Instruction manual for the COMPRES 10/5 assembly

Compiled by K. Leinenweber

A. Basic information

The COMPRES 10/5 assembly (Figures 1, 2) was first designed by Tamara Diedrich. The Fei-style 8/3 assembly was used as a model, and like that assembly the COMPRES 10/5 is based on a rhenium furnace with a lanthanum chromite thermal insulating sleeve, but there are important differences. Primarily, the bottom plug of hard alumina is replaced by layers of softer zirconia and magnesia, and a soft sleeve of magnesia is placed around the hard alumina thermocouple tube.

The parts for the assembly are available through the COMPRES multi-anvil project, run by K. Leinenweber, J. Tyburczy and T. Sharp at Arizona State University. The detailed specifications (materials and dimensions) of the parts are available upon request.

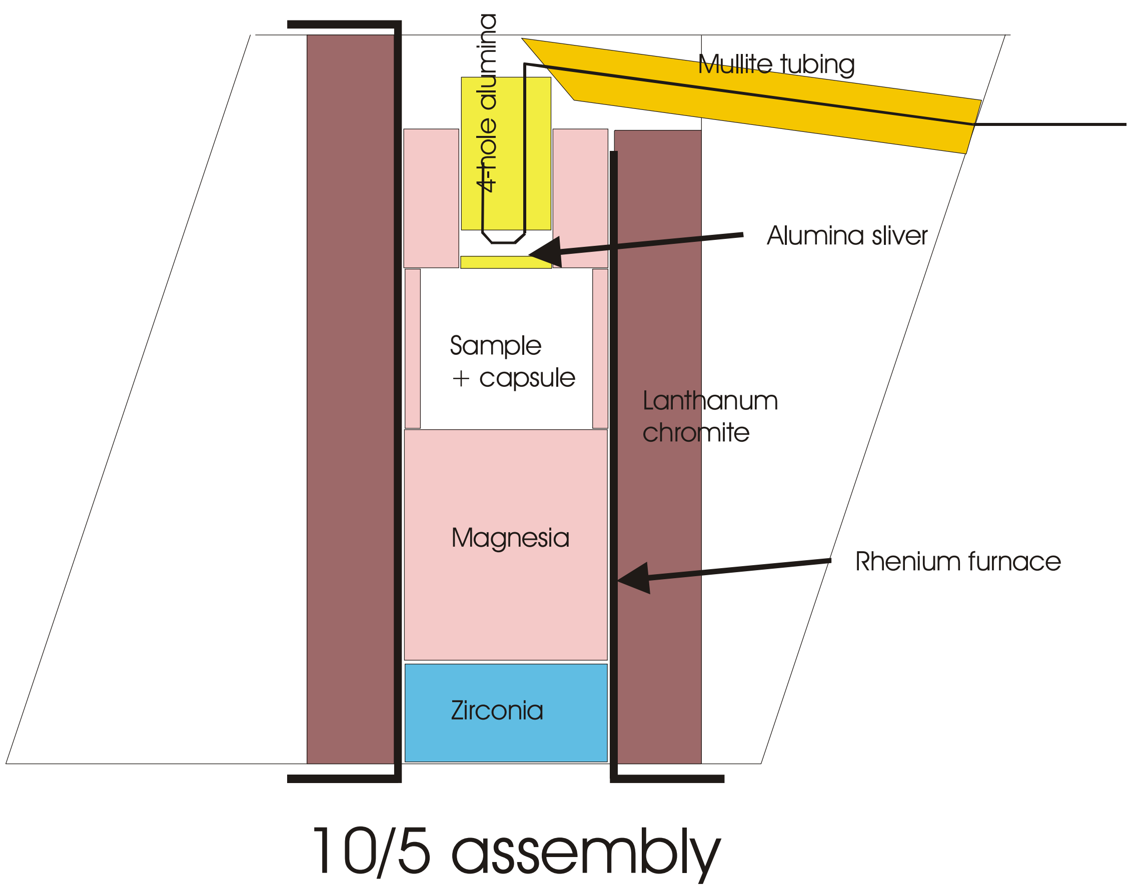


Figure 1. Schematic cross-section of the 10/5 assembly.

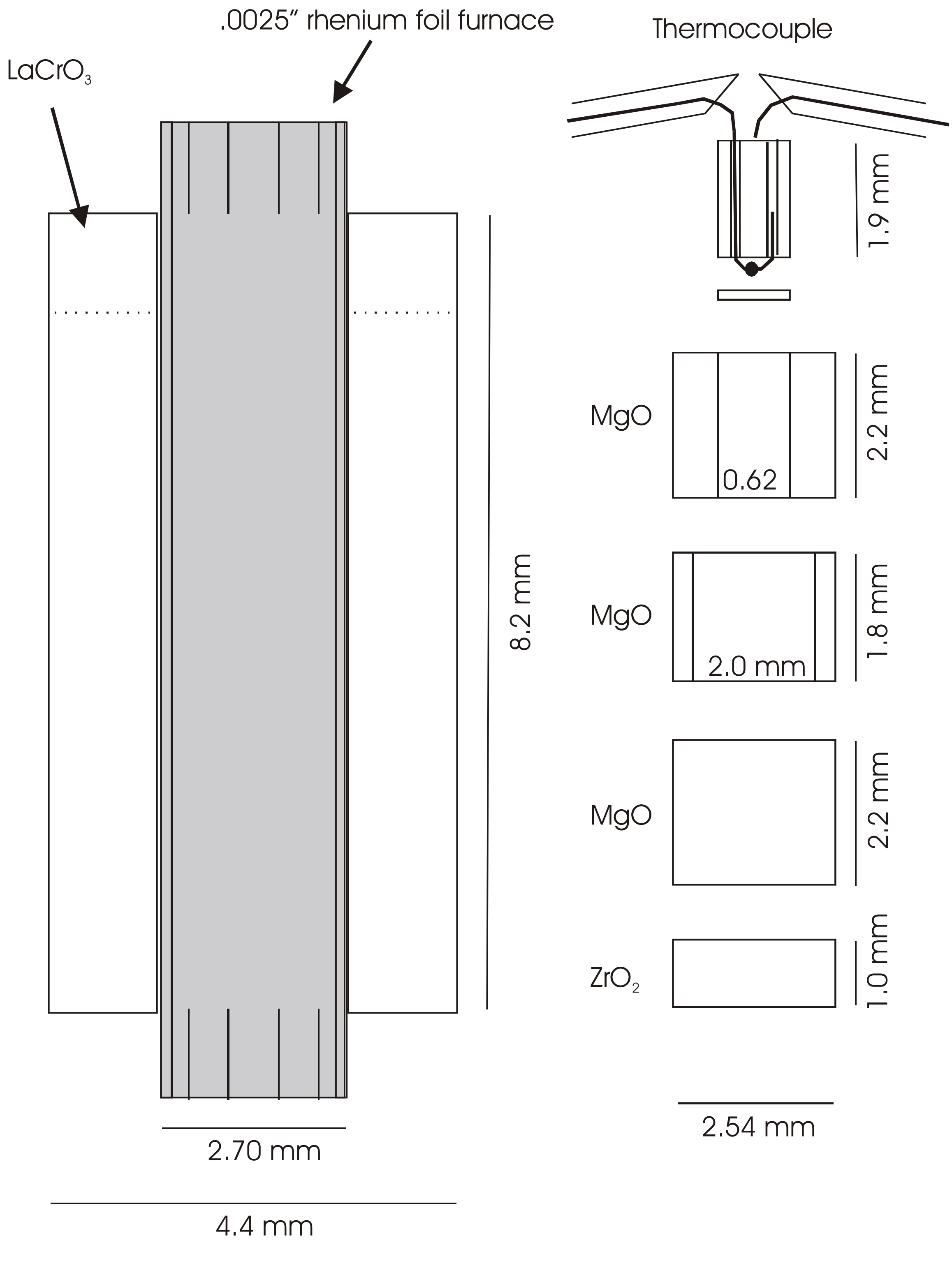


Figure 2: Dimensions of the inner parts of the 10/5 assembly.

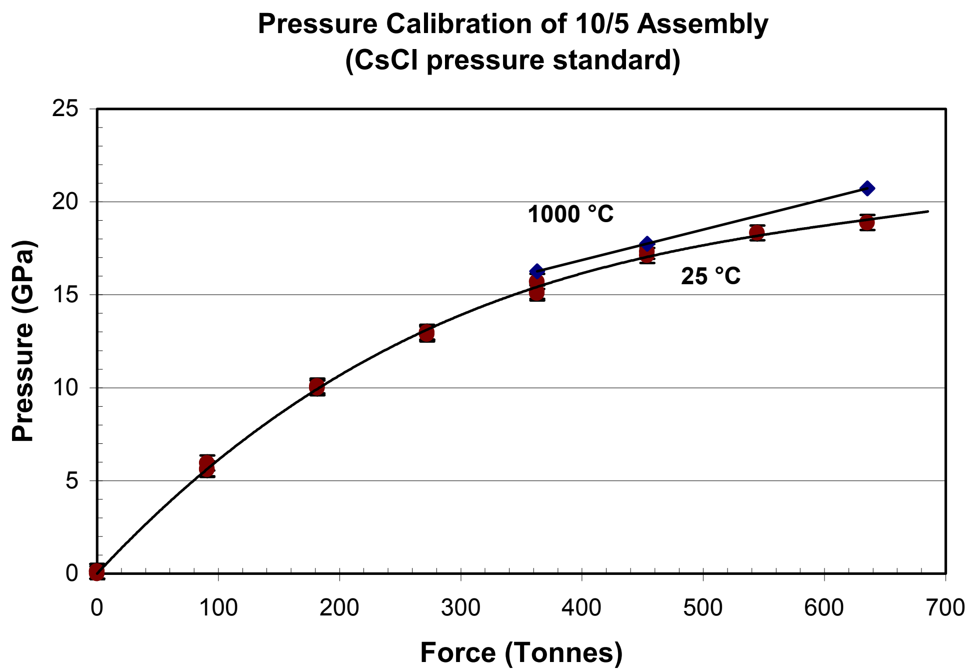


*Figure 3. Room-temperature and 1000 °C calibration lines from the 10/5 assembly (Tamara Diedrich, Thomas G. Sharp. Emmanuel Soignard, Wyatt DuFrane, Kurt Leinenweber). (*note: change to metric tons).*

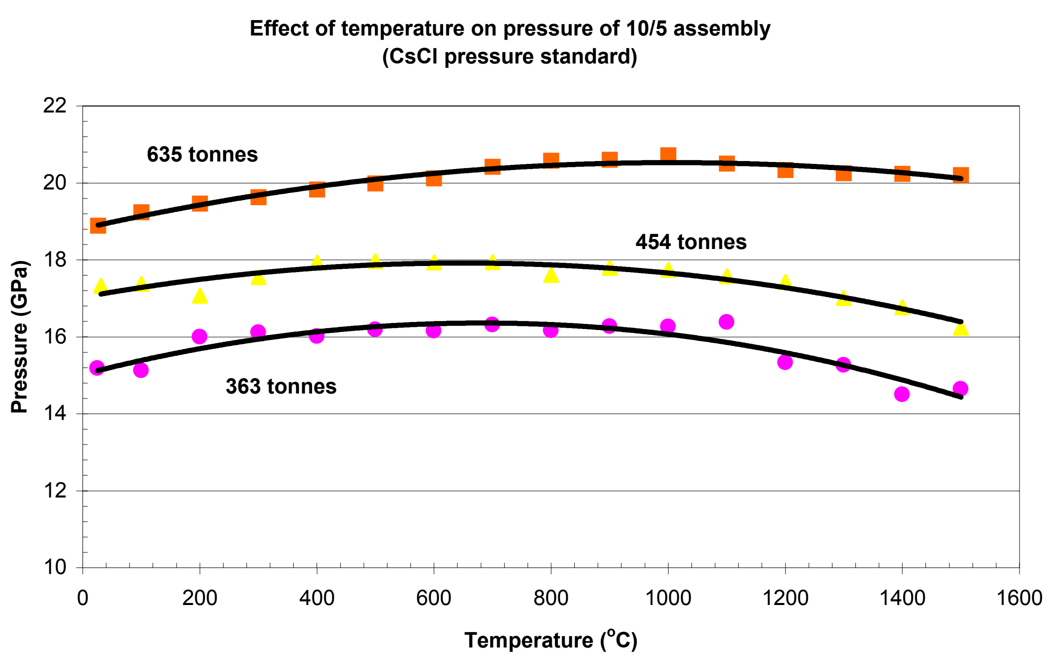


Fig. 4. Effect of temperature on pressure, at constant load. (Tamara Diedrich, Thomas G. Sharp. Emmanuel Soignard, Wyatt DuFrane, Kurt Leinenweber).

Based on the data in Figure 4, the equation for pressure as a function of load and temperature, valid from 400 to 700 metric tons and 25 to 1500 °C without extrapolation, is:

$$P = (1.3599*10^{-11}T^2 - 1.1982*10^{-8}T - 3.5714*10^{-5})F^2 + (-1.1804*10^{-8}T^2 + 1.2993*10^{-5}T + 5.1884*10^{-2})F. \quad (1)$$

where P is the pressure in GPa, T is the temperature in degrees Celsius, and F is the force in metric tons.

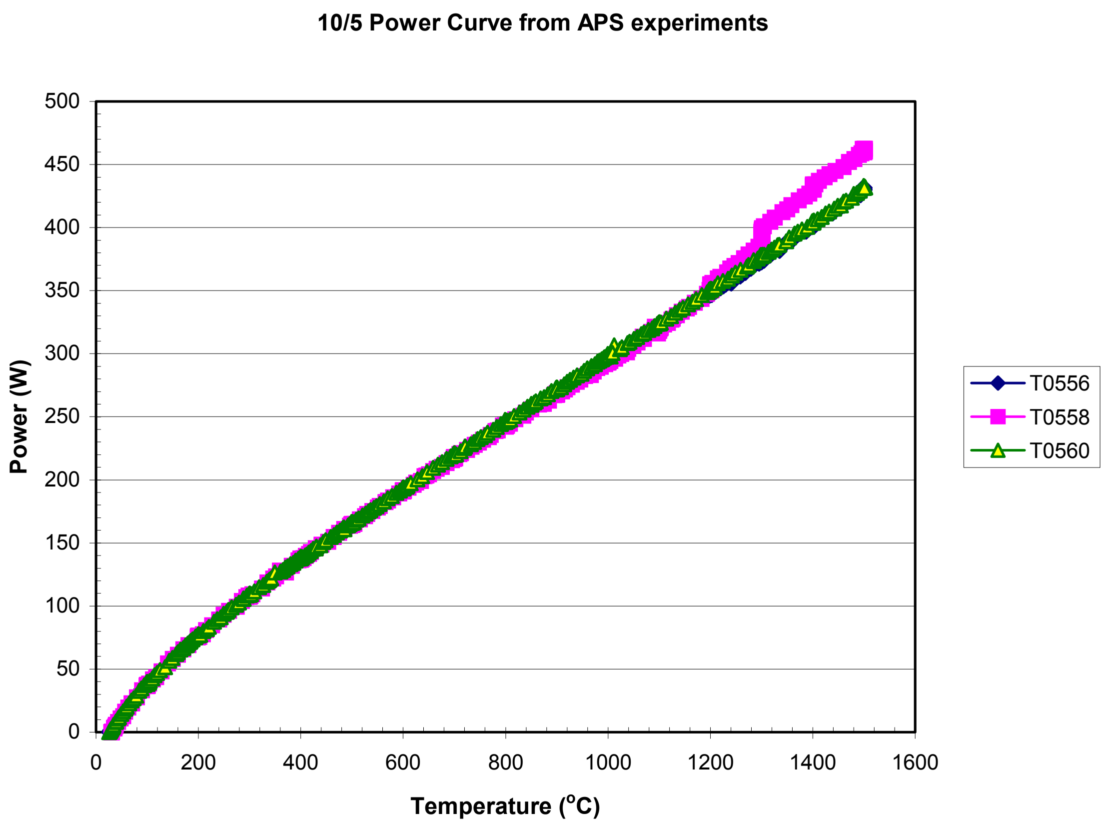


Fig. 5. Some power curves from the 10/5 assembly for examples. We have no idea what happened in run T0559.

ASSEMBLY INSTRUCTIONS

A. Setting up the furnace

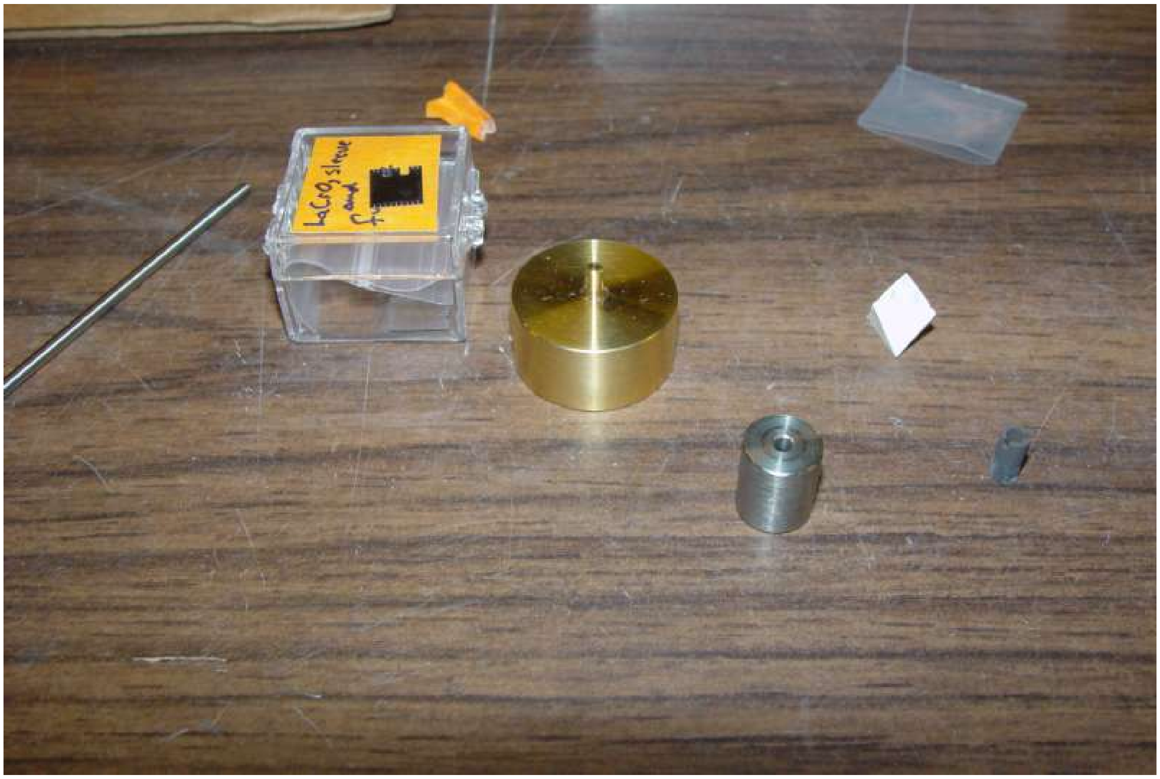


Figure 1: some items of the kit for making the furnace. From left: pin, rhenium foil furnace (on top of plastic box), brass dummy, stainless steel bushing, octahedron, LaCrO3 sleeve.

