Diamond Anvil Cell Manual

Cell Preparation, ac Susceptibility and electrical Resistivity Measurements,

Accessory Equipment

(version: November 2011)

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March 24, 2014

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1.1 Description of the Diamond Anvil Cell

The diamond anvil cell (DAC) described herein (pictured in Figure 1.1) was designed by Jim Schilling in 1982 and machined at the U. of Delaware. The original drawings are included in the DAC binder. The DAC is designed to reach pressures greater than 1 Mbar. In March 2011, Narelle Hillier reached a pressure of approximately 200 GPa, the highest pressure attained to date. Stefan Klotz designed the experimental setup and more detail can be found in his thesis (in German), in his papers, and in the theses of later students.

The following students have used the DAC for substantial portions of their Ph.D. work: Stefan Klotz (1992), Andrew Cornelius (1996), Craig Looney (1997), Sascha Sadewasser (1999), Shanti Deemyad (2004), James Hamlin (2007), Mathiewos Debessai (2009), and Wenli Bi (2011).

1.2 Checklist for Performing a DAC Experiment

This section gives a step-by-step checklist for performing a DAC experiment. The starting point assumes that the cell is still together after the completion of an experiment. The procedure is then:

1. Make sure that the capillary line coming from the He gas bottle to the top of the cryostat is secured and not very bent. If too much strain is placed on the capillary, especially at the joint with the membrane, it might break.
2. Take the clamp apart and store all of the components in a safe location. Ensure that the capillary line for the diaphragm is securely stored.
3. Clean the diamonds and inside of the clamp thoroughly with ethanol or methanol. (Do not use acetone as it could attack the glue holding the diamonds and coil system in place.)

Figure 1.1: The DAC. In addition to the piston and body, the force plate (which sits on top of the diaphragm, not shown), the retaining ring, and the torquing tool for the retaining ring are pictured from left to right in the foreground.

4. Ensure that the diamonds are still aligned.

5. Punch out a gasket and pre-indent to the desired thickness.

6. Drill the hole in the gasket. Make sure that the hole is centered on both sides!!!!! This is also an indication of how well the diamonds are aligned and whether the piston is wobbling at all. If required (for hydrostatic measurements), sputter gold onto the gasket.

7. If you have previously removed the coil system (usually this is not necessary) or are installing a new one, mount the coil system in the cell if you have not already done so.

8. Check coil system contacts. For the present (2011) side-by-side coil system, for which the primary and pick-up coils are both wound with 60 μm Cu wire, the resistance values should be about 23 Ohms for each coil (primary or pick-up), so the total resistance of the balanced coils will be about 46 Ohms. The resistance of the primary coil should be slightly larger than that of the pick-up.

9. Place an $\sim$100 μm MgB_2 sample in the compensation coil. It can often be placed in a small piece of tape, making it easy to remove at a later time.

10. It has been found that the low temperature background of the susceptibility is better (i.e. flatter) when the position of the dummy gasket in the compensation coil is adjusted so as to give the best compensation. To do this, measure the susceptibility of the body part of the DAC with no gaskets. Place the dummy gasket (same material as the real gasket) on a piece of clay in the compensation coil and balance the real (pre-indented and drilled) gasket on the body diamond - no clay is necessary. Adjust the height of the dummy gasket until the susceptibility signal is as close as possible to the original signal with no gaskets. It is easiest to rest the dummy coil on top of the clay in the compensation gasket and to gradually push it down further into the clay until the desired signal is achieved.

11. Put the (pre-indented and drilled) gasket onto the piston diamond, securing it in place with a small amount of clay. Be sure that no clay gets onto the diamond culet. If the sample is air sensitive and needs

to be loaded in the glove box, a small amount of UHU epoxy may be mixed with the clay so that the gasket will not be accidentally knocked off in the glove box.

12. Obtain a suitably sized sample and place it, along with a ruby piece (or pieces), on the piston diamond.
13. Put cell together and put in cryostat.
14. Check all electrical connections for the DAC and the thermometry. Make sure that the correct resistance is measured and that there are no ground contacts.
15. Pump on the cryostat insulation and the diaphragm membrane line before the first cooldown. (The insulation should be pumped on each time the system is cooled, while the diaphragm line should be pumped during every cooling prior to the application of pressure.)
16. Make sure that the ruby R-lines are visible.
17. Obtain a zero pressure measurement and make sure that the transition and background noise are as expected.
18. Load He pressure medium if performing a hydrostatic measurement
19. Normally measurements are made by increasing pressure in the temperature range 130-160K. Pressure can, however, be increased at any temperatures as long as the temperature is above the melting curve of He in the diaphragm.
20. When done, release the pressure very slowly (1 bar of diaphragm pressure per 15-30 minutes).

1.3 Care of the Diamond Anvil Cell

1.3.1 Diamond Care and Installation of New Diamonds

It is important that the diamonds are cleaned thoroughly before proceeding with any step involving the clamp. The diamonds are cleaned with ethanol or methanol using either Q-Tips or Kimwipes wrapped around tweezers. (If

the diamonds are still mounted in the cell, do not use acetone, as it attacks the glue holding the diamonds and the coil system in place.)

Every so often, one or both diamonds need to be replaced in the clamp. If only the piston diamond needs to be replaced, no disassembly is required (other than removing the piston from the cell). If the diamond in the cell body needs to be replaced, the CuBe backing plate must be removed to allow easy access. Any new diamond should be cleaned thoroughly in the fume hood prior to mounting in the cell. (Trichloromethane and ultrasound will clean the diamonds well. Chloroform also works well, and acetone can also be used.)

A thin (25 μm) disc of zirconium foil sits between each diamond and its backing plate. If the diamond needs to be replaced, the zirconium foil probably needs to be replaced as well. To punch out a zirconium disc, secure a piece of 25 μm zirconium foil between two thin sheets of, for example, brass; the 25 μm foil is too thin to be punched out by itself. Put the "zirconium sandwich" in the gasket punch (see subsequent section on gaskets) and punch out the disc the same way you would punch out a gasket. Remove the zirconium from the middle and press it by hand between two hard flat surfaces in order to smooth it out. Next, center the disc over the hole in the backing piece (WC, B_4C, or Russian alloy [NiCrAl]), and poke a hole in the disc, pushing the foil against the insides of the hole in the backing. Previously, the extra material was gently sanded away. More recently, however, the excess material is simply left in the hole. As long as the excess foil is pushed up flush against the sides of the hole so that optical access will not be hindered, this will not cause a problem.

