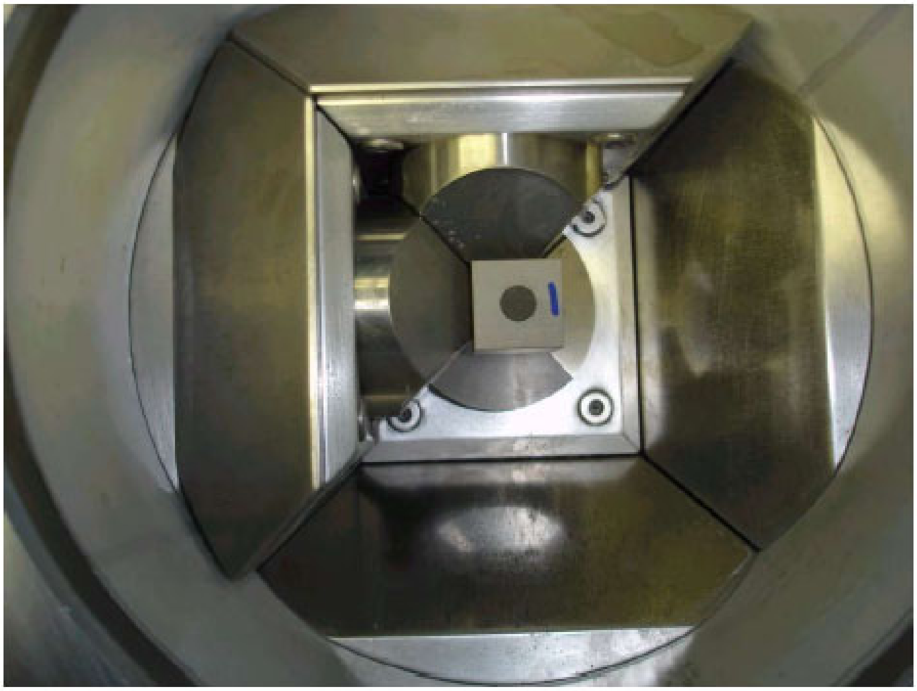
Bend the ends of the thermocouple down along the side of the tube. You'll want to test the connection again with the Ohm-meter.

SQUEEZING AND HEATING

Make sure you know which side of the lava cube has the blue wire, and which is the red. Clean all of the anvils with steel wool. Now you'll start to assemble the anvils. Before putting your anvils in, spray them with fluoroglide. Make sure that the proper Mylar sheets are in place to insulate the anvils from the ring (one on the bottom with connecting metal place in the center, two around the inside rim (see section labeled Mylar Sheets). Inspect the Mylar for holes, which can be patched.

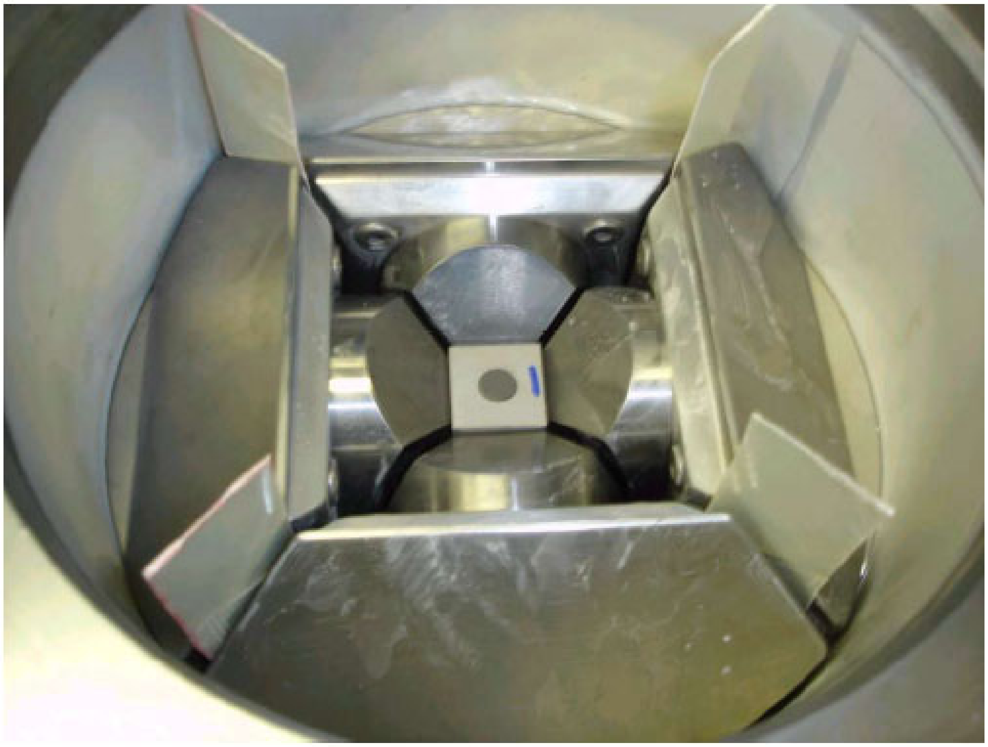
Assemble the bottom five anvils, placing two of the outer ring anvils directly on top of the copper leads (these are for the thermocouple circuit). For the bottom center anvil, clean the fluoroglide off of the bottom, and also clean the square which contacts the sample, because this will conduct the electricity to the sample for heating. For the middle layer, you'll want to put in two anvils next to each other and then put in the sample (Figure 3). The blue line in the picture marks the right side if you're facing Karma. The blue thermocouple wire should be facing the right side (**NOTE:** for the two anvils contacting the thermocouple, wipe the fluoroglide off of the contacting surface so that there is electrical contact to the thermocouple).