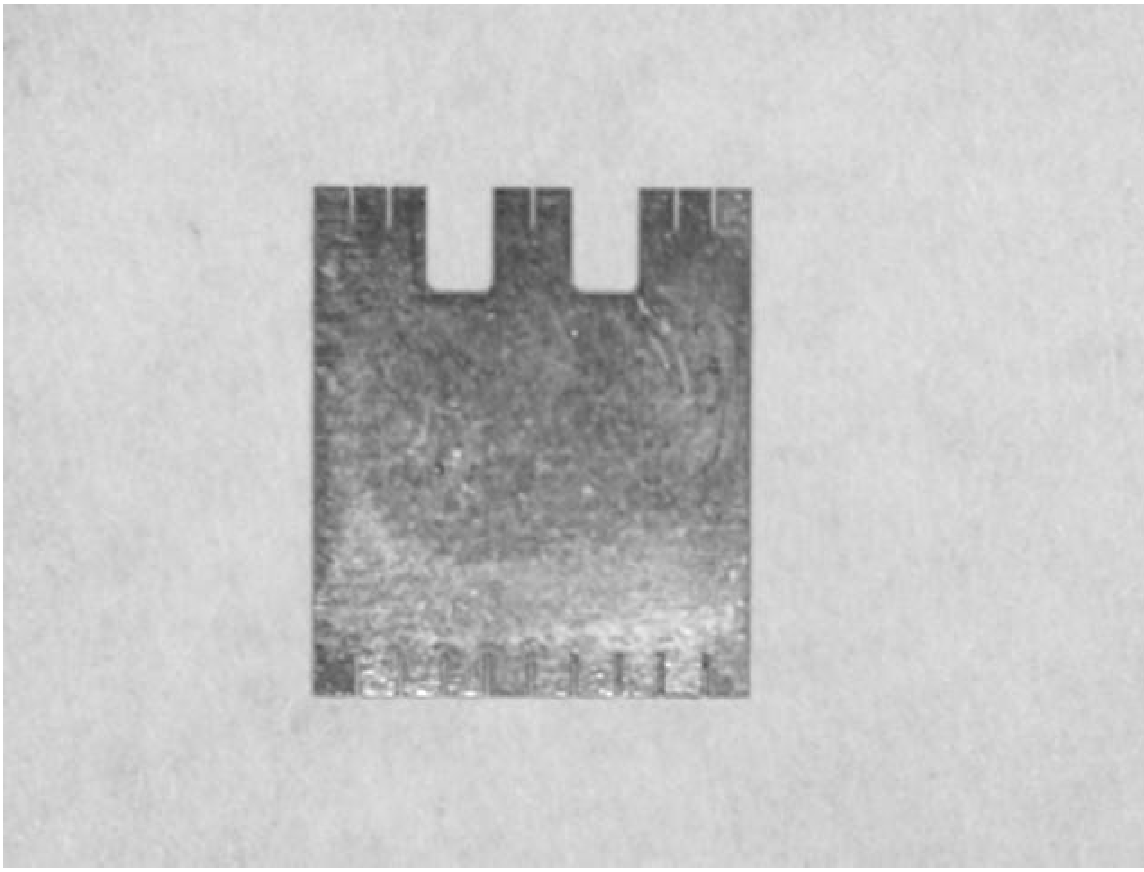


Figure 2. Photograph of the rhenium foil furnace for the 10/5 assembly (original wide thermocouple slot version).

1. Gather together the six items shown in Figure 1.
2. Using your fingers, wrap the rhenium furnace (which is supplied as a flat piece, Figure 2) around a #40 drill blank, to create a cylinder. Wrap the furnace lengthwise so that the seam is parallel to the drill blank axis, and the tabs are on either end of the cylindrical furnace (Figures 3 and 4). Use your fingers and if necessary a flat plier to work the furnace around the drill blank until it's fairly tight around the drill blank.

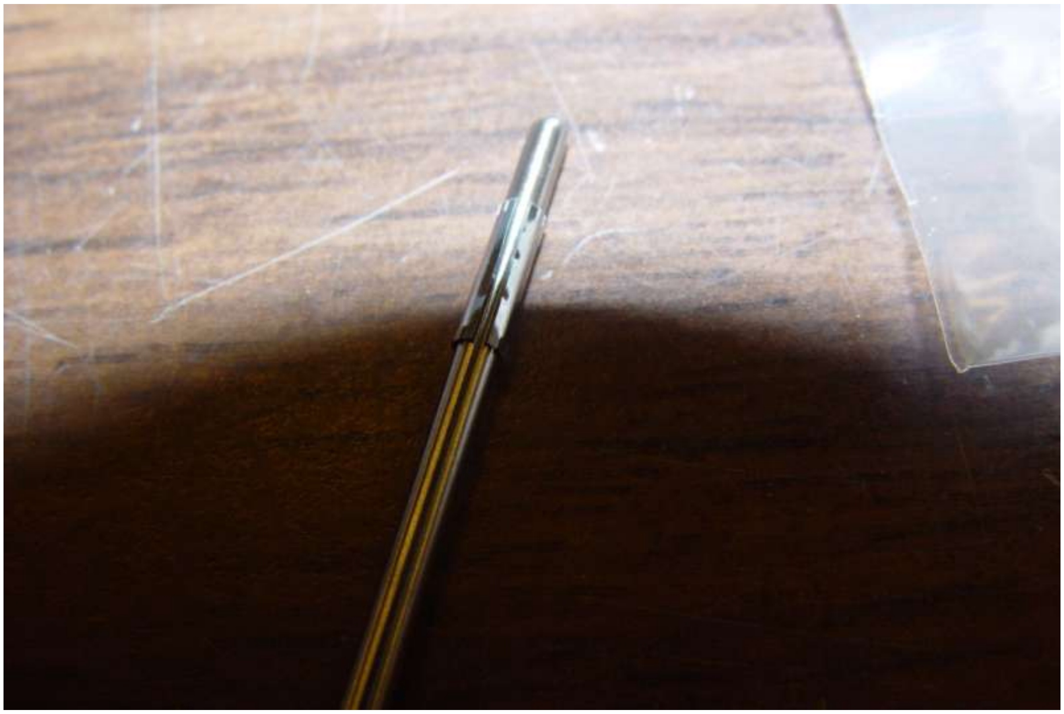


Figure 3. Re furnace wrapped around a drill blank.

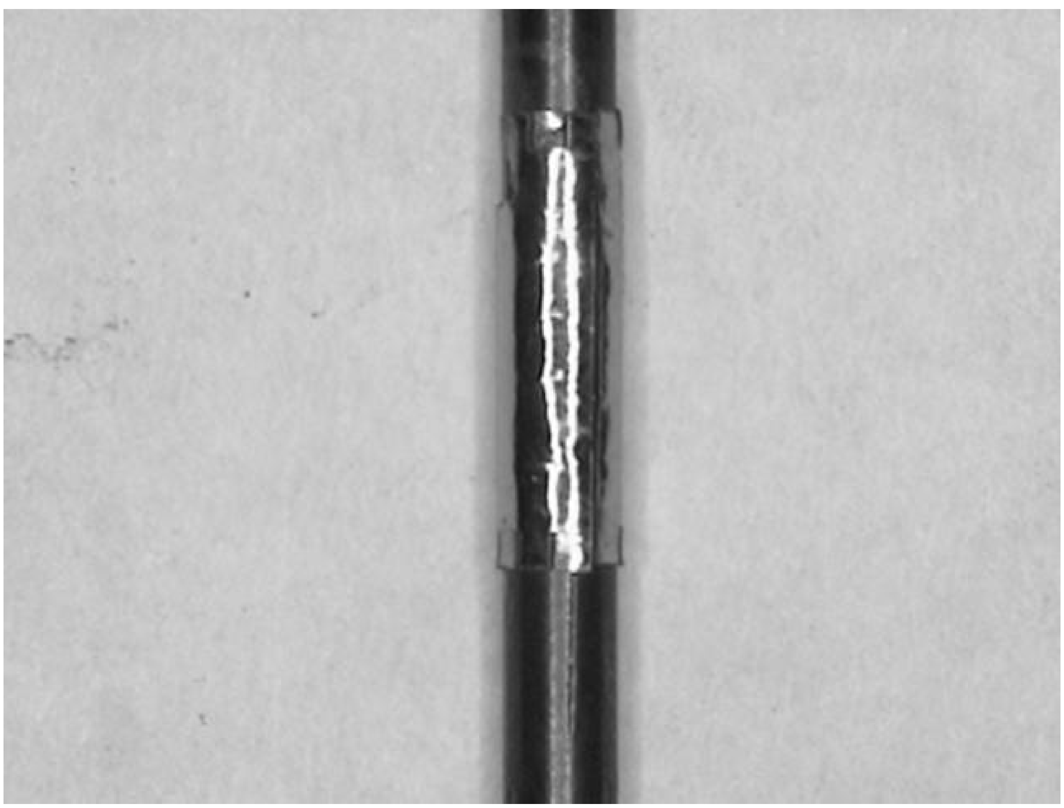


Figure 4. Close-up of Re furnace wrapped around the drill blank, with the seam facing the camera. The top and bottom of the furnace should be evenly matched on both sides of the seam (i.e. not offset) after wrapping.

3. Push the furnace so that the tabs on the end without grooves are protruding over the end of the drill blank (this side of the furnace is easier to insert into the bushing).
4. It is easiest to pre-shape the furnace using a metal bushing before putting it into the brittle LaCrO3 sleeve. Slide the furnace into a metal bushing with a #36 hole in it (Figs. 1, 5). If necessary, squeeze the furnace lightly with your fingers, or gently push the tabs towards each other, bending them inward just enough so one end of the furnace can just be slid into the stainless steel bushing (be careful not to kink the tabs). Use a flat surface such as a glass slide or the side of the workbench to finish pushing the rhenium into the bushing evenly.
5. Work the furnace carefully using the drill blank and bushing so that the drill blank fits inside the furnace while the furnace is entirely within the bushing.

Figure 5: #40 drill blank (left) and #36 stainless steel bushing (right).


Figure 6: Rhenium furnace inside the LaCrO3 sleeve.

6. Remove the furnace from the stainless steel bushing using a #36 drill blank (this drill blank fills the hole in the bushing with no extra space left over, and is used to push the rhenium cylinder out without bending the rhenium).
7. Slide the rhenium furnace carefully into the LaCrO3 sleeve, pushing the side with the thermocouple notches in first, until the notches coincide with the thermocouple grooves in the LaCrO3 sleeve. If you encounter resistance, remove the furnace from the sleeve and close the furnace diameter slightly so the furnace will slide in and try again. Adjust the furnace so that the rhenium protrudes equally from both sides of the sleeve and the thermocouple notches are aligned (Figures 6 and 7).

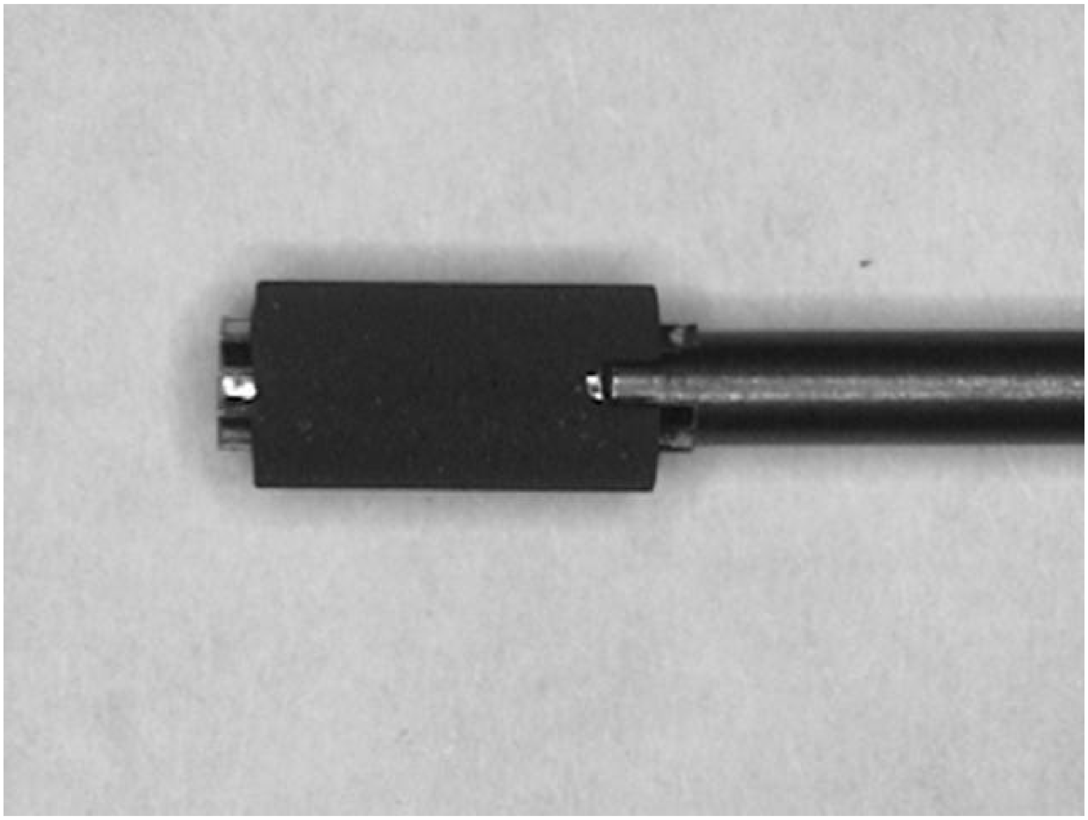


Figure 7 – Re furnace, pin, and LaCrO3 sleeve after centering the furnace and aligning the thermocouple grooves .

Figure 8: Rhenium furnace in LaCrO3 sleeve, after all the tabs have been folded outward, also showing the octahedron with thermocouple grooves in it.

8. Once the alignment of the furnace and sleeve are correct, fold all the tabs carefully back and outward so that they form a radial pattern of "petals" like a flower (Figure 8). Do this on both ends of the furnace Find an octahedron and put the LaCrO3 sleeve and furnace in it, with the thermocouple notches all aligned.

Figure 9. The brass dummy.

9. Find the brass dummy (Figures 1 and 9).
10. Push the LaCrO3 sleeve, furnace and octahedron onto the brass dummy with the thermocouple notches upward (Figure 10). Keep the LaCrO3 sleeve parallel to the dummy as you press it in so it doesn't break (one way to do this is to use the octahedron and a glass slide to align it as you push). There will be some resistance as you push the furnace onto the dummy, but if all the parts are correct the LaCrO3 sleeve should not break.

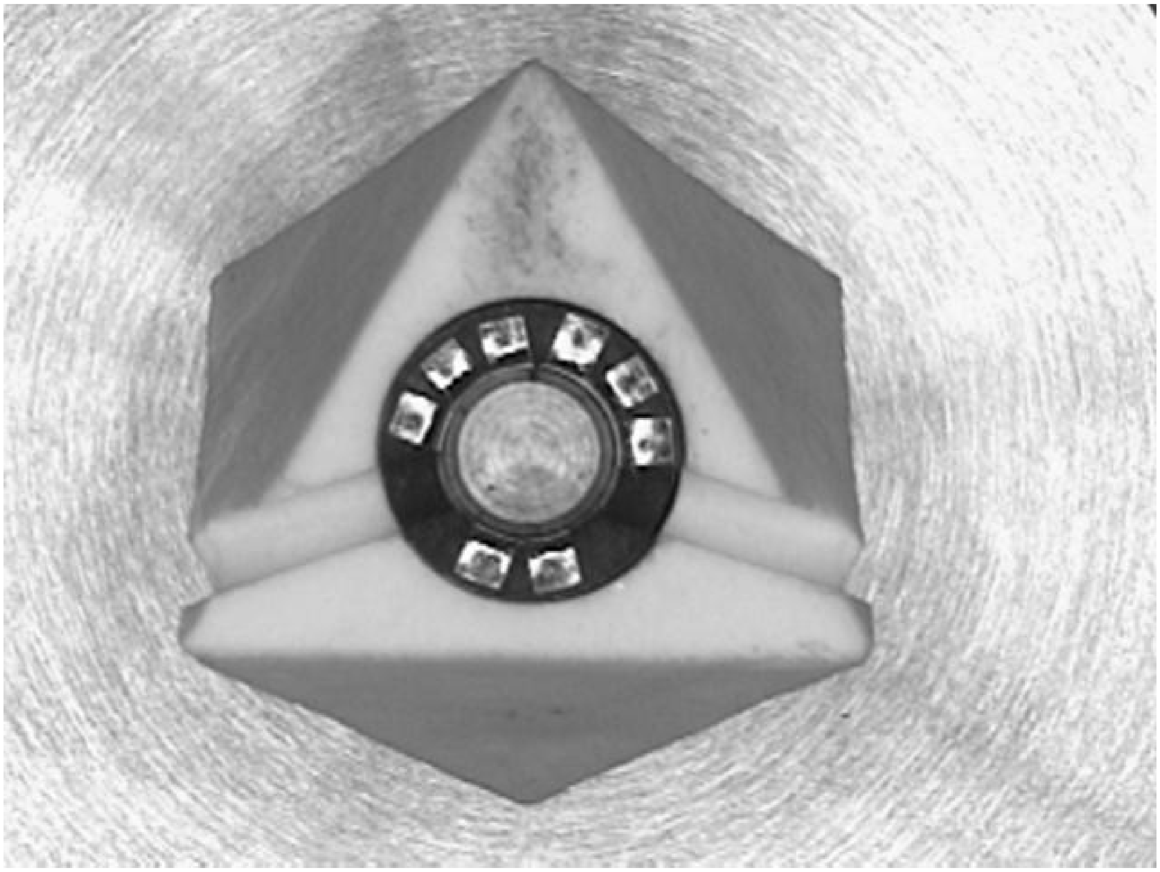


Figure 10: View of the octahedron/furnace resting on the brass dummy.