The clean diamond is then placed on the zirconium foil, centered over the hole. When placing diamonds, it is best to position the facets such that the diamonds will be in relative rotational alignment with each other. Make sure that the foil can be seen coming out from underneath the diamond on all sides. It should be possible to center the diamond within 250 μm of the center of the optical hole in the backing; the closer to the center the better. To hold the diamond and foil in place while the glue dries, a modest load should be placed on the diamond. Andy cobbled together a simple device for this purpose, consisting of a plexiglas piece that fits over the piston and the lower (cell) diamond support plate (the same diameter

plexiglas works in either case) and a cylindrical metal piece that fits snugly in the plexiglas and puts the diamonds under a modest load, with a piece of aluminum foil between the metal piece and the diamond for cushioning. The lab now has more sophisticated "gluing jigs," shown in Figure 1.2, which effectively immobilize the diamonds while a modest force is applied by a cylinder of plexiglas. The body (aka "lower") diamond and its backing plate fit inside the jig on the left, while the jig on the right bolts securely onto the piston. For both jigs, tightening the screws on the top pushes the plexiglas cylinder onto the respective diamond faces, thereby moving the diamond slightly. To view the centering, place the gluing jigs upside down on top of the light source so that the light passes through the plexiglass before reaching the diamonds. Once the diamonds are well-centered, they may be glued to the backing pieces by placing glue around the base of the diamond (using a needle, for example). The glue is made by mixing *Stycast 2850FT black* epoxy with 7.5% wt. of an epoxy resin hardener *Catalyst 24LV*. Be sure to mix it well (about 10 minutes) to ensure that the catalyst starts working and that the mixture is homogenous. The glue radius should extend beyond the zirconium foil. No glue goes under the foil or between the diamond and the foil. Be careful not to put too much glue around the diamond, as it may get in the way of placement of the coil. After gluing the diamond in place, allow the epoxy to harden slightly at 75°C for about 20 minutes. The diamonds should then be aligned. Afterwards, the gasket should be pre-indented, as explained in a later section. Typically, this is just a rough pre-indentation, as the diamonds may still shift slightly. It is also a good way to check the alignement. Leaving the cell closed, the glue should then be allowed to harden completely by leaving it at 75°C overnight. Note that it is important that the facets of the diamonds are in reasonable rotational alignment before the glue dries because the bottom plate can only be moved a couple of degrees – see section on diamond alignment. Also note, if replacing the bottom (body) diamond, make sure there is a piece of ruby on the inner edge of the optical hole in the backing plate. This serves as the "reference" ruby for the pressure measurement (see section on measuring the pressure).

1.3.2 Diamond Alignment

After replacement of one or both of the diamonds, the diamond alignment procedure must be performed. In addition, the diamond alignment must be

Figure 1.2: Jigs for supporting diamonds during gluing. The cell-body backing plate sits in the jig on the left, while the jig on the right slides over (and can be securely fastened to) the piston.

checked following each experiment (before gasket pre-indention), and corrected if necessary. *Most often, the cause of broken diamonds is misalignment.* There are 3 types of alignment: translational, rotational, and planar, and the beginner should perform them all under the high-powered Nikon metallurgical microscope. Later, when one can confidently and smoothly move the piston in the cylinder (this takes some practice), it is possible to do all of this under the stereo microscope. Using the high-powererd Nikon microscope may still be preferred, however, for very small culets, as it is easier to tell whether they are properly aligned. Just keep in mind, however, that both the X and Y directions are reversed in the metallurgical microscope.

Translational Alignment:

In translational alignment, the centers of the diamond culets are aligned. First, bring the diamonds close together without touching. The safest way to do this is to place the piston on the teflon support piece and slowly lower the cell body over the piston. Make sure that the piston and cell body are in the correct rotational orientation with respect to each other. The two brass spacers should be alongside the teflon piece. Their height, and the height of the teflon piece, is designed such that when the cell body is resting on the spacers, the diamonds will not be in contact with each other. With the spacers still in place, put the retaining screws in place and lower the side screws so that the piston is held firmly in place. The brass spacers can then be removed. Slowly bring the diamonds closer together by slowly loosening the side screw, all the while gently pushing the cell body down. This is to ensure that the piston is moving smoothly and continuously. If it gets stuck, it could slip, allowing the diamonds to hit each other suddenly, which might cause damage. It is usually a good idea to observe the diamonds through the microscope while lowering the cell body over the piston. Gradually decrease the separation of the diamonds until you can toggle between views of one culet or the other by slightly adjusting the focus. If the coil system is out of the cell, it is possible to observe the diamonds through the side holes in the body of the cell - this can be useful in alignment, as it will be apparent if the alignment is way off, and some adjustments may be made before performing the final alignment using the microscope. The CuBe plate (on which the body diamond is mounted) is held in place by two screws on the bottom of the cell. These must be loosened slightly to allow the plate (and thus the

diamond) to be moved. There are three additional screws on the side of the clamp that allow the bottom plate to be moved horizontally. To tighten one of the screws, one or both of the other screws often needs to be loosened first. Move the screws until the centers of the diamond faces are aligned. Be patient, and make small adjustments to the position of the diamonds, so as not to accidentally make it worse by adjusting it too far.

Rotational Alignment:

Note that this next step is often not necessary if care is taken while mounting the diamonds to align the facets. The procedures is still included, however, for when such adjustment is required. Rotational alignment is done by slightly loosening the two screws (previously mentioned) at the bottom of the cell that secure in place the CuBe plate to which the bottom diamond is attached. The holes for these screws have been made in such a way that the CuBe plate can be rotated by a couple of degrees. It will probably be necessary to slightly loosen the 3 translational adjustment screws (on the side of the pressure cell, mentioned above) to allow the plate to rotate. Since the facets are not perfect on the diamonds, a perfect alignment cannot be obtained, but it should be possible to align most of the facets to within 5%-10% of the length of a facet-side. After the rotational alignment is performed, it will probably be necessary to go back and fine tune the translational alignment. Repeat both procedures until both the translational and rotational alignment are satisfactory.