Figure 3



Put in place the next two anvils. You'll have to fiddle with it a little to get it to work, because the brick is slightly larger than then space created by the anvils. There should be four plastic rectangles available. Slide these in between the middle and bottom layers of anvils, to insulate them from each other (see Figure 4).

Figure 4



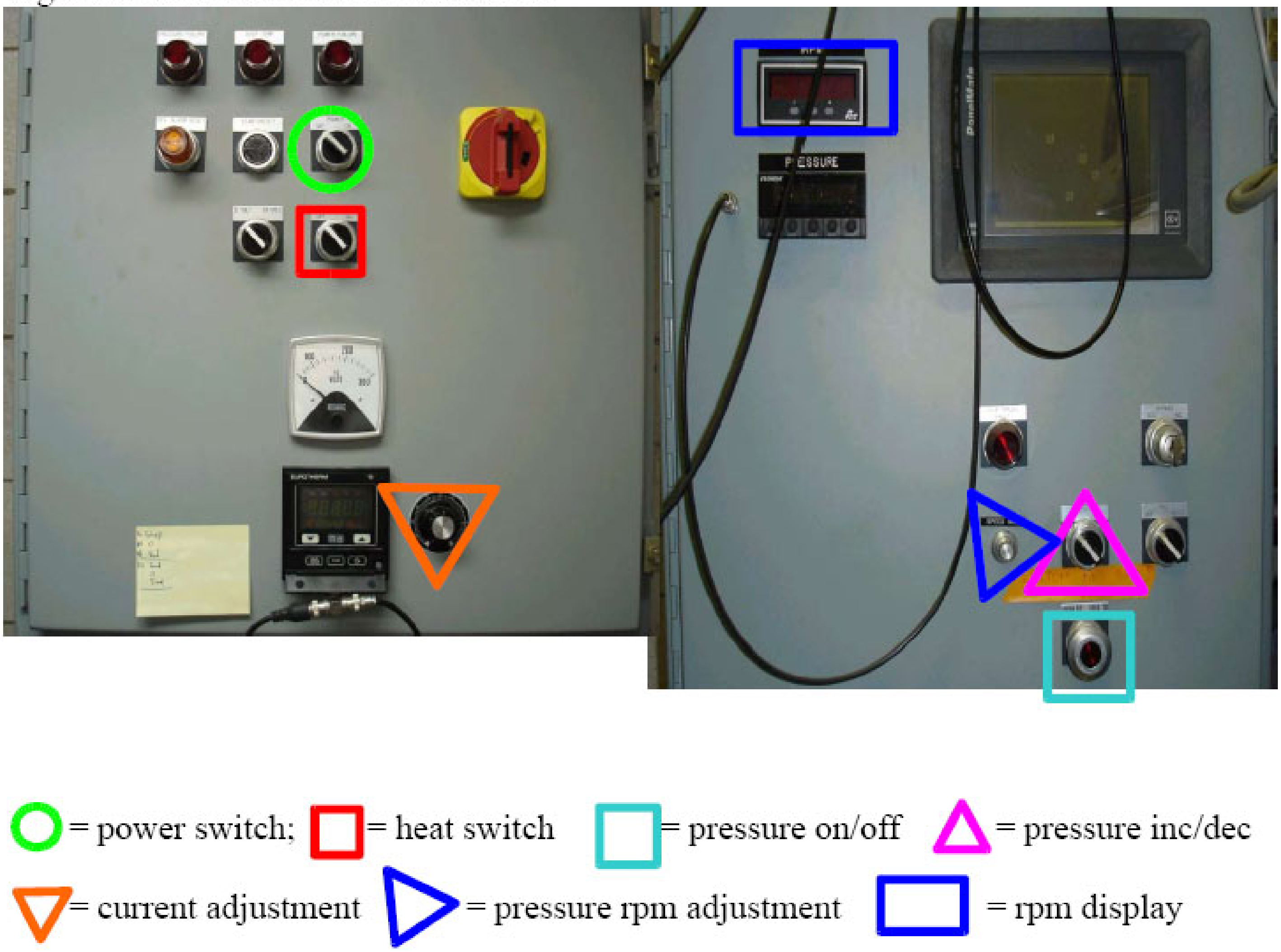
Put in the top layer of anvils. The four outer ones should be insulated by the four strips you just put in, and don't forget to wipe off the sample contact site for the top center anvil as well. Place the another circular Mylar sheet over the top, with the metal sheet sitting in the hole in the middle to complete the heating circuit.