B. Thermocouple preparation.

1. Get two legs of a thermocouple out. Keep track of the two wires by putting on the color-coded teflon insulation. A summary of some useful information for Type C thermocouples is shown in the Table below (type C with a wire size of .007" is standard for this assembly).

Composition	Polarity	Color of insulation	Shininess	Hardness
W / 5% Re	+	white	dull	harder
W / 26% Re	-	red	shiny	softer

Figure 1. Two legs of a thermocouple with teflon tubing (white and red) and two pieces of mullite tubing on each wire.

2. Add two pieces of one-hole mullite tubing per wire (Figure 1).
3. Thread the (+) wire, mullite end first, through the small piece of 4-hole alumina tubing (see Fig. 1 of section D), make a small hook at the end and hook it through the hole that is katy-corner to that one.
4. Thread the (–) wire through another empty hole in the 4-hole tubing and make a second hook. Cross the two wires by hooking the second wire over the first, using the fourth hole in the 4-hole tubing (Figures 2 and 3). (Note: having the

softer wire on top is intended to increase the likelihood that the two wires will be in electrical contact even before pressure is applied). For a picture of the thermocouple cross later on in the process, see Figure 2 of section D.

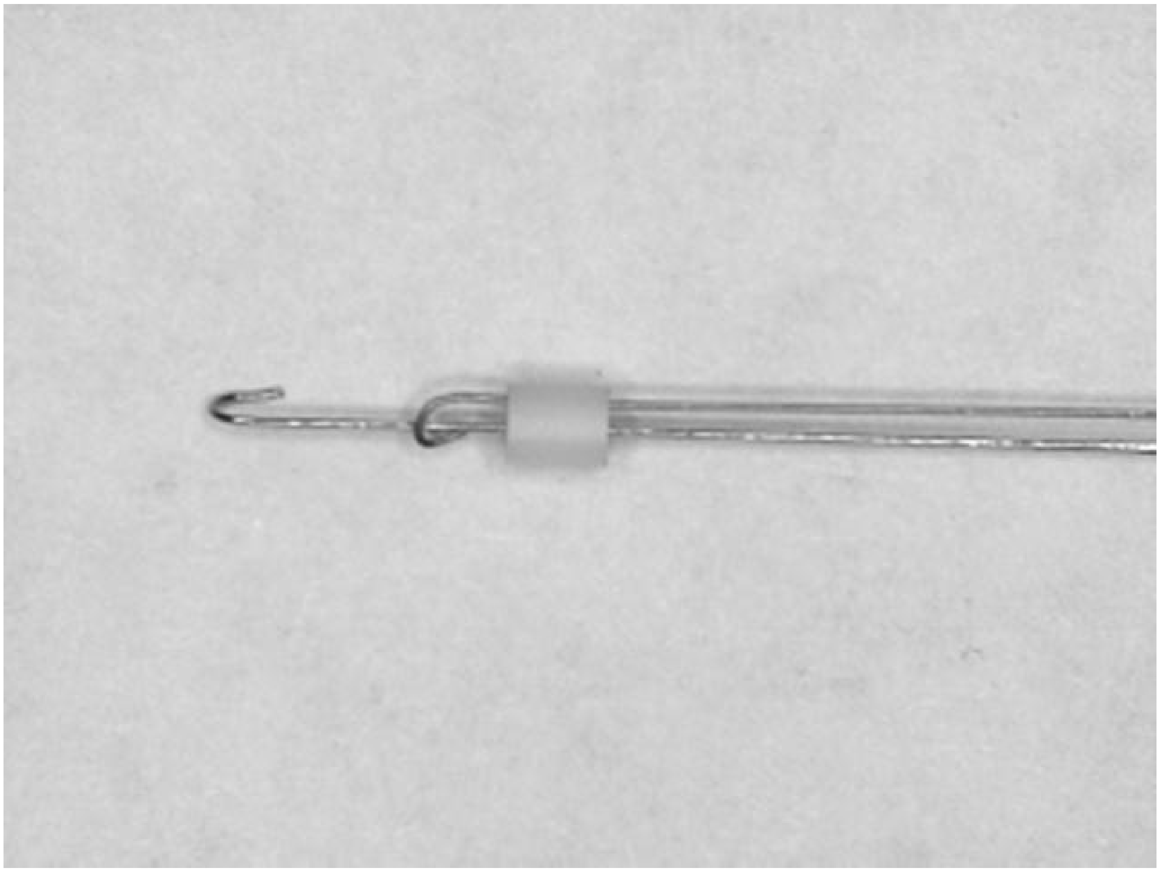


Figure 2: The two hooks in the thermocouple wires.

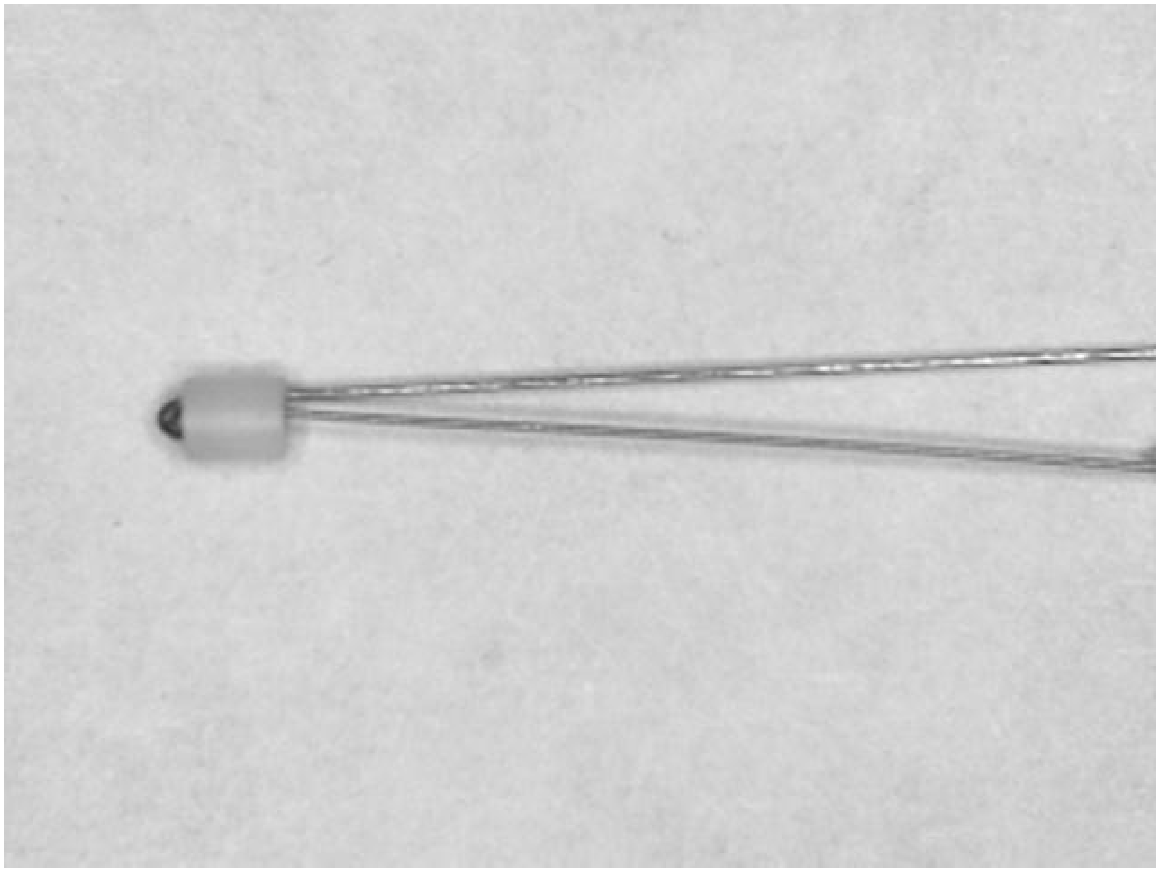


Figure 3: the thermocouple wires hooked and crossed through the 4-hole alumina.

5. Place the mullite all the way up against the 4-hole ceramic and pre-bend the two thermocouple wires, splayed apart and each one to an angle somewhat beyond 90 degrees, to match to slots in the octahedron (Figure 4). Be sure not to cross the wires where they are bent. Note: the wires can be bent in one direction easily but because they are very brittle they cannot be “unbent” much without breaking.

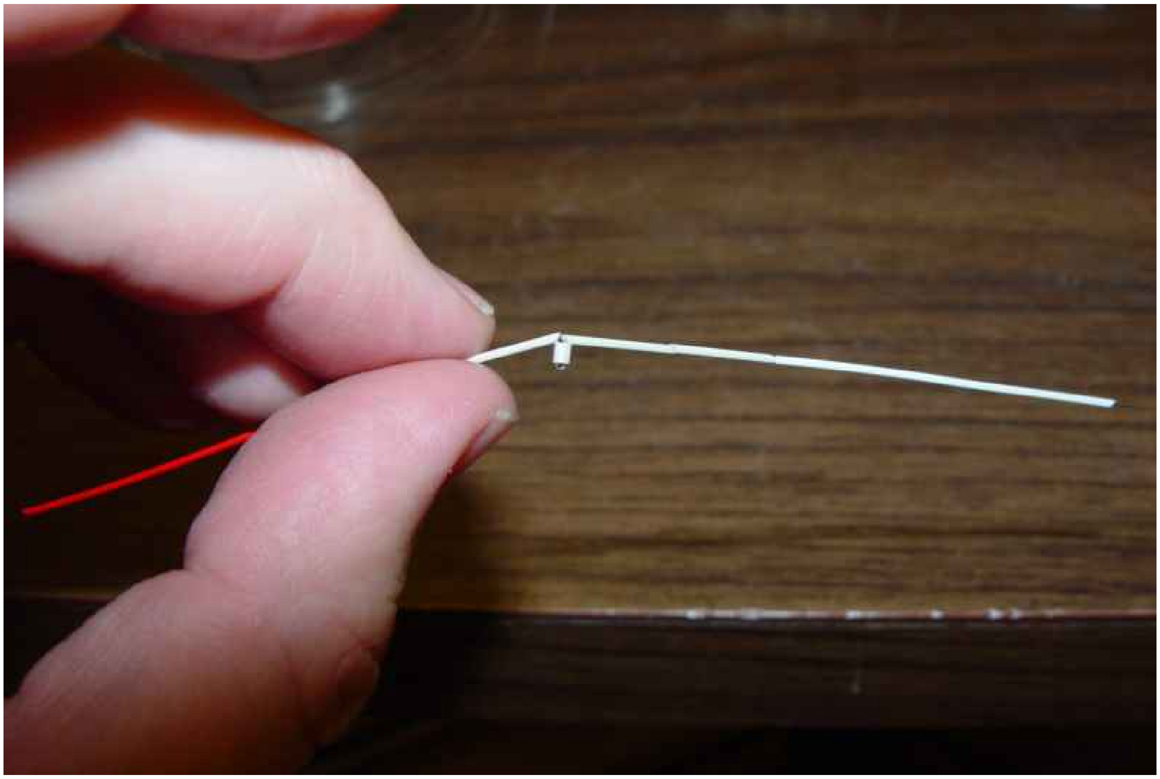
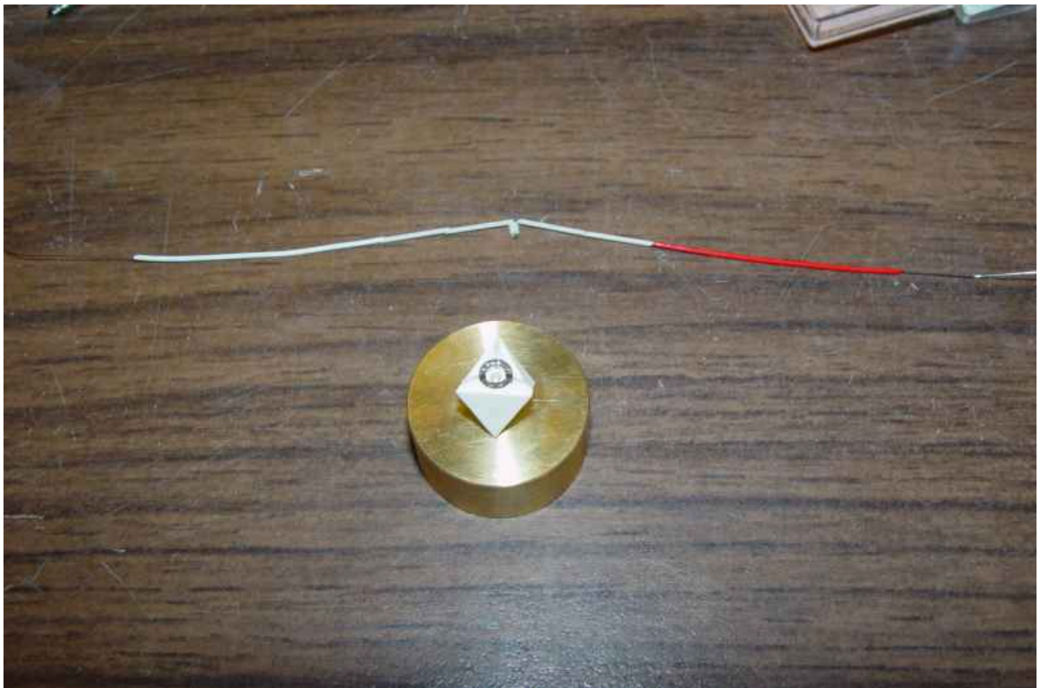


Figure 4: Thermocouple after bending, before placing into the octahedron.

C. Cementing the thermocouple into the octahedron.

1. Using a glass slide to keep it parallel, press a thick MgO sleeve (see Fig. 1 of section D) into the furnace from above. Then, push the sleeve all the way down to the top of the brass dummy using the #40 drill blank. The sleeve is visible in Figure 1A.



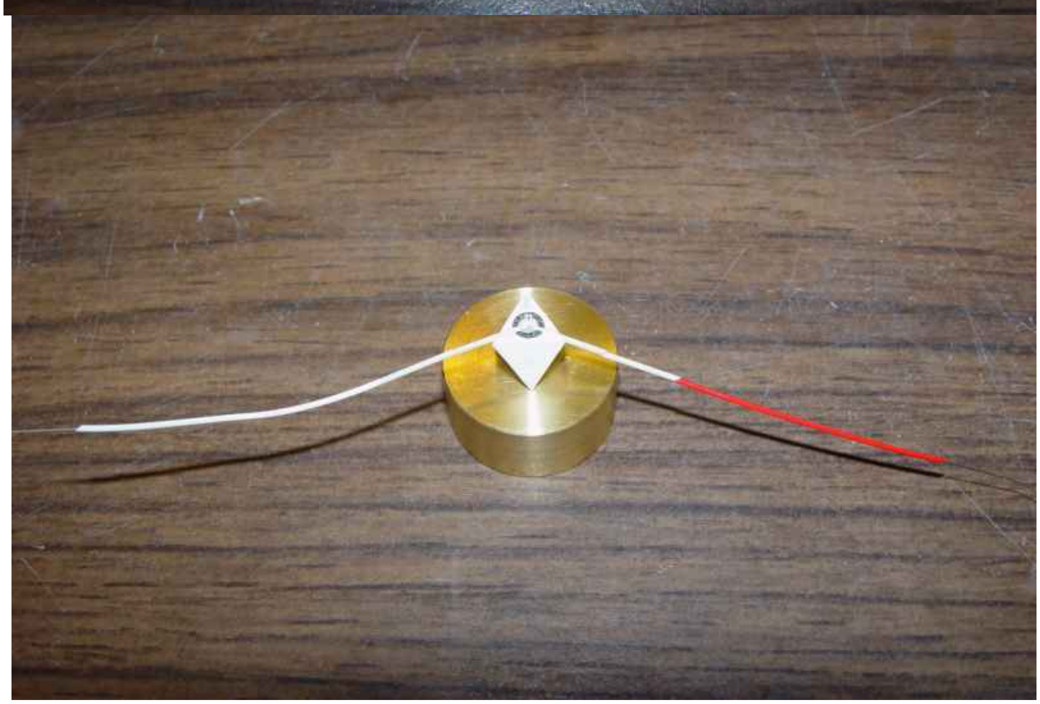


Figure 1.. (A). Thermocouple and octahedron. (B). Thermocouple placed into octahedron, before cementing.

2. Lower the thermocouple into the assembly. The 4-hole alumina goes into the MgO sleeve, and the two mullite pieces go into the grooves in the octahedron. Push on the 4-hole thermocouple cermic from above using a thin tool, to makke sure the thermocouple is all the way down to the brass dummy. This will help locate the thermocouple reproducibly, and also will help the thermocouple make electrical contact where the wires cross.
3. Make sure the two mullite pieces are moved forward far enough to electrically insulate the thermocouple from the Re furnace, and also provide enough coverage to insulate the thermocouple wires from the carbide anvil (cf. Figure 2).
4. Using Cotronics 940 cement and activator, cement the outer part of the thermocouple first (Figure 2) and let the cement set so the thermocouple is held down. The thermocouple should be all the way down in the groove and should exit the octahedron precisely on the octahedron edge, 2 mm from the corner; if the slot was cut properly and the thermocouple was held down all the way duri9ng the gluing, it should be in the right location. Correct location of the thermocouple wires will minimize thermocouple breakage. Note that the harder wire in particular may require special measures to hold it down during gluing (see Figure 3 for an example). Remove excess cement with a razor blade, restoring the original shape of the octahedron, as in Figure 2.