Planar Alignment: Beginner's Procedure

Next, the planar alignment procedure is performed to make sure that the faces of the diamonds are parallel. Place a large amount of salt on the upper diamond and GENTLY put the piston in the clamp. Do not put the top plate on the clamp during the alignment procedure so the three screws that move the rocker (on the piston) can be accessed without taking the plate off every time. Replace the bottom lamp on the high-power Nikon with the Na lamp. The Na lamp requires a couple of minutes to warm up. Under the microscope, manually apply force until the Na rings become visible. The fringes occur due to the thickness of the Na layer. If pressure is applied more than a couple of times, it is necessary to put on more salt as the salt layer will

become too thin (and the lines can be distorted, even when the alignment is perfect!) Make sure that the rings are centered on the diamonds by using the lines on the eyepiece. Simply count the number of fringes from the center to each edge. The upper diamond is on a rocker, and it is adjusted so the pattern is centered and the diamond faces are parallel. There are three screws that move the rocker. If the center of the pattern is completely off the diamonds, a decent amount of movement of the rocker is necessary. First, remove the force on the diamonds. Loosen two of the screws ($<10°$) and then tighten the third screw. (Tighten the screw or screws where the fringes are densest. Tightening a screw pulls the rocker up, thus increasing the diamond separation in the area of the screw. Note that both X and Y directions are reversed under the metallurgical microscope. Also remember that the clamp is upside down when under the microscope, but right-side-up when tightening/loosening the rocker screws. Thus, one must be careful about keeping track of the DAC orientation so as not to worsen the alignment with an adjustment!) If the alignment becomes so bad that the center of the pattern is not even on the diamonds, take the piston out of the cell and adjust the screws until the CuBe plate appears (to the naked eye) to be level with respect to the rest of the piston. This should at least get you in the ballpark. Once the center of the pattern is near the center of the diamonds, it is often adequate to tighten one screw without loosening the other two screws. Clean off the salt with methanol or ethanol (it may be necessary to first use the sandpaper which is on the end of the screw tool to remove the compressed salt), and the diamonds are ready.

Planar Alignment: Advanced Procedure - (Caution: Do not use this procedure unless you are VERY comfortable working with the cell, and have 5 or more successful measurements under your belt.)

Assuming the translational and rotational alignments have been performed, the diamonds are brought close together, as in the above description of the translational alignment procedure. The cell is oriented so that the piston is on the bottom (on the Teflon cylinder) and the body is on the top, so that the diamonds can be viewed through the microscope (binocular or metallurgical). The diamonds are then slowly brought all the way together and allowed to touch, by (for example) slowly backing off the safety screws. It is essential that the cell slides down very slowly and smoothly and gently.

The mildest of sudden movements could damage the diamonds. When the diamonds are all the way together, and the cell body is resting on the piston with its own weight, interference lines will be visible where there is a gap between the planar surfaces. The planes are closer together where the lines are closest together. Another way to tell where there is a gap between the diamonds is to observe the appearance of the interference fringes as the diamonds are gently brought into contact. Fringes will first appear on the side where diamonds the diamonds touch and will move toward the side where the diamonds are farthest apart. After separating the diamonds, make the appropriate adjustments using the three screws that hold the hemisphere in place. Tightening a screw pulls the rocker on that side, therby increasing the diamond separation on that side. Then bring the diamonds back together to check the alignment. If the fringes have gotten farther apart, you are on the right track; if they have gotten closer together you have made things worse. Repeat this procedure until the fringes disappear. After completing the planar alignment, it may be necessary to readjust the translation alignment. Keep alternating these alignment procedures until both translational and planar alignment are satisfactory.

1.3.3 Backing Pieces

Originally the diamonds were backed with B_4C, but tungsten carbide (WC) and a Russian Alloy (RA) are currently being used (since 2003 for WC and since 2006 for RA). These are less prone to cracking. Note that the Russian Alloy is Ni(Cr 39-41%)(Al 3-4%). More recently (2010), we have obtained some RA and WC backing pieces with a 15° half-angle cone in them. This improves viewing and optical measurements.

It is not uncommon for there to be tiny cracks in the backing pieces (to which the diamonds are glued). Very often, the cracks might not even be visible because they are obscured by the glue used for attaching the diamonds. Tiny cracks that do not cause any upheaval of the surface are probably OK. However, if the cracks grow so large that they have caused the diamond to pop off (due to an unlevel surface), then the backer must be replaced. Damage to the surface, including scratches and gouges from breaking diamonds should be removed using the diamond sandpaper on the grinder / polisher in the lab. Secure the loose backing piece to the holder using a small amount of

Figure 1.3: Picture of the jig (made by C. Looney) for removing the backing from the CuBe backing plate. The backing plate is placed upside down on the disc on the left, and the cylinder on the right is placed over the top. The dowel in the foreground is placed through the hole in the top of the cylinder and is used to press out the backing, with help of the hydraulic press. If using the backing pieces with the 15° cone, make sure the appropriate-cone-shaped dowel is used to remove the backing piece so that the force is evenly distributed and the backing piece doesn't break.

molten wax. Make sure that the backing piece is perfectly level.

Before removing the damaged backer, any glue holding it to the CuBe plate should be removed. Putting the backer/CuBe assembly into chloroform with ultrasound for a couple of hours should be sufficient. (Trichloromethane should also work; acetone also attacks the glue but is not as effective.) Next, press the damaged backer out of the CuBe using the hydraulic press and the jig made by Craig Looney (see Figure 1.3). Usually the backer comes out with the application of less than 100 lbs. under pressure. If removing a backing piece with the 15° half-angle cone, be sure to use the dowel with the cone on the end.

When the backers are slightly undersized, they need to be glued in place using the following procedure. Before putting in the new backer, make sure