Figure 5

Next, place the big metal disc (Figure 5) on top of the anvils, with larger surface pointing up, and check that it looks flat, or level with the huge blue cylinder which now contains your sample.

Now you'll have to roll the sample chamber directly underneath the gray steel cylinder press in the center of Karma. Just pull up on the two lock pins in order to move the sample chamber, and replace them once the chamber is in the right spot. There are only two positions that the chamber can be locked in. WARNING: don't run over the thermocouple wires with the wheels of the sample chamber.

Figure 6: Power and Pressure switches



Now you'll want to turn the power switch on (Figure 6). If the controller shows a temperature reading of ~ 22, that means your thermocouple is still intact! Next turn on the pressure switch (Figure 6). To increase the pressure, you'll start with the Enerpac. Make sure that the handle on the Enerpac is all the way to the up position (Figure 7) which is the increase mode (Its labeled). Open the pump valve (Figure 7) several turns.

For the Enerpac, there is a single button controller attached to the Enerpac with a long insulated wire, and usually draped over the pump valve. Use this button to increase the pressure. You'll see the whole sample chamber start to lift up slowly. When it almost contacts the cylinder its inching towards, you'll want to increase the pressure in small spurts, so as not to damage the thermocouple. You'll here creaking, which is normal, as contact is made and the pressure increases. After ~ 25 tons of pressure, you can just hold the button down and go. Raise the pressure to ~ 150 tons (from 100 � 120 you'll here a weird noise, don't worry). At this point you'll want to close the pump valve, and then release the pressure from the Enerpac by pulling the handle towards the down position about 10 degrees, and then returning it to the up position.

Having programmed in your desired pressure into the controller (if you don't know how to do this, seek help), and with the pump valve closed, switch the pressure to increase (Figure 6), and turn the pressure rpm to 600 (Figure 6). Your pressure will increase to the set pressure. Again, creaking is normal. At this point, be sure so set up your reaction monitor on the computer next to Karma (instructions not shown).

Once Karma reaches the set pressure, turn the heat switch to on (Figure 6), set the current adjustment knob to 36 A(Figure 6), and then run the program that you've set for the temperature run (no longer than 4 hours dwell time). The temperature controller is just like the ones for the tube furnaces in the main lab.

At this point, your reaction will heat up, and the pressure will increase further (coefficient of expansion!). When the reaction is over, yurn the current dial to zero and turn the heat knob off. Karma will usually take overnight to cool.

TAKING YOUR SAMPLE OUT

After Karma has cooled to r.t. do the following:

1. Turn power knob to OFF
2. Turn pressure knob to DEC
3. When P = 105 tons, make sure to "spin down" the silver saucer (Figure 7), by increasing the pressure with the Enerpac:
    a. make sure Enerpac handle is in the increase (right) position (Figure 7)
    b. open pump valve just ~ 1/4 of a turn
    c. increase pressure to ~ 160 tons
    d. turn up pressure RPM to 2000
    e. repeat until silver saucer is as low as possible
4. Once the silver saucer is as low as possible, you'll need to decrease the pressure to zero. You do this gradually (8-10 tons at a time) turning the Enerpac handle to the left about 10 degrees, and then replacing in to the right. This opens and closes the Enerpac valve just a little bit at a time to slowly release the pressure.
5. When P ~ 1 tons, you lower the sample chamber by opening the pump valve several turns, turning the Enerpac handle to the down position, and pushing the Enerpac button until the chamber is all the way lowered and the wheels are touch the base.
6. Turn the pressure inc/dec knob to the neutral position (Figure 6)
7. Push the pressure on/off button to off
8. At the computer terminal push the "stop sign" to stop data collection. Now you can just take your sample out by disassembling the anvils (you can leave the lower layer in for several runs until you have to take them out and clean them and replace the Mylar). Check for holes in the Mylar if you're not going to replace it. Vacuum out the other peices in the sample chamber. You can use a hammer to crack open your cube, and pry out your sample with pliers. Congratulations! You've just squeezed the heck out of something.

Figure 7: Pumps for Karma

□ = pump valve ○ = Enerpac handle △ = silver saucer

MYLAR SHEETS

There are 2 kinds of Mylar Sheets that you'll need for Karma. One is just a long rectangle that fits around the inside rim of the sample chamber. The other is a disc that serves as a cap. You'll need two of each. For the rectangles, place one flush around the inside rim, making sure that it is just short enough so that the edges do not overlap. Do the same with the second sheet, except that the place where the edges meet should be at the opposite side of the sample chamber. For the discs, the first one goes on the bottom of the chamber (underneath all the anvils) and must have 3 holes cut in it, two small ones for the copper leads sticking up, and a big one in the middle for the connecting metal sheet. The second one goes in between the top layer of anvils and the big metal disc (Figure 5). This time there is only one hole for the connecting metal sheet.

Figure 8: Mylar Sheets

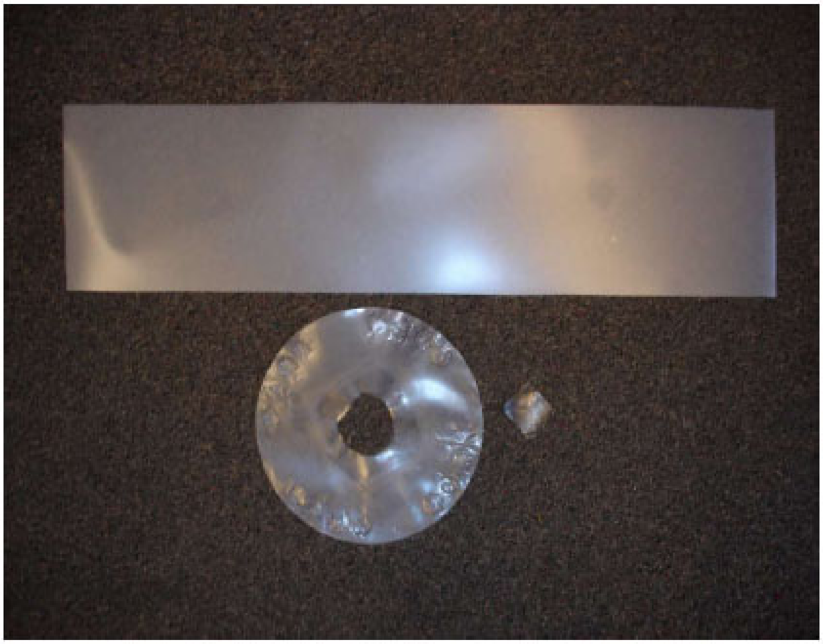


Figure 9: Wire with insulating sleeve (mullite)

APPENDIX

BORON NITRIDE SAMPLE HOLDERS

If your sample reacts with Gold, or if you need to heat it in the temperature range of 1100 - 1300 C, then you'll want to use a boron nitride capsule. The BN capsules are made from BN rods that you can order from a chemical supplier. The rods are the same diameter, and length as the Alumina sleeves, but have thicker walls and also a bottom. NEVER use the last one, but take it down to Ken in the machine shop as a model for new ones. The BN caps just kind of nestle on top.

PREPARING THE THERMOCOUPLE WIRE

If you run out of thermocouple wire, you'll need to cut more. They come in big spools, and the two types were mentioned in the text above. Since you have to differentiate the wires, it helps to cut them to different lengths. The wires should be cut into 2-3 cm lengths.

PATCHING THE MYLAR

As you use the Mylar in your reactions, you'll notice some holes. You can patch these by simply placing a piece of Mylar over the hole and attaching it with some tape. This way you don't have to replace all the Mylar if you've only used it once or twice.