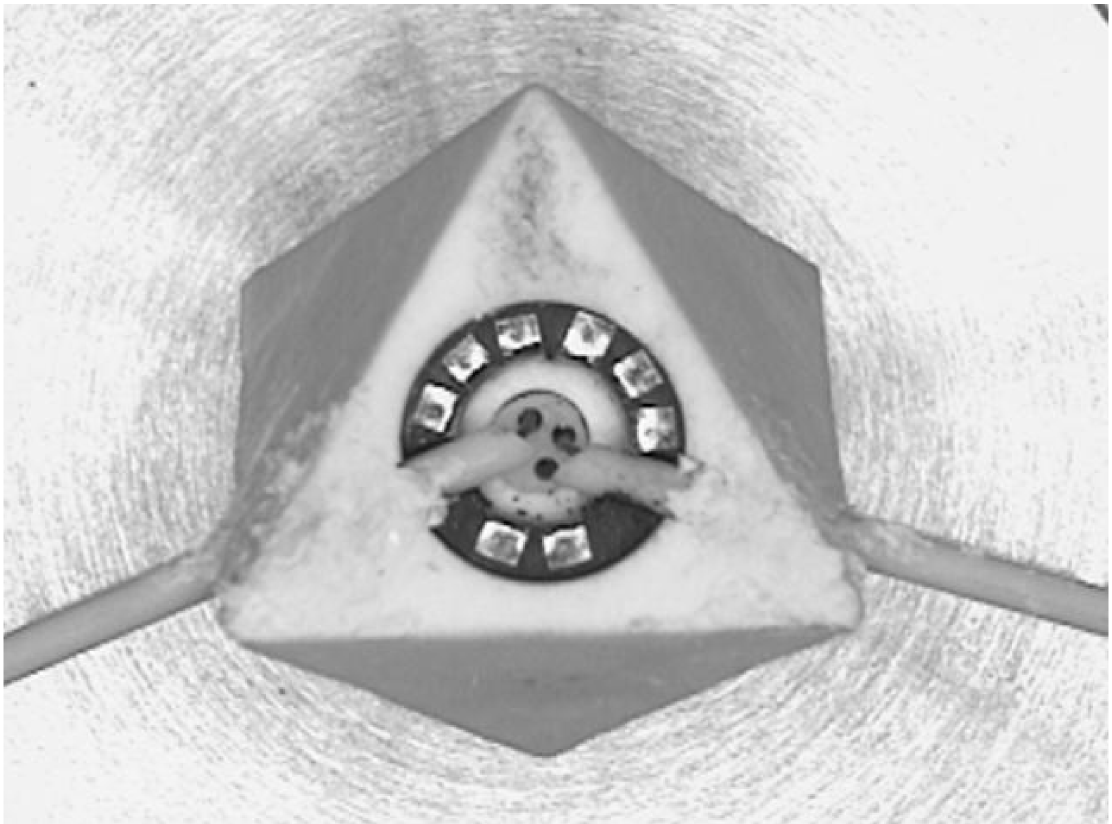


Figure 2: Cementing, part (1). Cement the outer part first.

Figure 3. Holding down a poorly-behaved (+) thermocouple wire for cementing under a binocular microscope.

5. Mix another batch of Cotronics 940 cement and fill in the rest of the thermocouple groove and the middle hole, working the cement into all the spaces with a toothpick (note the cement should not be runny). Clean the cement off the rhenium tabs with a razor blade and/ or needle, and remove excess cement to again restore the shape of the original octahedron (Figure 4). Make sure the elevation of the rhenium tabs is higher than the cement, so the cement does not flow over the rhenium tabs during compression and block the furnace current.

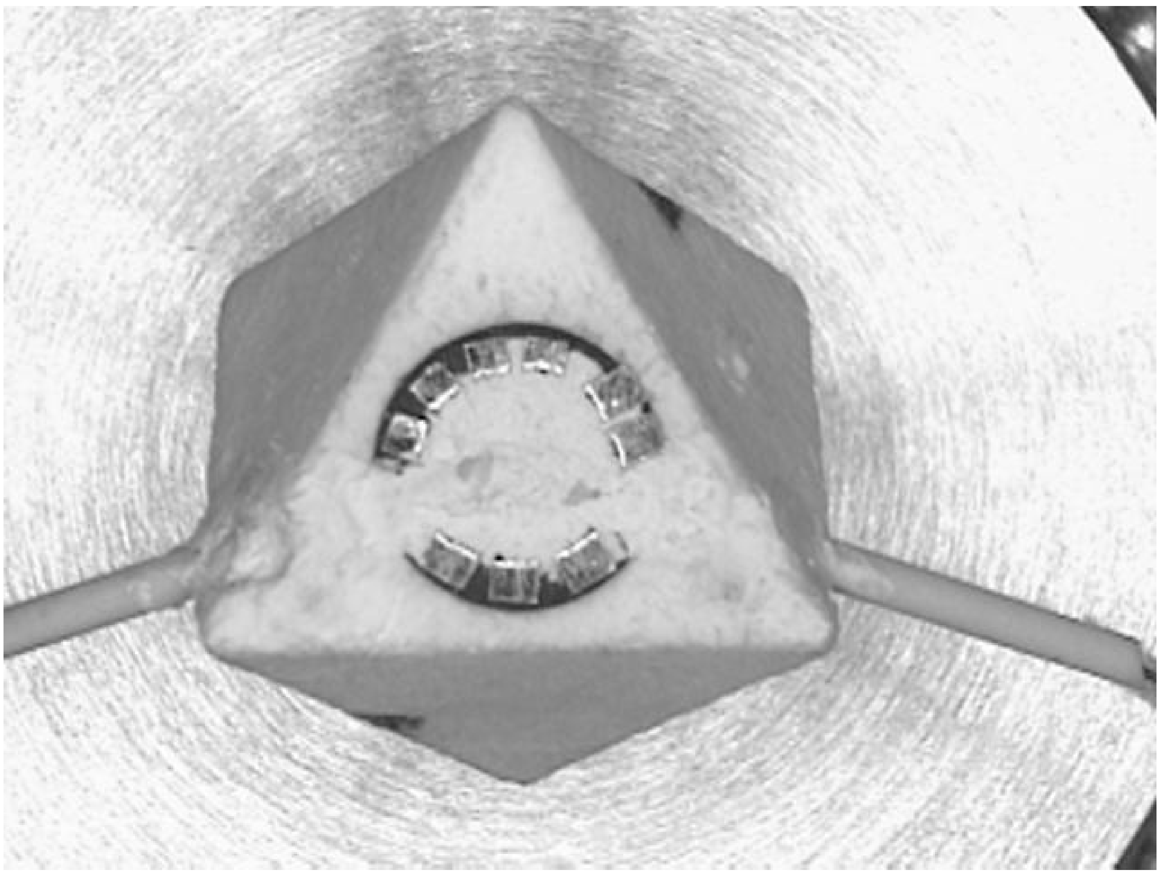


Figure 4: Cementing part (2). Cement the inner part and scrape the cement off the rhenium tabs.

6. Place the octahedron, including the dummy, into a drying oven (approximately 100 °C) for at least 15 minutes. If time permits, leave it in the oven overnight.

D. Loading the sample

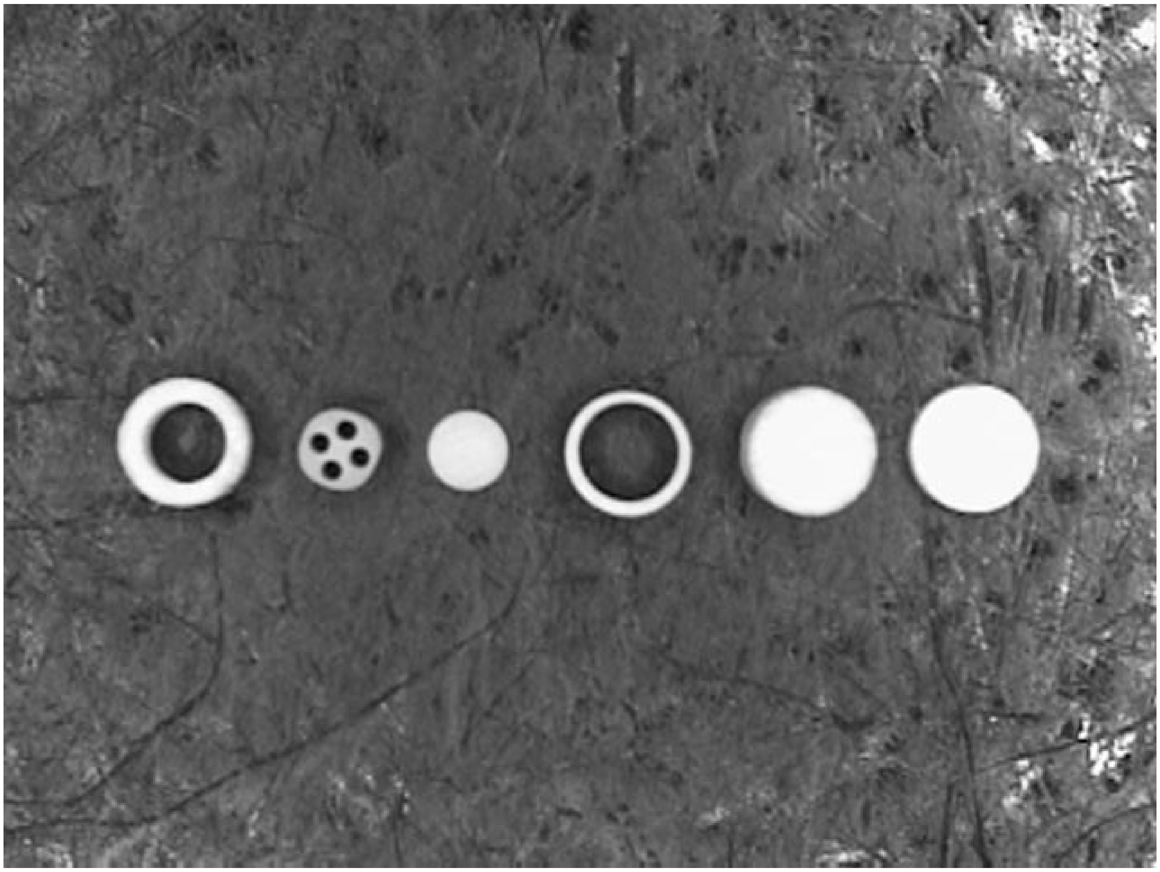


Figure 1: Inner pieces for 10/5 assembly. From left: thick MgO sleeve, 4-hole alumina thermocouple insulator, alumina sliver, thin MgO sleeve, MgO plug, zirconia cap.

1. Gather the four remaining pieces for the inside of the assembly: alumina sliver, thin MgO sleeve, MgO plug, zirconia sleeve (Items 3-6 in Figure 1).
2. Remove the octahedron from the oven and pull it off the brass dummy. The thermocouple is now cemented in, and should be visible by looking down the hole from the other side (Figure 2).

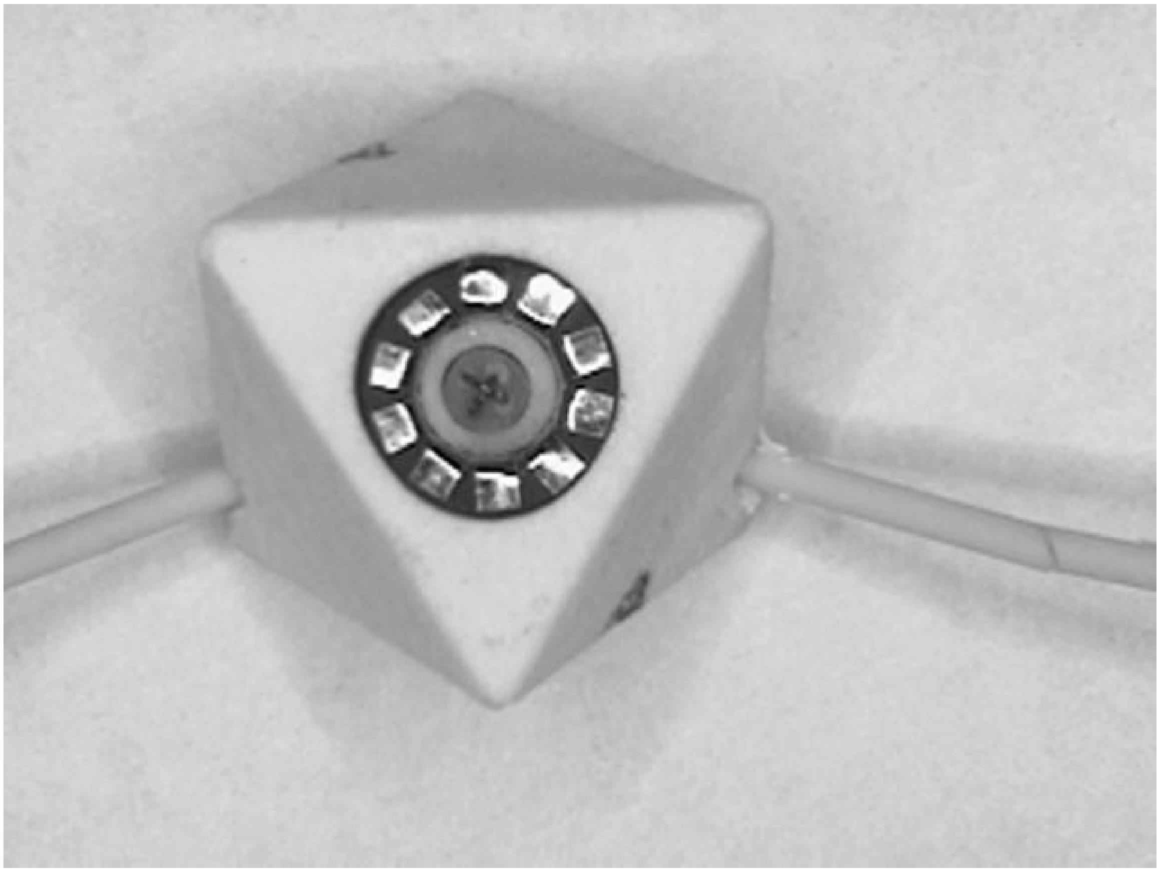


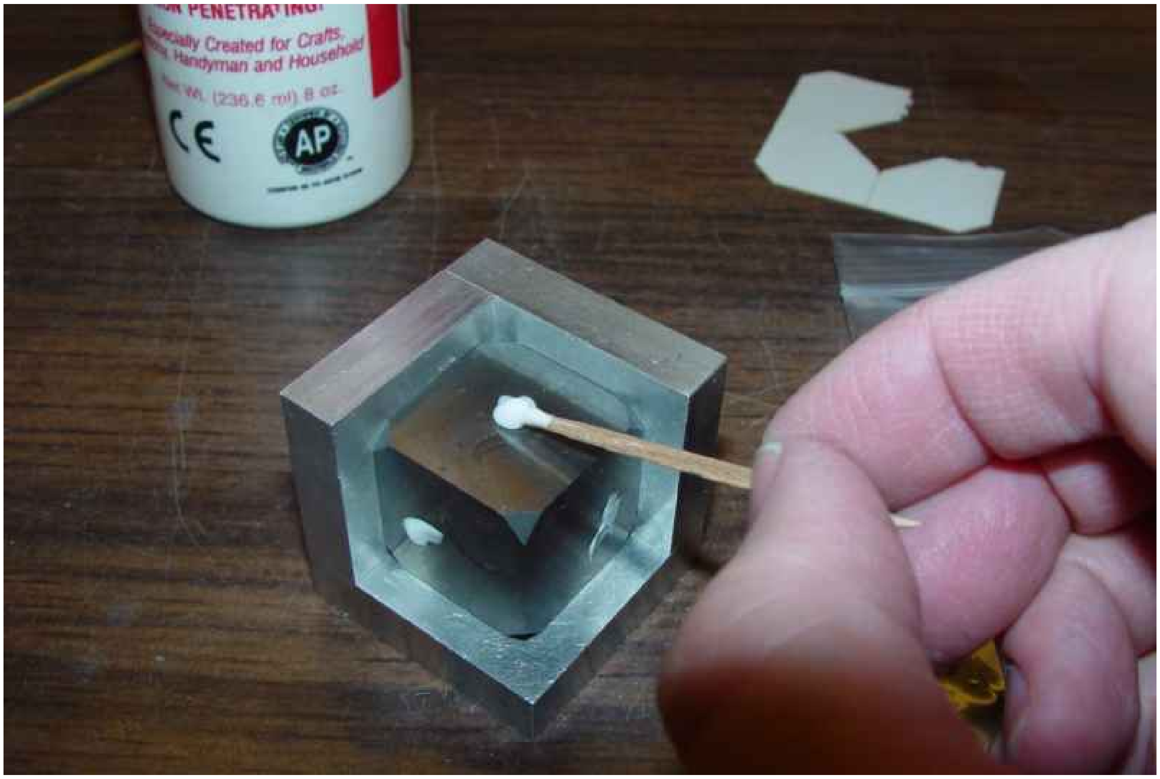
Figure 2: octahedron looking down the sample hole towards the thermocouple cross.

3. Viewing the octahedron in the direction of Figure 2, place the alumina sliver on top of the thermocouple cross to cover it.
4. Put the sample + capsule in on top of the thermocouple (Note: this part of the assembly is highly run-dependent and the capsule materials are not supplied. The primary information is that the outer diameter of the full sample + capsule is 0.100 inch (2.54 mm) and the length is 0.070" (1.78 mm). The thin sleeve can be used if your capsule is 2.0 mm in diameter. Alternatively, a second thick MgO sleeve can be used if the capsule is 1/16" (1.6 mm) in diameter. If the sample is inert and electrically insulating, the entire Re furnace can be filled and the sleeve can be dispensed with. Alternate capsules, such as BN, can also be used in place of MgO.)
5. Lower the MgO plug into the furnace on top of the sample.
6. Lower the ZrO2 cap into the furnace. At this point, the furnace should be "full" and the ZrO2 cap flush with the surface of the octahedron.
7. There shouldn't be any need to glue the ZrO2 cap on, it should stay put. If it does not, the best glue is probably a bit of 940, the same cement used for the thermocouple.

E. Assembling the cubes.

Gather together eight tungsten carbide cubes with 5 mm truncations (Toshiba grade F was used for the calibrations shown in this manual, part A).. Get four pieces of 10/5 paper. Glue the 10/5 paper to four of the cubes using any glue that works. A white glue is the cheapest and most benign.