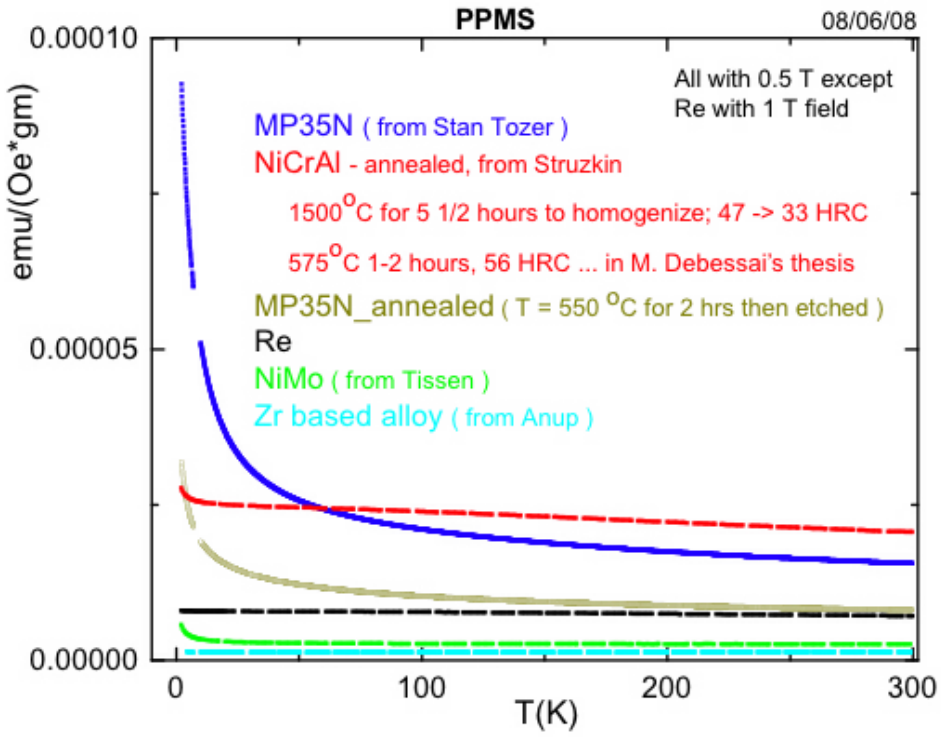

Figure 1.4: PPMS measurements on various gasket and backing piece materials show magnetization versus temperature.

both its surfaces and that of the backing plate are clean. Mix the UHU epoxy, and put a thin film of it in the cavity of the CuBe backing plate. Put in the backer, making sure that the taper is in the proper direction. Also, make sure that the glue does not cover the optical hole in the backer. Put the assembly in a vice and heat with a heat gun to cure the epoxy, being careful not to exceed 200°C or the hardness of the CuBe may diminish. See Table 4.1 for curing information for the UHU epoxy.

The WC and RA backers currently being used fit snugly into the CuBe backing plate and should be pressed into place using the hydraulic press (up to ∼1 ton). No glue is needed. Be careful to place the backing piece in the

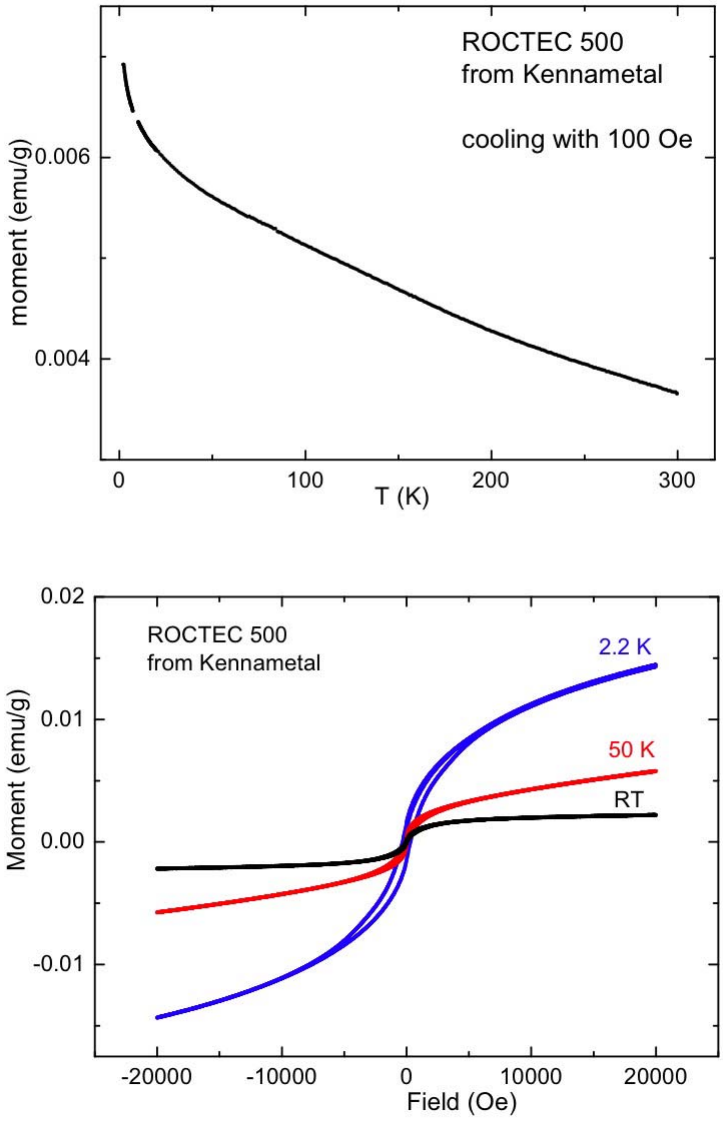

Figure 1.5: PPMS measurements on a cylinder sample of WC (ROCTEC 500) from Kennametal (from Wenli Bi's thesis).

backing plate or hemisphere with the 1° taper in the correct direction. Before inserting the cleaned backing pieces, place a small amount of molykote on the (clean) inside surface of the backing plate. This helps the backing piece to slide in smoothly and makes it easier to remove later.

It is important that both gasket and backing piece materials are non-magnetic, otherwise a large temperature-dependent background will be present in susceptibility measurements. Mathew Devessai performed PPMS measurements on various materials to test their magnetic properties. The results can be seen in Figure 1.4.

The WC backing pieces with the 15° half-angle cone were made by Kennametal. Before having the new pieces manufactured, the magnetic properties of a small piece of the material (ROCTEC 500) were measured in the PPMS. There is a very small ferromagnetic impurity at room temperature, but the temperature dependence is small, as seen in Figure 1.5. Refer to Wenli Bi's thesis for more details. In subsequent susceptibility measurements, the temperature-dependent background has proven to be small.

The Russian Alloy backing pieces with the 15° half-angle cone were made in the department machine shop. First, a disk of the appropriate thickness was cut from one of the several rods of Russian Alloy material belonging to the lab. Next, the disk was cut into three pieces of the same size. These pieces were annealed and homogenized in the oven for 1 hour at 1200° C and then water quenched. The resulting material is very soft. It is from this material that the backing piece with the 15° half-angle cone were cut, with the exception of the 1° cone on the outside. The outer diameter was also left very slightly larger. These pieces were then hardened in the oven at 750° C for 2 hours. The dimensions may change very slightly upon hardening, hence the reason why the 1° cone on the outside was not cut earlier - this dimension is very critical. The machine shop technicians then able to cut the 1° cone on the hardened backing pieces.

1.3.4 Cleaning the Diamond Anvil Cell

After each experiment, the clamp is taken apart. Clean all parts of the DAC, especially the piston and cylinder, to make sure that it is easy to put the piston in and out. Make sure the wires are all okay. It is a good idea to

use the multimeter to make sure all of the connections are good. The radial connectors to the BNC cables have, on occasion, had the ground short out the normal connection. They become stressed at the point where the DAC ends and often break. A small amount of time when checking things out can save a lot later. It may be necessary to clean the back of the upper diamond to insure that the light through the fiber optic cable reaches the cell. The cleanliness of the diamond can be checked by looking at the piston through the stereo microscope; it is possible to focus on the back of the diamond from above. Simply focus on the culet like normal and then move the focus down. The bottom of the diamond will then become visible. If the back needs cleaning, find some of the thin walled hollow tubes (colored heat-shrink tubing) and cut a piece of the appropriate length. Wedge a small piece of Kimwipe in the tube and wet with methanol. (Do not use acetone, which will erode the glue and can cause the diamond to come off — this happened to C. Looney). Gently twist the tube on the back of the diamond. Make sure the wedge is firmly in place or it might come out on the diamond, and it is very hard to get it out. (It took Andy a day and a half to get a piece out once - he finally got it out by using a syringe to get it unjammed and one of the tubes with a small piece of chewing gum to remove it.)

1.3.5 Gasket Preparation

A gasket ($\sim$2.8 mm diameter) with a central bore (size will vary depending on the culet size) constitutes the pressure cell into which the sample, pressure medium, and ruby are placed. Various materials have been used as gaskets in high pressure experiments. Gasket materials must be both hard and ductile. In addition, for susceptibility measurements it is important that they are non-magnetic and non-superconducting. Materials which have been used include:

- Ta(W 10%)
- W(Re 25%) ... superconducts at $\sim$ 5 K
- Re ... superconducts at $\sim$ 5 K under strain
- Ni(Cr 39-41%)(Al 3-4%) ... Russian Alloy
- Ni(Mo 15.3%) ... from V. Tissen

- CuBe ... annealed at 315°C for 3 hours

A new gasket must be made for each experiment. The gasket sheet needs to be rolled down if the thickness is greater than desired. Currently, gaskets are approximately 250 - 300 μm thick (but gaskets up to 500 μm have been successfully used). Andy rolled out his gaskets (which were 90% Ta and 10% W) using a roller from the machine shop (located in the sheet metal room). The gasket material can damage (and has damaged) the rollers, so care needs to be taken. Roll down the metal slowly, and make sure the rollers are clean in the area that you will be rolling the metal. Always roll in the same direction and note that the edge that goes through the roller first will be quite a bit thinner than the rest of the gasket material. Due to this large gradient in the metal thickness, Andy only rolled enough to make one row of gaskets at a time. Because of the difficulties with the Physics Department rollers, subsequent (90-10 TaW) gasket material was rolled out by David Kralik on 7/8/96 using the rollers in room Jolley 103 of the Mechanical Engineering building. He successfully rolled a 1 mm thick disk down to about 300 μm. Although the group has moved to different gasket materials in recent years (most notably rhenium for higher pressures) and has been able to purchase such material in sheets of the appropriate thickness 250 μm, the above information might still prove to be useful if it is necessary to roll out gaskets for any gasket material.

Once the material has been rolled out to (or purchased at) the proper starting thickness, the gasket blanks are made using a special tool that punches out 3 mm diameter disks, shown in Figure 1.6. Take the time to maximize the usage of the gasket material. Sharply pound the tool to insure a clean edge on the gasket. After punching the gasket, you will need to flatten it with the hydraulic press ($\sim$1/2 ton). Sand off any rough edges. *Make a small mark on the edge of the gasket using a scalpel. This is done so that it will be possible to orient the gasket.* The gaskets are small and easy to lose, so extras should probably be scotch taped to a sheet of paper, with a note regarding their thickness, etc. The gaskets need to be preindented before an experiment can be done. First, make sure the diamonds are aligned. Next, note the thickness of the gasket using the Tesatronic which reads in μm (take the difference of the values with and without the gasket between the pointers). Mount the gasket on the piston diamond using, for example,

Figure 1.6: Gasket punch

a small amount of vacuum grease or clay to temporarily keep it in place. Place the scalpel mark in a direction that can be remembered so the same orientation can be kept, and make sure the gasket is centered as well as possible on the diamond culet. Set the piston (with mounted gasket) on the Teflon cylinder, and CAREFULLY lower the cell body over the piston. Alternately, if you are confident the gasket will not fall off when the piston is inverted, the piston (with mounted gasket) can be carefully lowered into the body of the cell using the tool that screws into the back of the piston. Either way, take care to insure that the guide dots on the piston and body coincide (this guarantees a consistent orientation) and that the screw holes in the piston align exactly with the guide slots. (Note: usually the coil system will obscure any view of the diamonds, but if the coil system has been removed, you can view the diamonds and gasket through the holes in the sides of the cell body.) Before the diamonds press against the gasket, make sure one of the guide screws is properly in place to insure that the piston does not rotate once both diamonds are touching the gasket. GENTLY allow the diamonds to touch the gasket, and then put the other guide screw in place. Place the top plate, screw ring, and the torque wrench on the cell. (If you have lowered the cell body onto the piston, you will of course have to carefully return the cell to an upright orientation first.) To avoid any impulsive forces to the diamond, when putting on the top plate, gently lower it on the piston by putting a screwdriver in the hole of the top plate. The gasket is now ready to be pre-indented.

The final gasket will be pre-indented to a thickness which depends on the culet size being used. For a 500 μm culet, this is generally 60-90 μm. Typically, the gasket thickness should be about 1/3 of the hole diameter. There is approximately 25 μm backlash when force is applied manually. Therefore, you take the thickness of the gasket, add 25 μm, then subtract 60 μm (assuming that is the desired thickness after pre-indentation) from this value. This is how much the gasket needs to be indented. From past use, we found that 1° of manual force corresponds to indenting 3.47 μm. For example, if the gasket is 282 μm thick, then 282 + 25 (backlash) - 60 (desired thickness) = 247 μm needs to be indented, which corresponds to an angle of 71.2°. If polar graph paper is mounted on the force table, the angular displacement of the handles of the torquing tool can easily be determined by eye. The force should be increased by applying it a few degrees at a time and then

completely removing it. (Stefan once broke a diamond by applying the force all at once). This prevents the diamond from cold-welding to the gasket. When the desired angular displacement has been achieved, take the gasket out of the cell to measure its thickness; if necessary, continue this procedure (with the cell and torquing tool mounted in the same orientation as when removed) until a thickness in the desired range is achieved. When finished, note the total angular displacement (all subsequent gaskets of this material pre-indented to this angular displacement will have nearly the same thickness). Clean off the grease, or whatever else was used to keep the gasket on the piston diamond, and sand off any edges on the gasket.