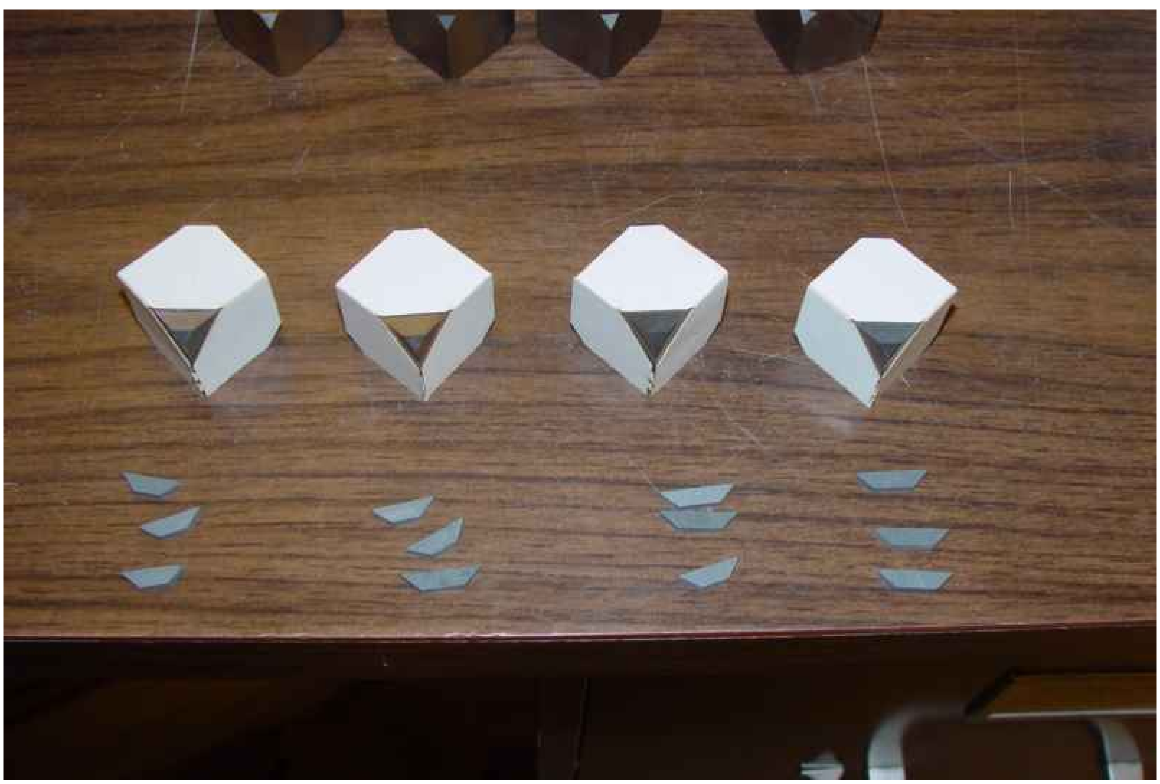
White glue, paper, and a carbide cube. The cube is held inside an optional “metal corner” in this photograph.



Put the glue on the cube.

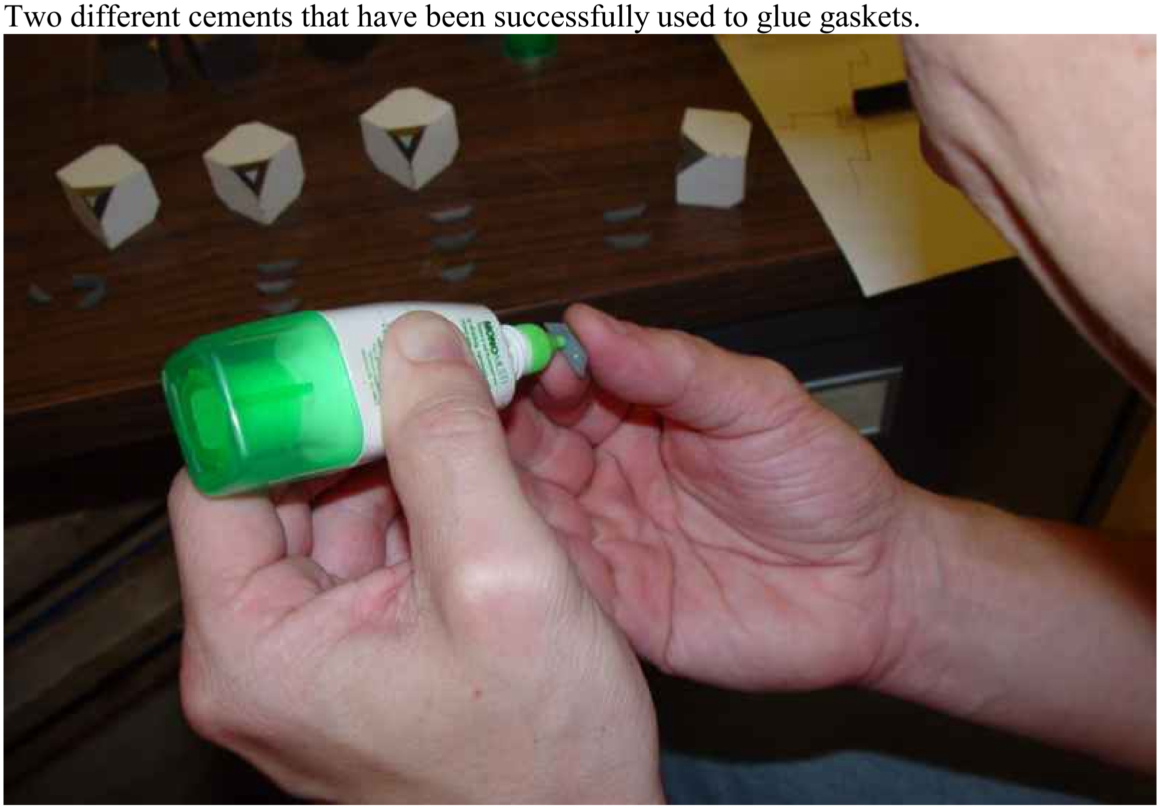
Fold the paper and stick it to the cube, with the back of the paper flush with the back of the cube.

Here, the glue is setting on 4 cubes while they are held by the other four (E. Soignard's trick).

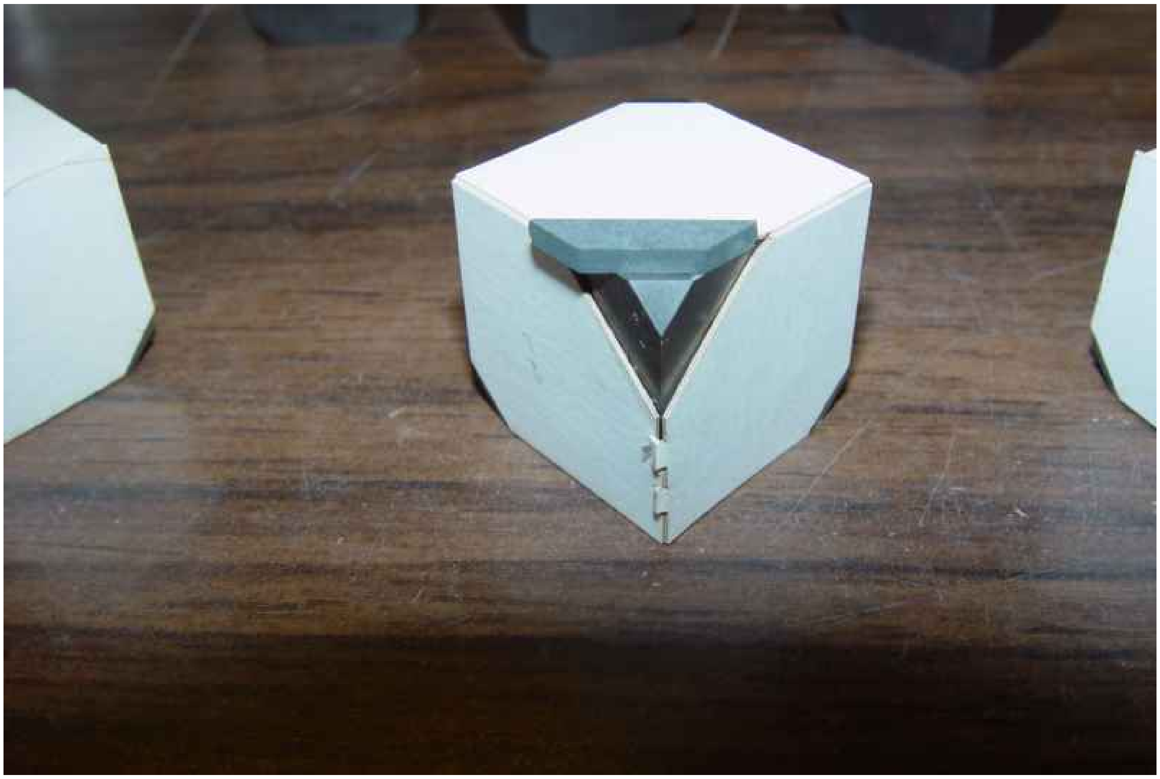


Find six long and six short pyrophyllite pieces. Line up the 4 cubes with the paper and organize the 12 gasket pieces according to the Figure above. Pyrophyllite pieces are divided as follows: Cube 1: short, short, short. Cube 2: long, short, short. Cube 3: long, long, short. Cube 4: long, long, long.

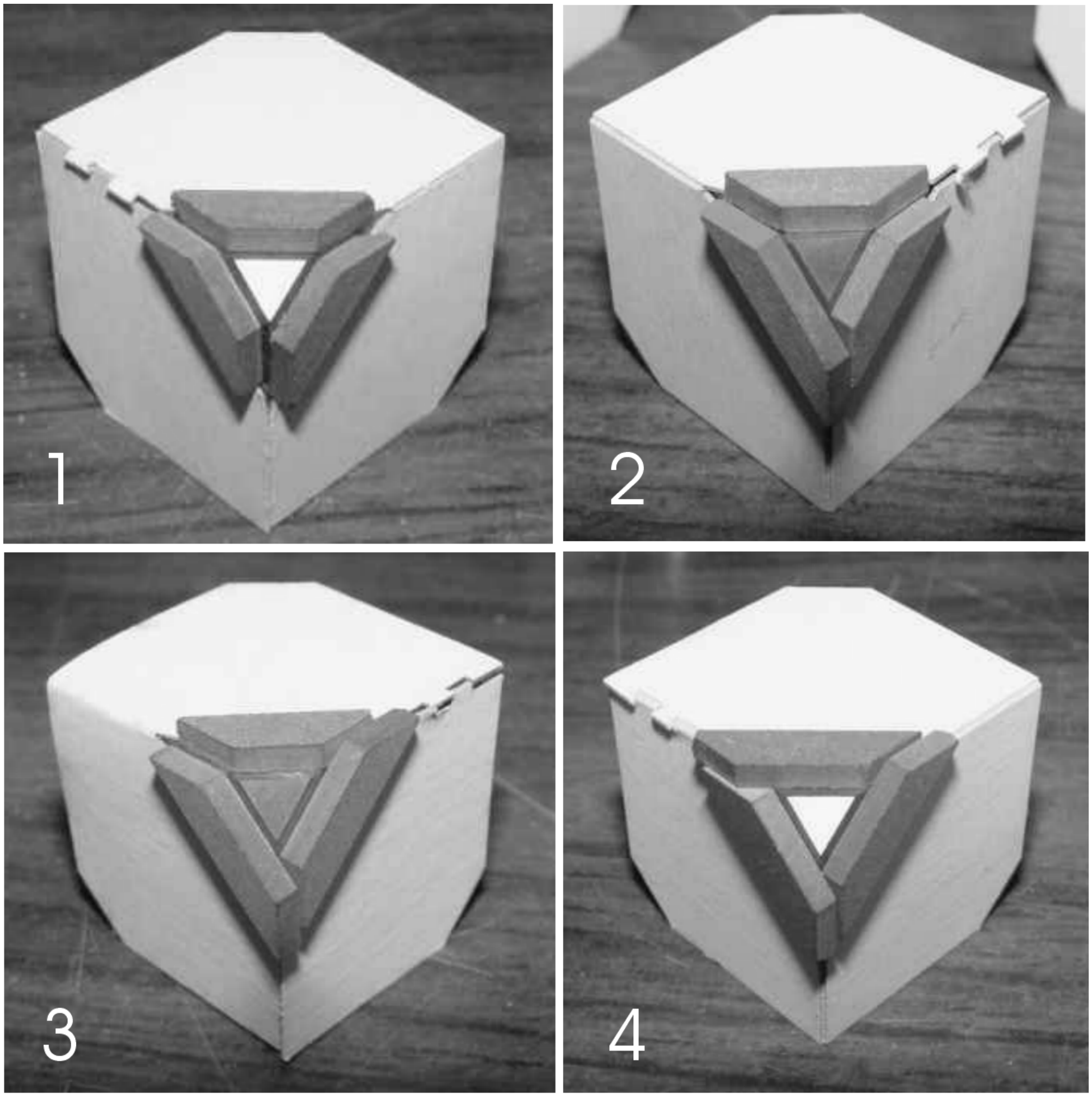
Two different cements that have been successfully used to glue gaskets.



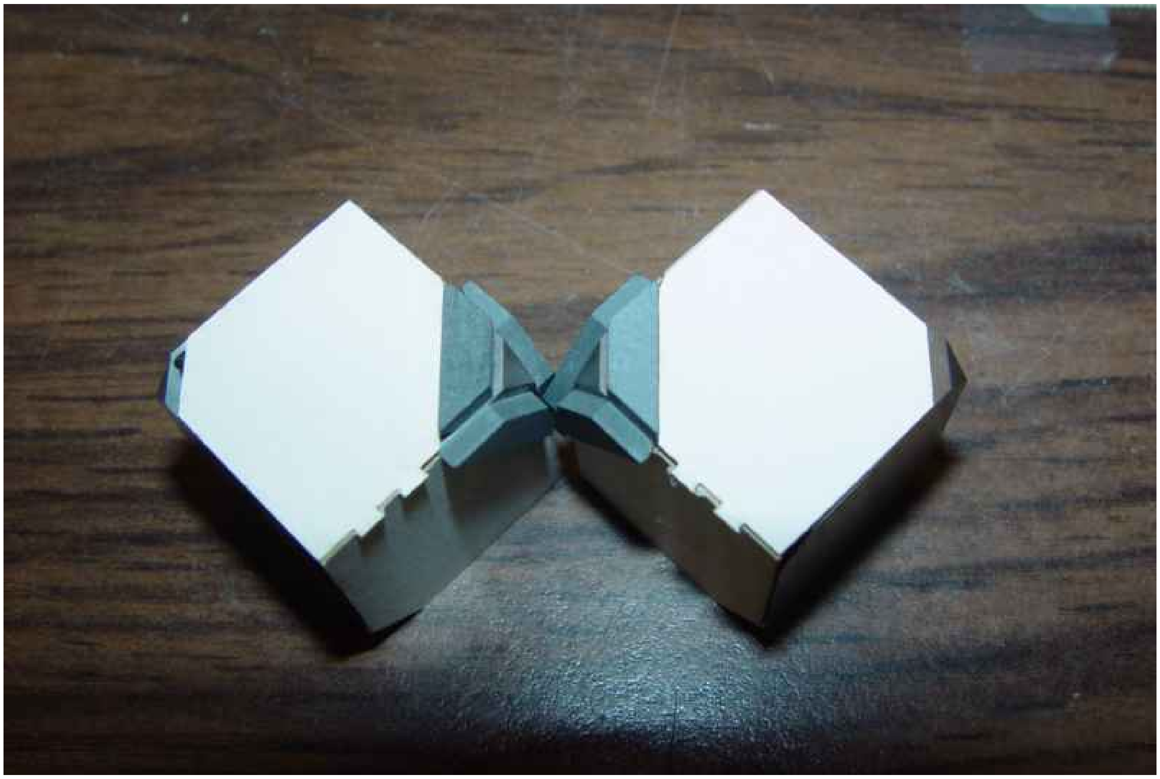
Glue the pyrophyllite pieces, with 2 small dots of glue per piece.



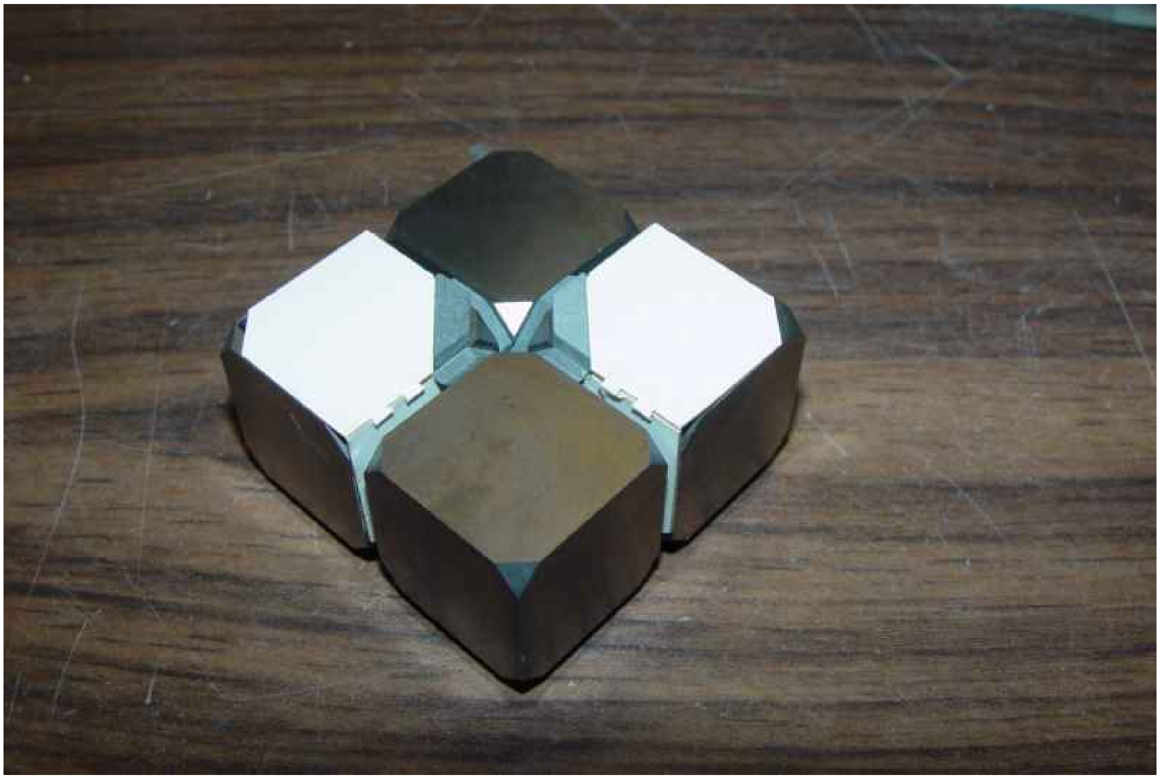
Glue the longer pieces so that they overhang to the left, as shown. The shorter pieces should be placed in the center with no overhang on either side.