Next, a hole must be drilled in the gasket. The diameter of this hole, which will contain the sample, rubies, and pressure medium, is typically 1/3 to 1/2 of the culet diameter. Presently the holes are drilled by means of a small Electrical Discharge Machining (EDM) rig right in the lab. For detailed operation instructions, consult the EDM manual. In the experience of this author (C. Looney), the EDM drilling represents a substantial improvement over previous drilling methods. (Among other things, it is easier to learn and use, results in better centered and cleaner holes, and often takes less time.) For a few years prior to the purchase of the EDM rig, the holes were drilled using a special drill in Anne Hofmeister's lab in the Department of Earth and Planetary Sciences. Prior to that (1992 - 1998) the holes were drilled on the watchmaker's lathe in our lab. So that the reader can appreciate the improvement in the drilling procedure, or in case it becomes necessary (due to a failure of the EDM rig, for example) to once again use the watchmaker's lathe for drilling gaskets, the instructions for drilling the gaskets on the watchmaker's lathe have been preserved below. After drilling (by any method), any resulting ridges around the gasket hole should be removed or smoothed out as much as possible (using a pointer, for example). However, be careful not to introduce any deep scratches on the flat surface that could keep the gasket from sealing! Thoroughly clean off any grease, glue, or whatever else may have been used to secure the gasket in whatever drilling procedure was used.

A note on thicknesses, diameters, and centering criterion: Andy performed simulations that showed that a thinner gasket allows a higher maximum pressure. To compensate for the volume loss due to the thinner gaskets, the hole diameter was increased to 330 μm. However, care should be taken in

using the thin (60 μm) gaskets, as it is possible for the final thickness to be as low as 20 μm or even less (depending on how much the diameter decreases) because of the compressibility of helium. See page 67 for more details.

A note on centering: Craig's rule of thumb was that the width of the flat ring around the hole should not vary by more than 25%. He never had a gasket fail (in a total of 5 applications of pressure), although it should be noted that he never attempted to go beyond about 70 kbar of pressure.

Drilling Gaskets with the Watchmaker's Lathe

First, a small indentation is made at the center of the diamond indentation using a sharpened pointer to guide the drill bit. Make sure that the pointer is sharp. Center this as well as possible. It is much easier to do this procedure if the 0.5x objective is used on the stereo microscope, because the working distance is then large enough to do this while observing through the microscope. It is also a good idea to superglue the gasket to an object that can be firmly held, so as to prevent slippage. If the initial mark is off-center, it can be moved slightly by placing the pointer in the hole and pushing in the direction of the center. Do not apply too much force or the pointer will break. Take the 150 μm drill bit and rotate it by hand in the indentation to see if the hole will start on center. If it does not, move the hole some more and check again. When the starting indentation is satisfactory, the gasket is mounted on the watchmaker's lathe with vacuum grease and centered as well as possible with the eye. Looking at a dentist's mirror on a stack of books through the stereo microscope, the gasket is gently moved until the drill is properly centered with the pointer mark. A small amount of super glue is applied around the edge of the gasket very gently and allowed to dry. Andy found it much easier to do this through the microscope without looking at the mirror. Allow the super glue to dry thoroughly. A smaller hole, with a diameter of 100-200 μm, is first drilled. The #10 (#6) holder of the lathe holds the 150-250 (330) μm bits. A little bit of Balzers vacuum oil placed on the bit makes the drilling easier. Only a small amount of force is necessary to drill the hole. Excessive or impulsive force will break the bit! One can easily observe when the bit goes through the gasket. The gasket should then be removed from the lathe and the hole checked for appropriate centering. If this starter hole is off center, use the pointer tool to enlarge and center it

(checking with the appropriate sized drill bit). The final hole is then drilled. If the final hole is insufficiently centered (check it under the microscope after removing it from the lathe), discard the gasket and start over. To obtain high pressures, it is of utmost importance that the final hole is centered. Note again that only a very SMALL amount of force is required for drilling both the (smaller) guide hole and the final hole.

Gold Sputtering the Gasket

For hydrostatic measurements, it is important that a thin layer of gold be sputtered onto the both sides of the gasket both before and after pre-indentation. This helps to esure that the gasket seals, trapping the helium in the sample hole. To do this, first make sure that the gasket has not scratches and that it is very clean. For gaskets that must be hardened, however, it is important that the gaskets be pre-indented BEFORE carrying out the hardening procedure. To clean both sides of the gaskets after hardening, use the Ar-etch feature on the hummer unit. Then proceed to sputter gold on both sides. In this case the gasket is not sputtered with gold before pre-indentation. To proceed sputter gold on the gasket, place the gasket in the sample chamber of the sputtering device (Anatech, Ltd. Hummer VI-A). Close the lid and turn on the power. Flush out the sample space with Argon by opening the knob with the "1" all the way counterclockwise. The sample space should seal, i.e. you should not be able to lift up the lid anymore. After it has been pumping out for about 20 minutes, the pressure should be about 20 milliTorr. Close the "1" valve and briefly open the Argon bottle to fill the line with Argon. Open the valve again and flush the sample space. Repeat several times. After the sample space has been flushed out with Argon, close valve "1" all the way and open the Argon bottle. Slowly open the valve until a pressure of 60-80 milliTorr is reached (it should be stable). Basically, you want a flow of argon in the sample chamber when you are sputtering. The settings should be on "Manual", "pulse OFF", and "plate DC". Turn on the voltage control and increase the dial until the current is 10 mA. Set the timer to about 12 minutes, and switch to "Automatic". This will ensure that the sputtering turns off after the desired time has elapsed. You should see a purple glow when the sputtering is taking place. You will probably want to keep an eye on it so that the pressure and current (via the voltage) can be regulated. After sputtering gold on one side, turn off the

device, and allow the pressure in the sample space to reach an atmosphere so that you can open the lid. Flip the gasket over and repeat the same procedure for the other side.

1.4 Using the Apparatus

1.4.1 Preparing the DAC for a Measurement

After each measurement is completed, certain procedures must be followed. It is necessary to carefully follow these procedures, because once they have been done, it is often impossible to correct mistakes without having to start completely over. It is worthwhile taking the time to do each step very carefully and not to rush anything. Make a note of any observations or values that might seem important. You cannot take too much data.

After the gasket, sample, etc. from the previous measurement have been removed, the diamonds and the clamp should be cleaned with ethanol or methanol (administered using Q-tips or Kimwipes wrapped around tweezers, for example). It might be necessary to use the sandpaper on the end of the screw tool to remove the gunk from the diamond (worry about the face of the diamonds and not so much about the sides). The sandpaper is simply attached with epoxy, so it is easy to replace when it loses its effectiveness. Make sure there is no vacuum grease remaining. If you have removed the coil system, or are installing a new one, it can be placed in the cell at this time. Whatever the case, make sure that the wires on the coil system are intact (check contacts: resistance should be about 23-24 Ohms for each coil [primary or secondary] in the present [2003] side-by-side configuration wound with 60 μm wire) and secured in place and that the solder leads will not come in contact with the screws that hold the coil system in place. More recently, the coil holder is glued to the bottom of the cell to hold it in place. This eliminates the need for screws. The primary and secondary coils should be brought out of the cell on opposite sides and then threaded through the teflon screws which will be screwed into the sides of the cell. Note that the wires from the coil should be soldered to the small circuit boards on the side of the cell using Cd solder. If lead solder is used, the coils will pick up the superconducting transition from lead at around 7K.

Before an experiment, the inside wall of the cell body (where the piston goes)

and the outside of the piston should be cleaned with acetone to remove any dirt or grease. Spray the piston with TFL spray. After allowing it to dry, wipe off any excess with a Kimwipe. After cleaning the piston, be careful not to touch it, as grease from your hands will prevent smooth movement of the piston at low temperatures. It is best to wear gloves to avoid this.

The gasket, sample, and ruby (or rubies) are now mounted by one of the following two methods.

Method 1 (Andy): Lowering Piston into the DAC body

The gasket is placed on the piston diamond in the proper orientation. To make sure the gasket is on properly, gently tap the piston: the gasket should gently rock back and forth. If it continues to move in the same direction, it is not on properly. If this problem persists, check to make sure any ridges on the bottom of the gasket have been sufficiently removed. (Such ridges are much less of a problem now that the gasket holes are drilled by EDM.) After placing the piston on the Teflon cylinder, the clamp body is gently slid onto the piston. On cell I, there are dots on the two halves of the clamp that need to be aligned (on the top of the clamp). On cell II, there are two faint arrows that should be aligned. Before the diamonds press against the gasket, put one of the screws in the clamp. Slowly lower the cell body until both diamonds touch the gasket. Turn the cell over while gently pressing the teflon cylinder. This will ensure that the diamonds remain against the gasket. Remove the piston and make sure the gasket is properly on the lower diamond.

Make sure that the upper diamond is clean, and once it is, do not touch it with anything besides the sample and ruby. Find a sample of suitable size. If it is expected that the signal will be easy to see, a sample with dimensions (70 x 70 x 20 μm) should easily avoid being squashed by the walls of any gasket starting with a hole diameter of 200 μm. If it is necessary to boost the signal size, you can try using a sample with a longest dimension of up to half the initial hole diameter and a thickness of up to 30 μm; however, be aware that by pushing the limits of the sample size you greatly increase the likelihood that the walls of the gasket will press on the sides of the sample or that the diamonds will press directly on the sample, thereby introducing an undesirable non-hydrostatic component to the pressure. In such a case

it is essential to monitor the space around the sample after every change in the pressure (take photographs with the digital camera if necessary), and to measure the gasket thickness at the end of the experiment to insure that it is greater than that of the sample. There are generally two ways of making the sample the right size: for sinters, the sample can usually be scraped and a piece the right size can be found among the shavings; for single crystals, the samples are usually very small and a portion of the sample can be cut to the needed size using a good scalpel (this should be done in a container with walls using vacuum grease because the sample is bound to fly around when cut). A scalpel should be kept and used solely for this purpose. The sample is placed on the upper diamond with a very small amount (or none if the sample will stay on the diamond) of vacuum grease to insure that it stays on. Use only a very small amount of grease or it may obscure the ruby pieces. Try to put the sample on the diamond so the sample will be in the center of the gasket hole. Sometimes there is a faint outline of the hole that remains on the upper diamond to make this easier. Ruby, typically a single piece, is then placed alongside the sample. The ruby will stick against the diamond electrostatically so grease is not necessary. Try not to get the ruby too close to the sample because it might press against the sample. One should try to make the ruby slightly taller than the sample. This is because the R_1-line is extremely sensitive to non-hydrostatic conditions (it broadens significantly, see Andy′s thesis). If non-hydrostatic conditions exist for the ruby, they might also exist for the sample. Finally, the piston (with sample and ruby mounted) is lowered into the cell body and secured against rotation.

Method 2 (Shanti): Lowering Cell Body Over the Piston

The upper (piston) diamond is cleaned, and then the gasket is attached (in the appropriate orientation) using, for example, clay or the 2-component (clear) UHU epoxy. The sample (of appropriate size, see comments above) and piece or pieces of ruby are then placed on the diamond inside the gasket hole. Shanti has never found it necessary to use vacuum grease to secure the sample or the ruby pieces. The piston is then placed on the Teflon cylinder, and the body of the DAC is carefully lowered onto the piston. Make sure the orientation dots are aligned, and screw in the guide screws well before the cell diamond reaches the gasket. Engage the safety screws to prevent the cell diamond from touching the gasket. Slowly loosen the side scrws to lower the

cell body over the piston until both diamonds touch the gasket. Turn the DAC over so that it stands upright, securely holding both top and bottom to prevent any movement during this process. The DAC is now ready for the next step.