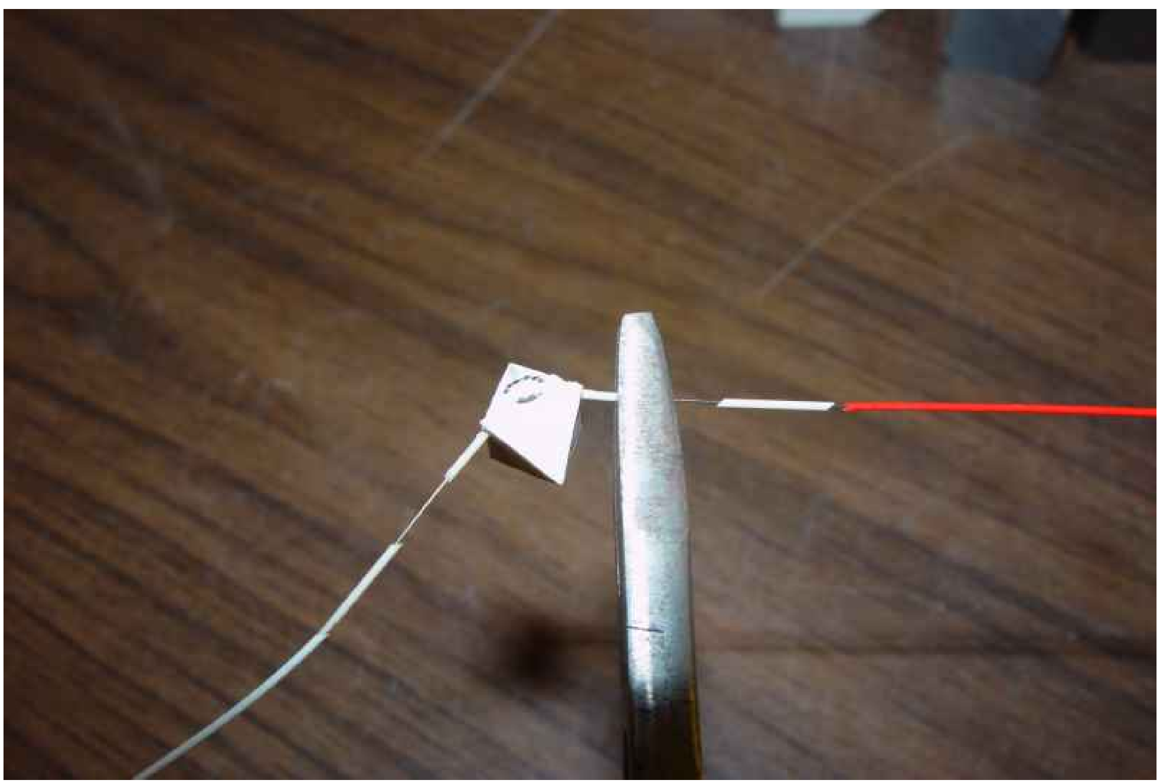


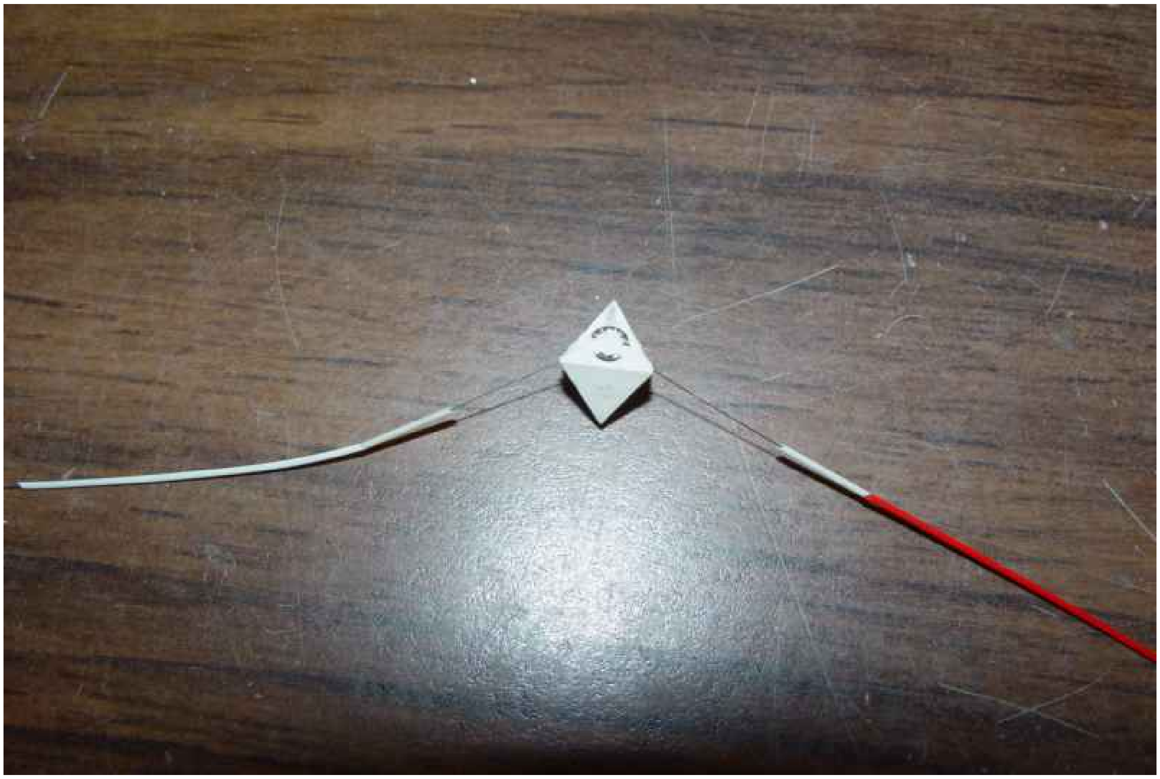
Glue the pyrophyllite to each of the four cubes. If all the short ones are glued in the center and the long ones are glued with an overhang, the final arrangement will look like this..


Pre-arrange cubes 1 and 2 so the gaskets interlock like this, and there are four spaces exposed..

Cut small notches in two of the spaces where the thermocouple will go (looking ahead 5 figures) with a file, large enough for the thermocouple tubing. Make sure the notches are centered well so the tubing will rest properly (this may be adjusted when the octahedron is placed).

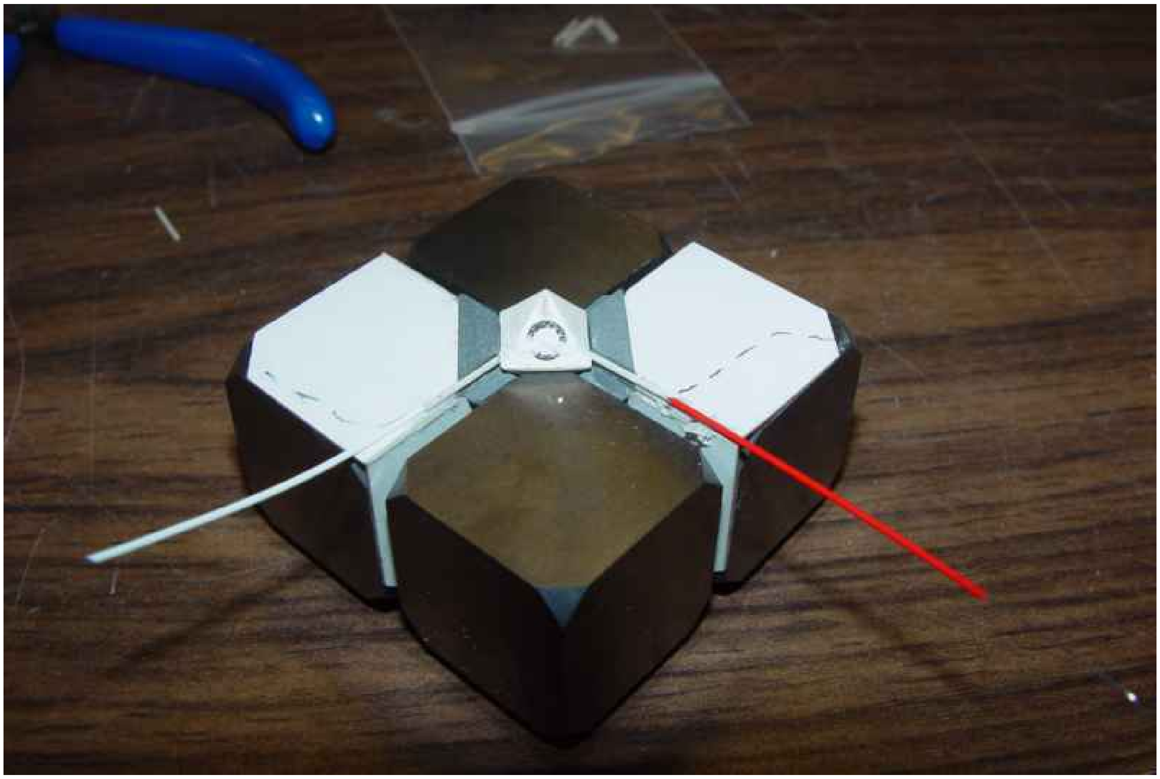

Arrange the four of them on a nesting plate.


Carefully crunch away the thermocouple tubing so that the wire is bare all the way to the octahedron edge, without bending the wire.

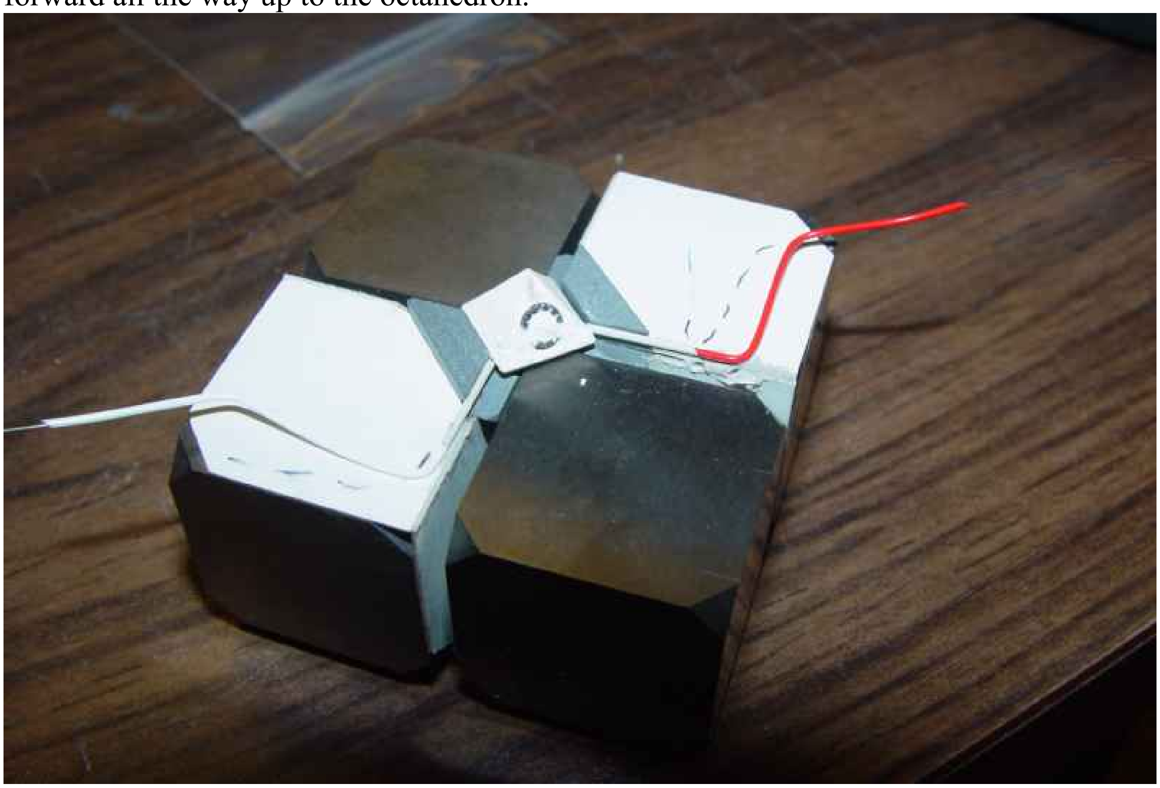


After crunching away the first mullite pieces.

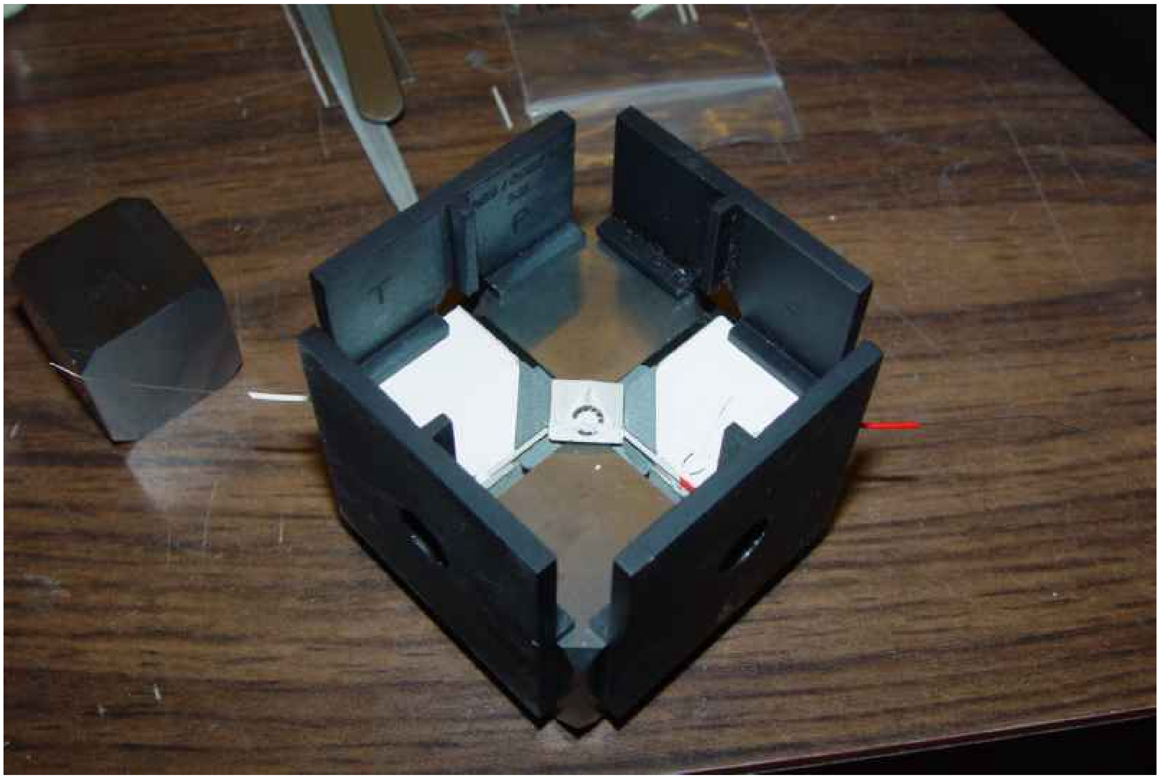
*Using your fingers, with the second mullite pieces right up against the octahedron, bend both wires along (100).



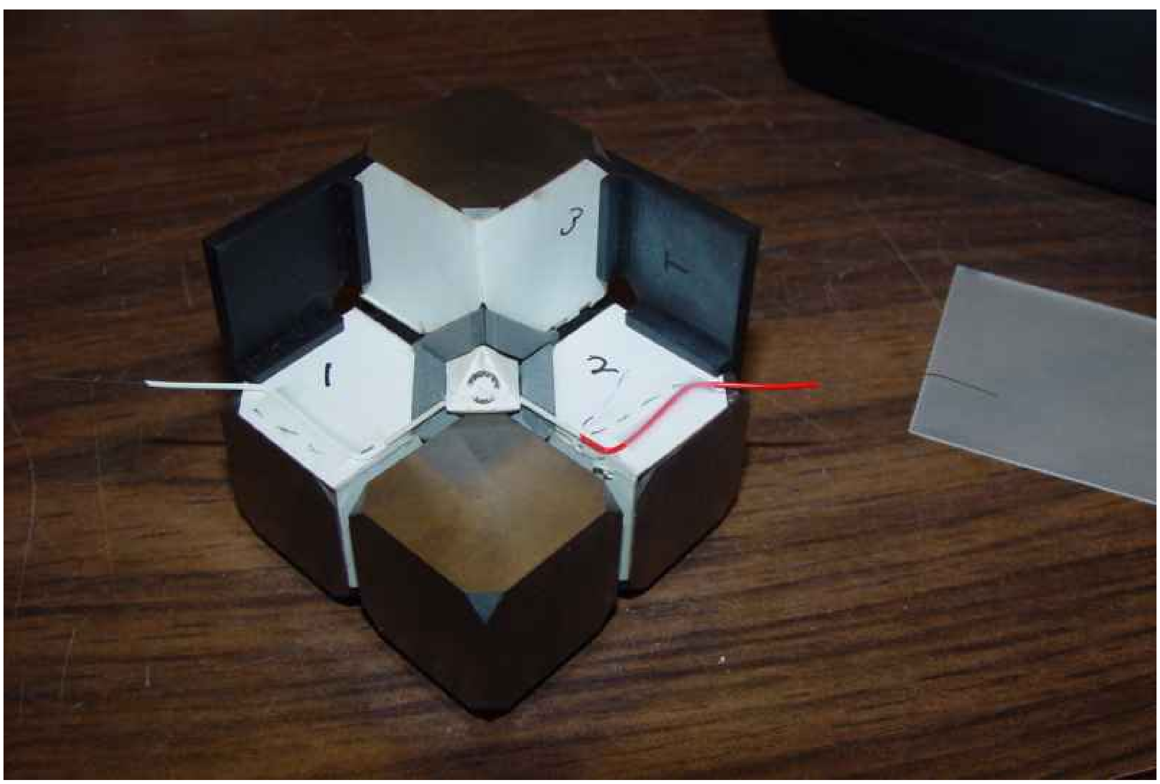
Place the octahedron in the 4 cubes. Make sure the teflon tubing and mullite are pushed forward all the way up to the octahedron.



Bend each wire at a point about 2 mm beyond the mullite, and again as they exit the 4 cubes, as shown. Tape the teflon tubing to the paper with a piece of scotch tape. Verify the centering of the octahedron and mullite tubing before proceeding further.

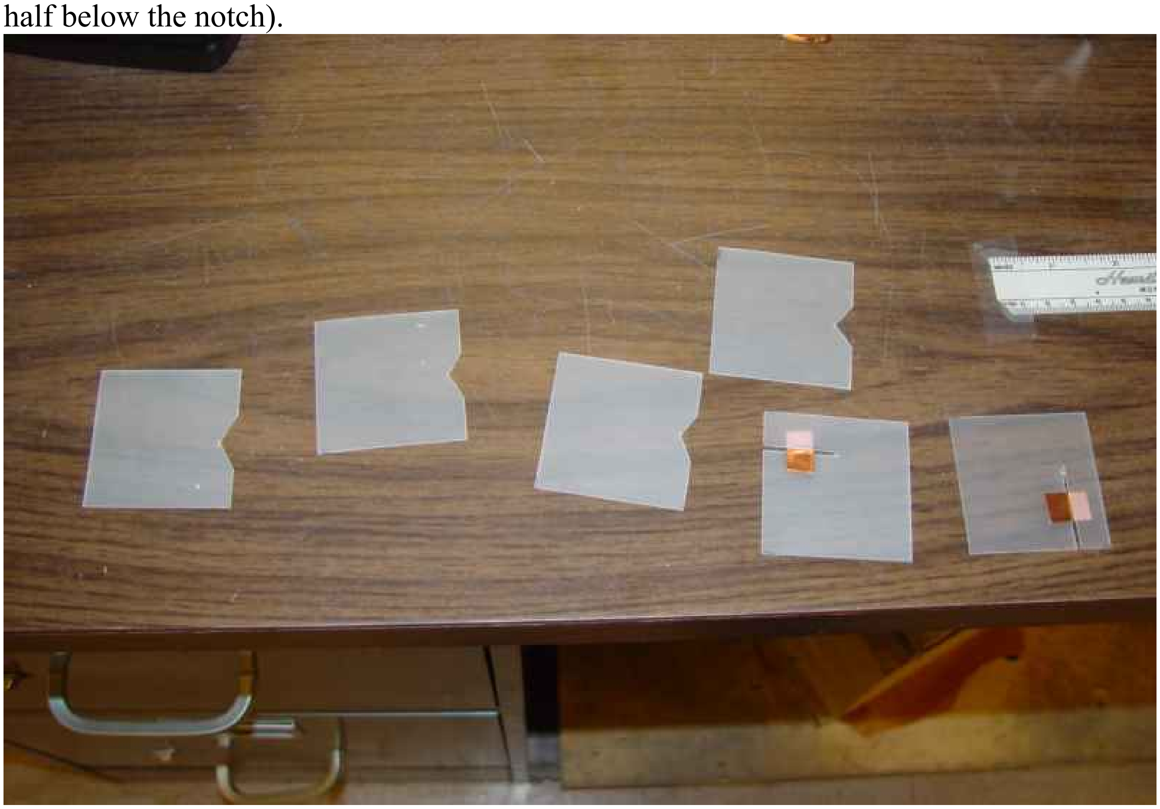


Use four more nesting plates to arrange the other 4 cubes.

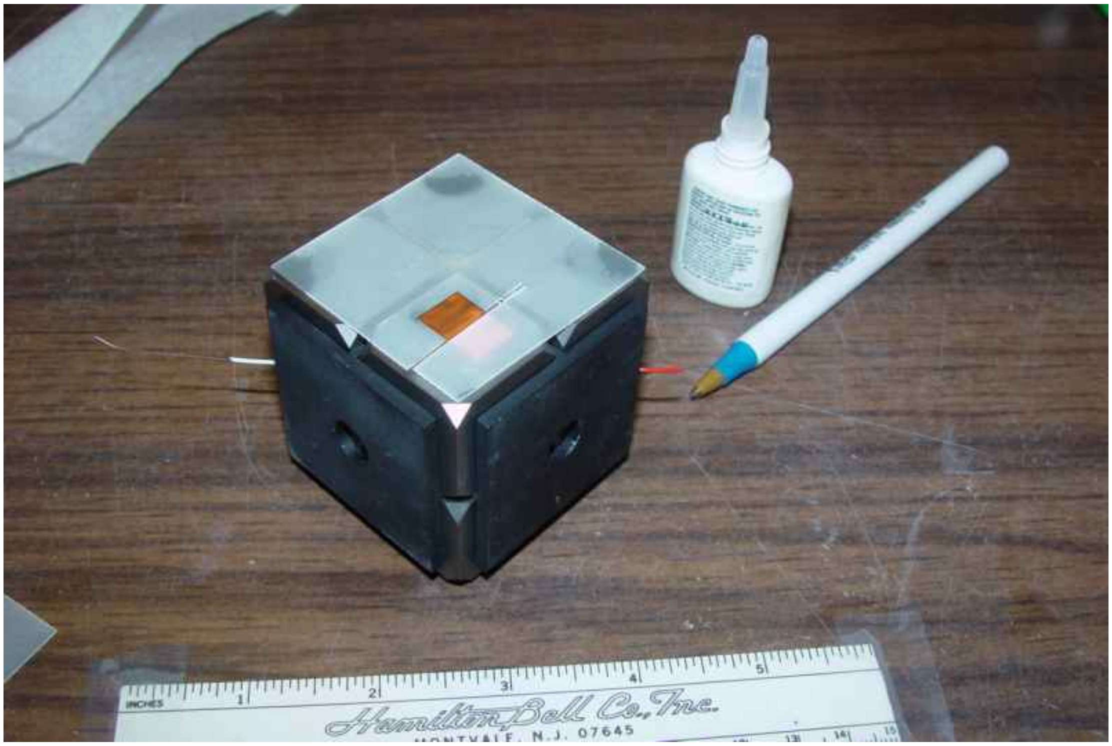


A view of how cube 3 fits into the arrangement (cubes are numbered).

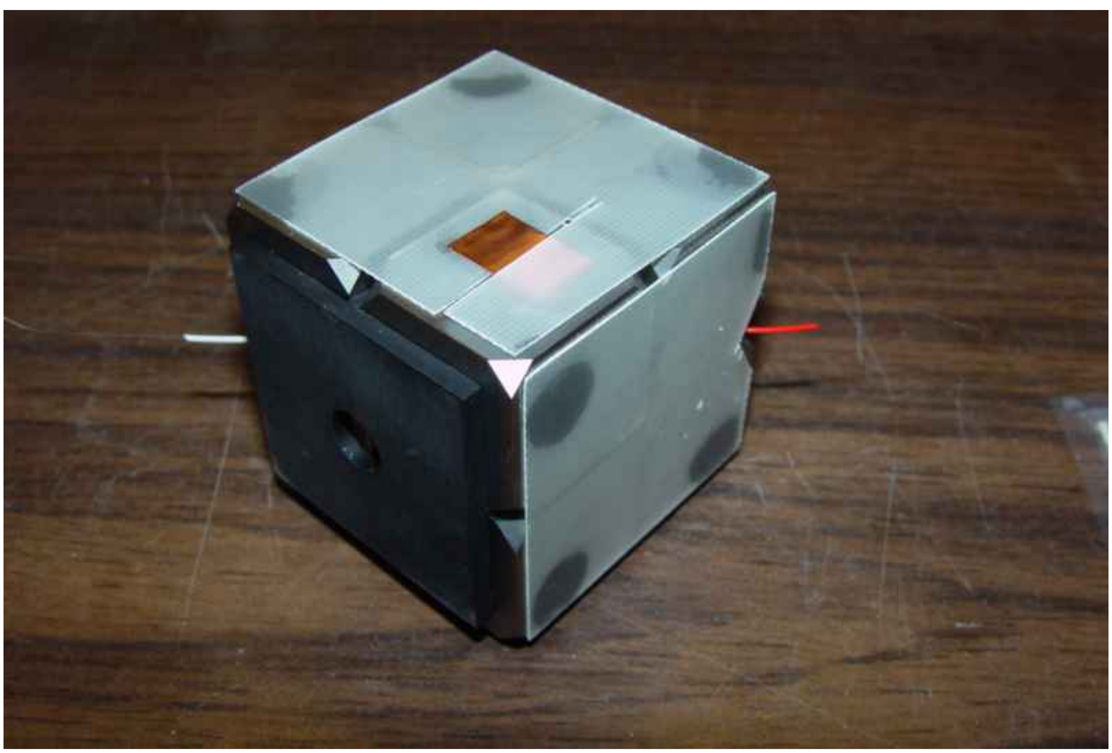
Prepare six 2-by-2-inch squares of G-10. Notch two of them with wide notches, as above, and use crazy glue to put a piece of copper foil across the notch (half above and half below the notch).

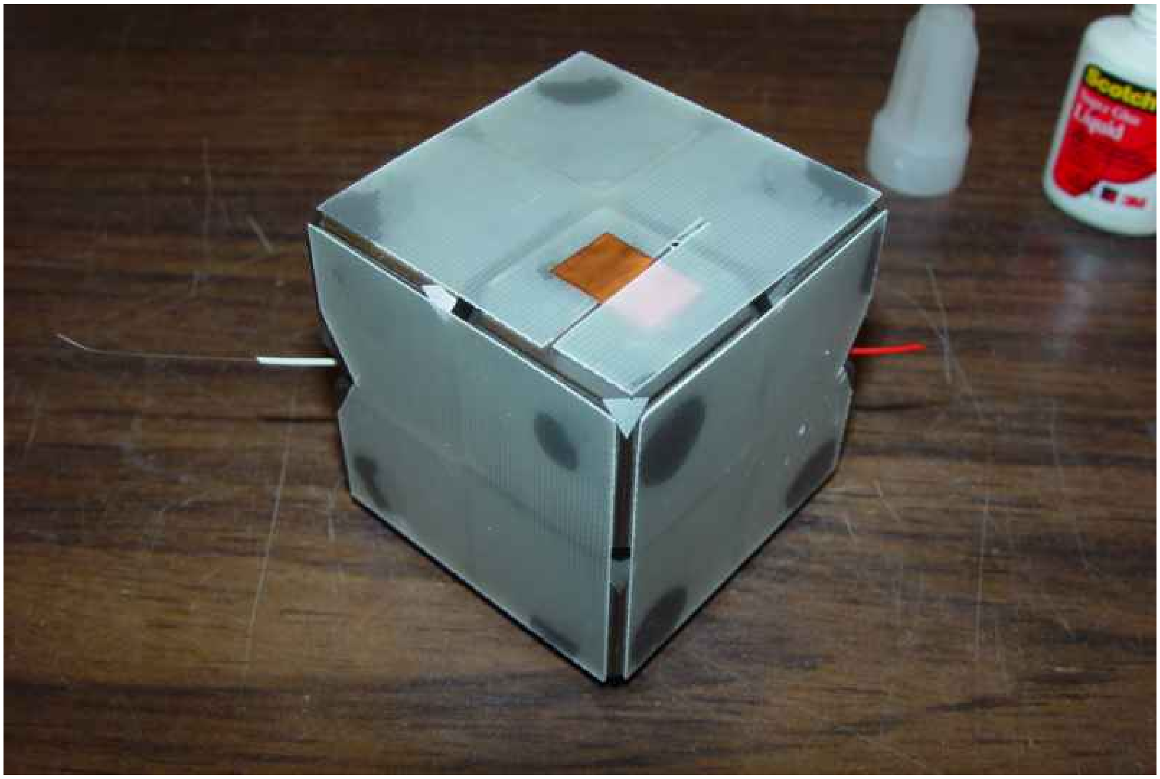


Put little V-shaped cuts in the other four G-10 squares. The set should look like this.



Glue a G-10 square with copper on top of the set of carbide, making sure that the copper is on the same cube that contacts the furnace of the assembly, and that no glue is in the electrical contacts. Let the glue set.


Remove one nesting plate and glue a plain G-10 square to the side, with the V facing the thermocouple wire. Let the glue set again.



Remove another nesting plate and glue a second plain G-10 square on. Do the same with the two back sides, one at a time, without lifting the nest of cubes, and letting the glue set each time. Then, roll the nest of cubes carefully over and glue the second G-10 square with copper on the bottom, again making sure the copper is in contact with the same cube as the furnace. Allow time for the krazy glue to finish setting on the nest of cubes. It is now ready to place in the multi-anvil press.

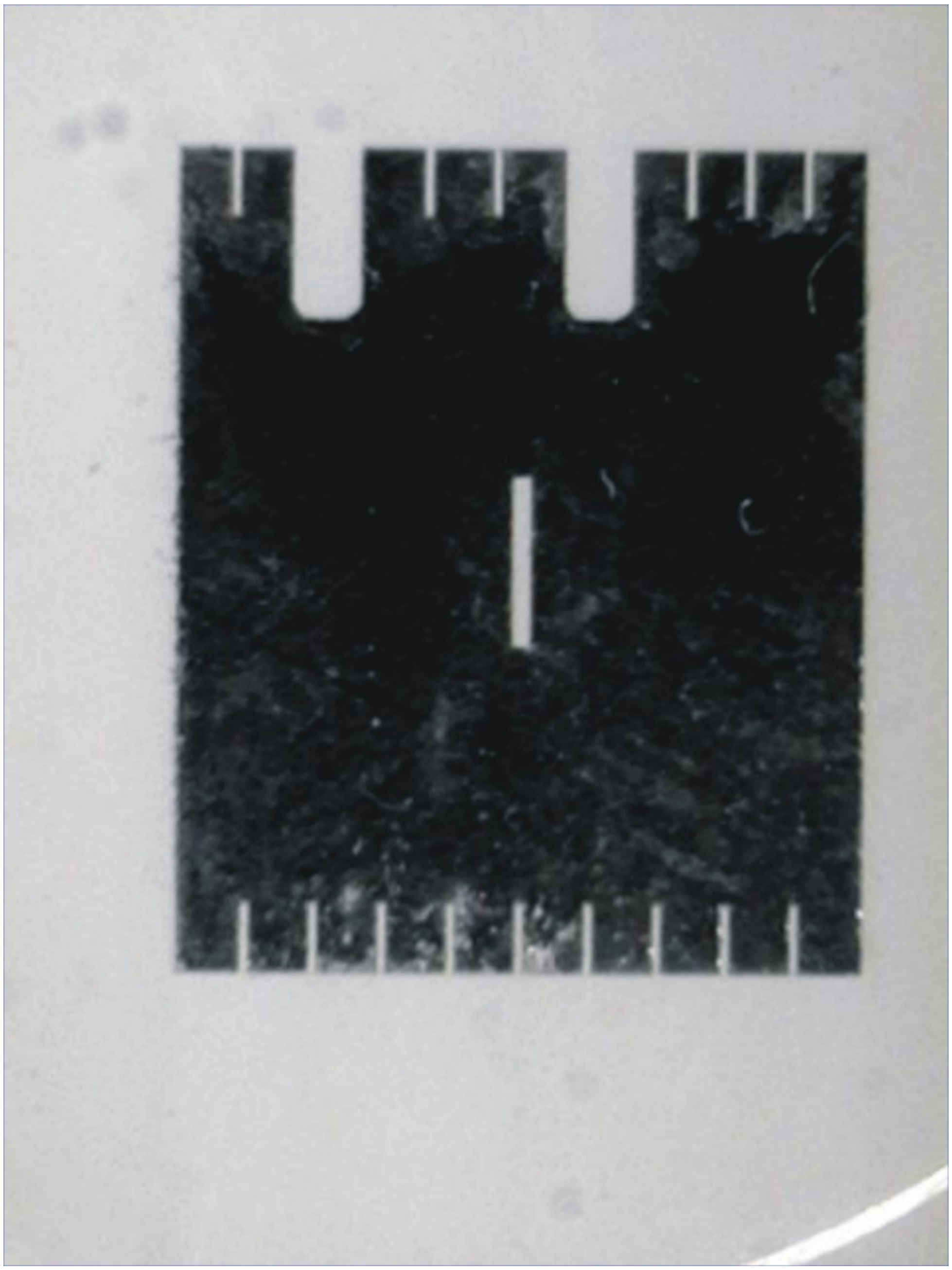
F. Special recommendations for running the assemblies.

The following are suggestions and notes for getting the assemblies to be accurate and well-behaved.

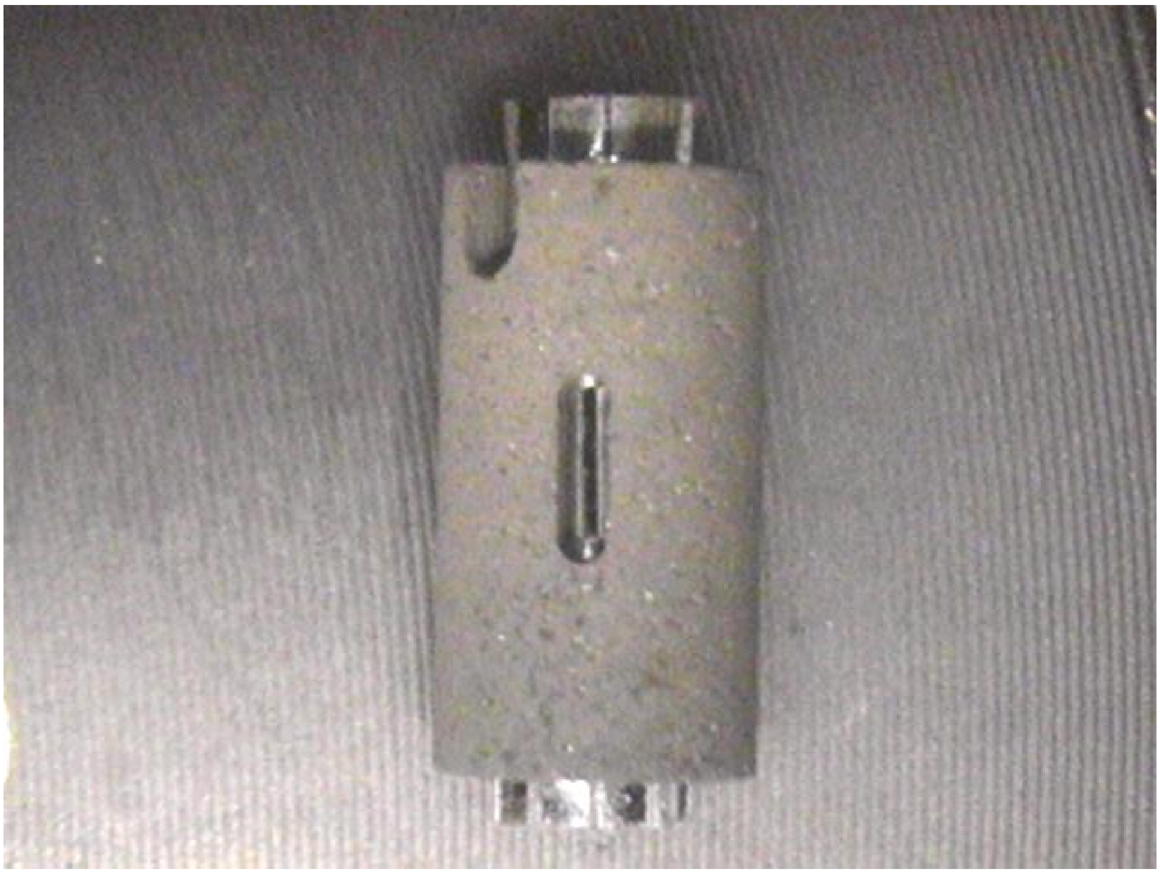
1. Instead of using type C extension wire inside the multi-anvil module to connect to the thermocouple, it is recommended that actual thermocouple wire is used instead. Use teflon tubing to color-code the wire similar to the assembly wires. This wire can be connected to the type C extension wire (available from Omega) outside the module, where the temperature is at ambient. The errors in temperature measurement due to the various connections in the thermocouple circuit will be minimized by doing this. (Thanks to Tami Diedrich).
2. Use the rough pump to raise the ram until it just touches. One trick is to listen for the first "sound" from the assembly, such as a click or other sounds that the assembly makes when the anvils first touch, before any oil pressure rise as seen. This will minimize the pumping-up time without placing stress on the assembly at the beginning (Thanks to Emmanuel Soignard).
3. Pump up at a rate of about 40 tonnes per hour.
4. Start heating after the pressure is reached, and heat at about 100 degrees per minute or less.
5. Any variation from a pressure-up, temperature-up pathway, in that order, may cause the pressure calibration to change.
6. Hold the oil pressure constant during heating to reproduce the pressure calibrations given in this manual.

G. Special preparations for beam time.

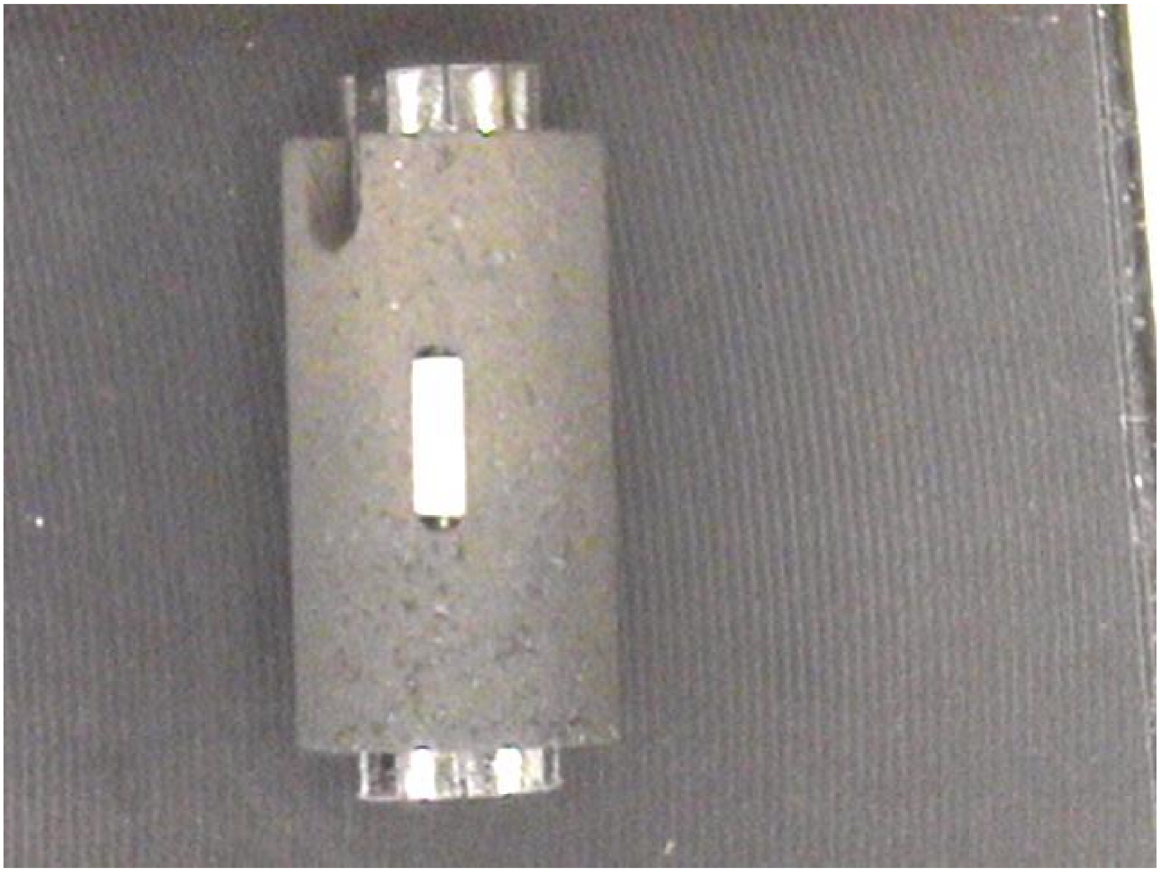
The 10/5 beam line assembly has two modifications. First, the LaCrO3 sleeve has two slits in it, and alumina pieces are provided that are inserted into the slits as windows. Secondly, openings are cut into the rhenium furnace. Special steps need to be taken to make sure that these slits are aligned in the direction of the x-ray beam. The instructions here are appropriate for the Large-Volume Press at the beam line at GSECARS, Sector 13 at APS.

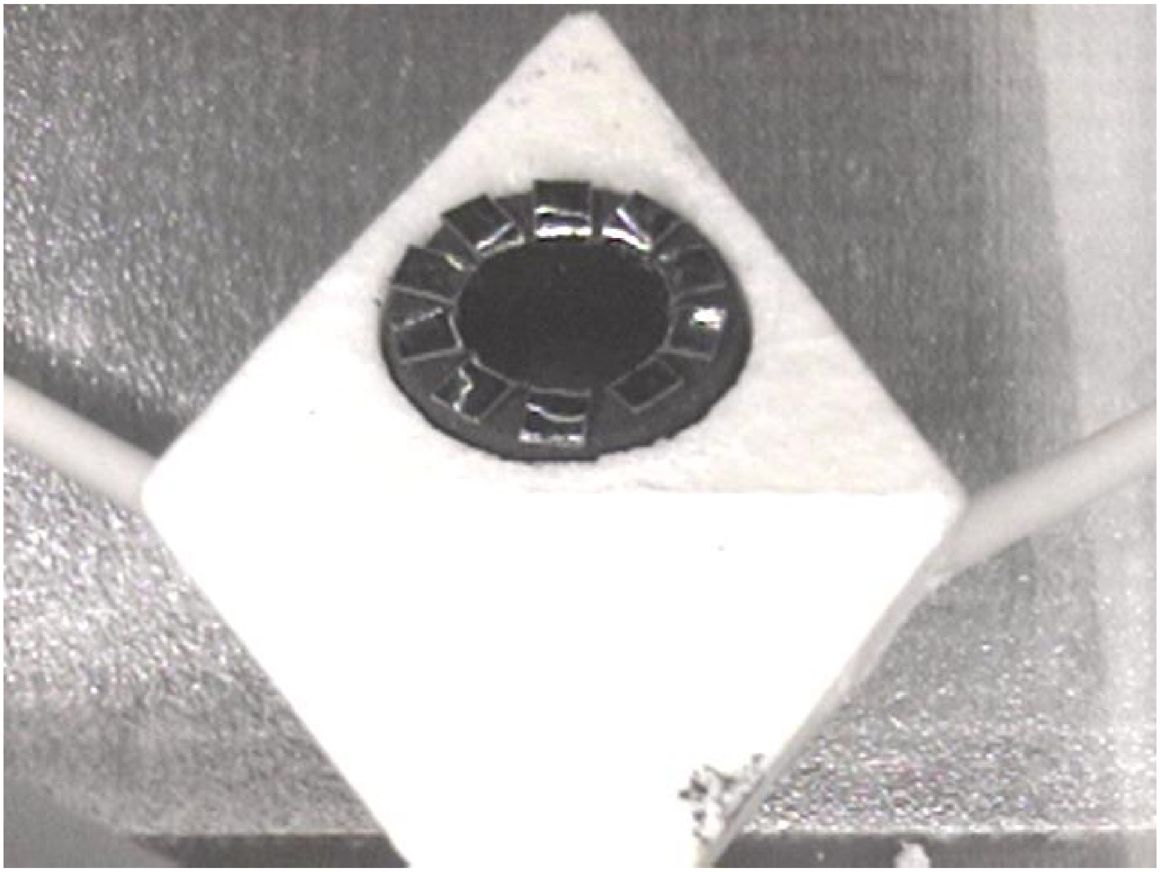


1. Locate a rhenium beam line furnace. Unlike the conventional furnace, the beam line furnace is slitted in the middle, and the thermocouple trenches are off-center, as shown in the picture above.
2. Roll the furnace around the #40 drill pin as before. Note that it matters which side you roll around the pin, because the slit in the rhenium has to line up with the slit in the lanthanum chromite. The above figure shows the *inside* face of the furnace, as it is rolled around the pin. Note that the x-ray slit is to the left of the nearest thermocouple groove when you are looking at the inside face of the furnace.



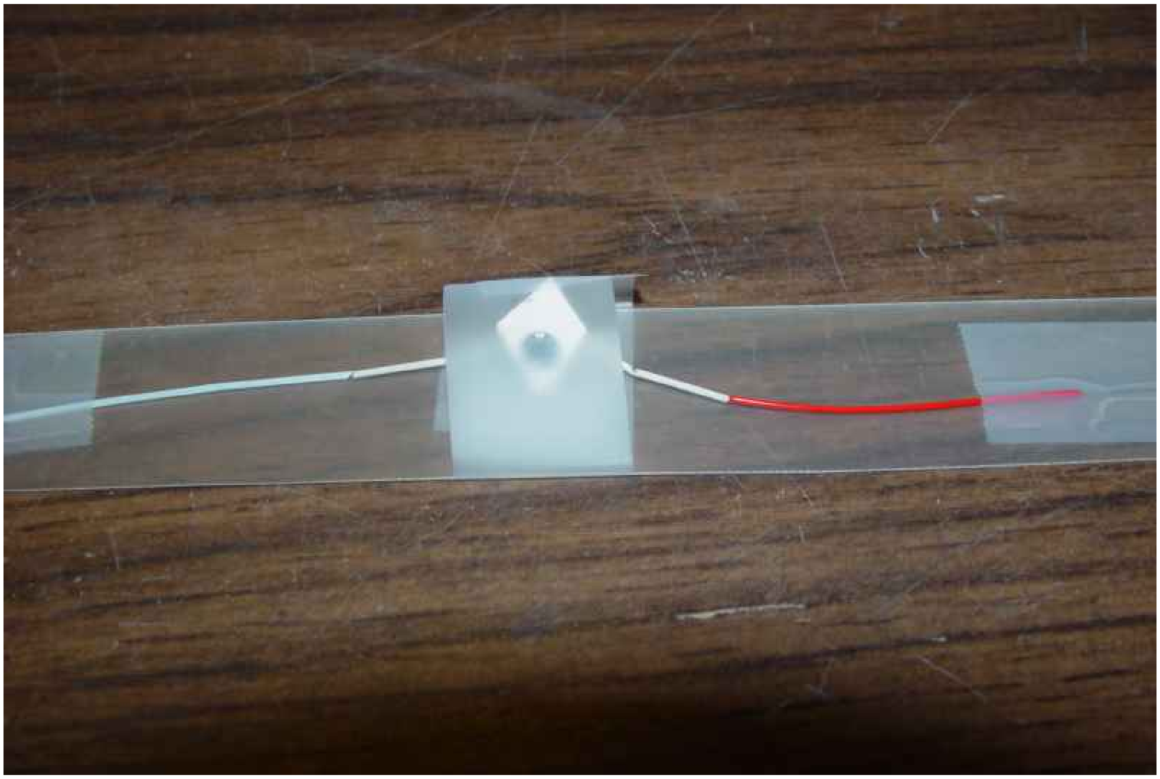
3. Insert the furnace into a slitted lanthanum chromite sleeve with the thermocouple slots in the correct orientation. Move the furnace around until both the front and back x-ray slits in the rhenium are aligned with the x-ray slits in the lanthanum chromite as closely as possible. The thermocouple grooves should match with no adjustment necessary when the slits are aligned (see note 2 if this is not the case).


4. Place an alumina window into the slit in the lanthanum chromite with tweezers, and then push it in with your 2 thumbnails. It should click into place fairly easily and not pop back out. Do the same for the slit on the other side of the sleeve.

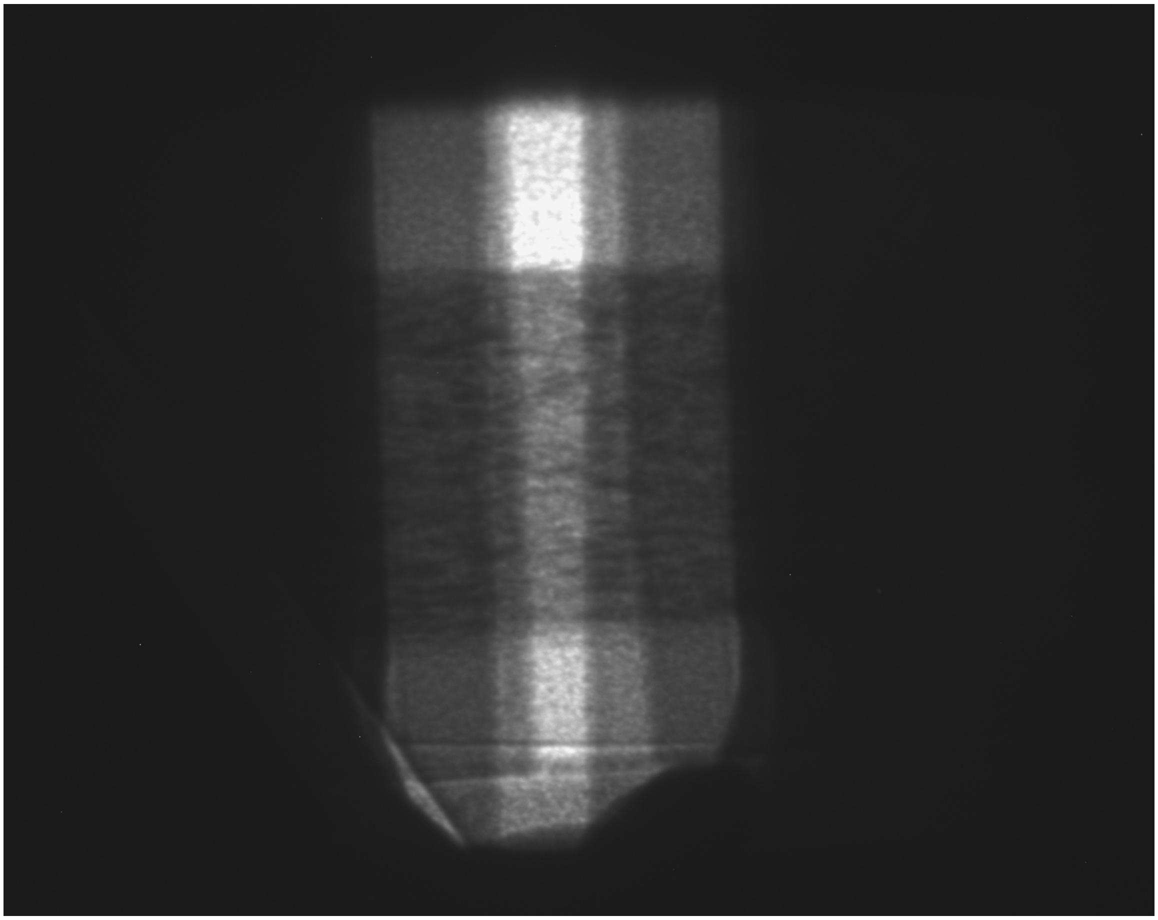


5. Place the sleeve in the octahedron as before. Use a pencil to mark the two edges where the x-ray beam will enter and exit the octahedron, which should line up with the windows in the furnace and sleeve. The pencil mark is visible in the above photograph. Cement the thermocouple as described in section D.

Transporting the assembly to the beam line:



1. Tape the octahedron and thermocouple to a 7- or 8-inch strip of G-10 or other backing material, as in the picture. Be careful not to kink the thermocouple as you are taping it.
2. Cut an 8-inch segment off of a 1” piece of PVC pipe (available at home supply stores) and slide the assembly into it. Tape the ends so the assembly doesn’t slide out.
4. Pack the pyrophyllite gaskets, paper, G-10 squares, and copper pieces. Also, pack the sample sleeves, samples, MgO plugs and ZrO2 plugs.
5. Bring two extra thermocouple “legs” covered with insulation to use as extensions at the beam line. The thermocouples at the beam line need to be 1 foot long to come out of the multi-anvil module that is used there. At the beam line, wind the wires together and crimp with a piece of copper, or use some other connection technique, but be aware that there is potentially only a small space between the anvils, so don’t use anything more than about 1 mm thick as a connector or a lead.
6. Assemble the octahedron, cubes and gaskets at the beam line, making sure to mark the locations of the x-ray windows on the octahedron itself and on the nest of cubes as you put them together.
7. Place the assembly into the Large-Volume Press with the x-ray windows aligned with the x-ray beam and the furnace vertical. The radiographic image should look similar to the one in the picture below.



Radiographic image of a 10/5 assembly. The slots in the LaCrO3 define the wider opening (0.6 mm wide) while the slots in the Re furnace define the narrower opening. The dark horizontal layer is a CsCl pressure standard.