A Note Regarding the Rubies

Originally, Stefan used multiple ruby pieces. On the other hand, Andy used a single ruby piece, with a thickness greater than that of the sample, in order to test for non-hydrostatic conditions (the R_1 ruby line should broaden noticeable if the diamond presses on it directly.) Shanti again reverted to the multiple ruby technique, with a new twist: the rubies were specially grown in a strain-free environment, and as a result, there are supposedly no variations between ruby pieces in the fluorescence spectrum. This advantage probably outweighs the advantages of the single ruby method (especially in light of the fact that it is now possible to accurately monitor changes in the gasket diameter, from which the height of the gasket can be estimated, by means of the digital camera). The small ruby spheres currently (2008) used have a 3000 ppm chromium concentration. For more information, refer to: Chevrin, Canny, and Mancinelli. High Pressure Research, 2001, Vol. 21, pp. 305-314.

A Note Regarding Non-Hydrostatic Measurements

Note that for non-hydrostatic measurements (typically with very small culets), the hole in the gasket is completely filled with sample to ensure that a large enough signal is attained.

1.4.2 Initial Procedures

Certain things need to be done at the very beginning of a measurement. These include checking contacts and pumping various lines.

First, the resistances of each pair of coils, primary (field) and secondary (pick-up) should be checked; the resistance should be about 23-24 Ohms for each pair in the present (2003) side-by-side configuration wound with 60 μm Cu wire. Next, make sure that there is no electrical contact between the field and pickup coils and that both coils are electrically isolated from the body of the cell.

Once the sample has been properly mounted and the piston has been inserted, the membrane is placed underneath the top plate and gently lowered onto the piston. Put a small amount of molykote on the bottom side of the membrane. Also put a small amount on the underside of the retaining ring. Do NOT put any between the membrane and the top plate. It used to be important to carefully orient the diaphragm line so that it could pass through the holes in the support and the baffles. (Andy took special care to align the diaphragm line with the hole for primary coil leads, which were always brought out through the same hole.) Now, however, the orientation is not so critical, since slots have been cut into the baffles and (more importantly) into the support plate. Nevertheless, take care not to stress the diaphragm capillary line, especially near the membrane. Make sure the protection screws are slightly loosened (turned counter-clockwise) to insure that the piston can move down. These screws are on the clamp to insure that the diamonds will not touch each other if there is a gasket failure, but many students have not used them (see below). These screws also help to protect the coil in the case of diamond failure.

With the torquing tool in place, torque the tightening ring until it no longer turns easily by hand (using the knurled rim, not the bars.) From this point, the piston is backed off to create an $\sim$30 μm gap between the diamond and the gasket, which will insure that helium can easily enter the cell during the cryogenic loading procedure. Since a 1° rotation of the torquing tool corresponds to 3.47 μm of piston motion, the torquing tool needs to be backed off by about 8.6° to give the desired gap. This corresponds to a distance of about $\sim$0.45 cm on the outer rim of the DAC. This procedure used to be done under the metallurgical microscope to determine when the diamonds had sufficiently pressed the gasket, but it has been found that working "by feel" (as described above) works just as well, and it greatly reduces the risk of damaging the diaphragm capillary, which had to be threaded through the microscope support. Note that special care has to be taken if an air sensitive sample is being measured: backing off the piston by too much may allow the sample to oxidize. It will be necessary to get the cell into the cryostat and flushed with He as quickly as possible.

If you wish to use the safety screws, here is the procedure (modified from the original procedure somewhat due to the fact that the metallurgical microscope is no longer used). Just before backing off with the torquing tool to

create the gap, screw in the safety screws as far as they will go with "gentle" torquing. (Don't crank them down, or you will push the diamonds apart slightly and thereby introduce errors into the screw adjustment. Just go as far as you can using, say, only your thumb and middle finger on the screwdriver.) At this point the screws will prevent the diamonds from coming any closer together, because the motion of the piston is inhibited. The thread on the side screws is 4-48, so 1° of the safety screw rotation corresponds to 1.47 μm of piston travel. Keeping this in mind, you can figure out how much to back off the screws to prevent the diamonds from touching each other in the event of a gasket failure without limiting the ultimate pressure of the experiment. Previous editions of this manual have suggested backing the protection screws off by 1/8 of a turn, which would allow 66 μm of piston travel. This is a bit simplistic: if the (pre-indented) thickness of the gasket is (say) 50 μm, the diamonds can still touch in the event of gasket failure; on the other hand, if the (pre-indented) thickness of the gasket is 90 μm, the screws will prevent further motion of the piston when the gasket has been compressed to a thickness of about 24 μm. This could bring a premature end to the experiment, since the final gasket thickness may reach less than 20 μm. (The final gasket thickness depends on numerous factors including original thickness, original diameter of hole, ultimate pressure, and how much pressure fluid escapes during the initial pressure application). Perhaps the best advice is to calculate the number of degrees the screws must be backed off to give 10 μm of clearance in the case of gasket failure. You will of course need to accurately know the gasket thickness (which you should already have measured and recorded) in order to do this. Note that even with this criterion, a 20 μm error either way could either allow the diamonds to touch (in the event of gasket failure) or bring a premature end to the experiment by limiting the ultimate pressure. That is why this author (C. Looney) always made sure they were well out of the way. It is also likely that (even if properly set) the safety screws will not prevent damage to the diamonds in the event of a typical gasket failure involving a "side-blowout"; that is, the safety screws give no protection from lateral forces.

It is now time to screw the DAC onto the cryostat insert. Push the fiber optic cable up so it will not interfere with the DAC. The DAC should be oriented so that the hole where the pickup coil comes out of the DAC is nearest the radial connectors on the insert. Previously, there were three thermometers

in the DAC: an Rh-Fe resistor near the cryostat heating element, and Pt and Ge resistors mounted in a missile-shaped holder that screws into the back of the piston. (The fiber optic cable also goes into this holder.) The Pt thermometer was never calibrated but used the standard Pt-curve. The Ge sensor (N2G) was recalibrated in 1995 by Craig Looney. The original manufacturer's calibration from the 1970s was found to have a couple of calibration points with errors on the order of tenths of Kelvins. [Note: Any such errors, in any thermometer calibration, can cause a kink in the DAC susceptibility data that can be (and has been!) mistaken for the signal from the sample.] Calibration charts and circuit diagrams for the DAC thermometers should be kept handy in the DAC folder. The N2G_low calibration was used for the Germanium resistor. In August 2011, the Rh-Fe resistor was also replaced with an Allen-Bradley carbon resistor that has a room temperature resistance of $\sim$50 Ω. The Pt and Ge thermometers were also repaced with a single Cernox thermometer that can be used over the entire temperature range during measurements. This thermometer is placed in the same holder that previously held the Pt and Ge resistors. Be careful when screwing the thermometer holder into the clamp. Andy liked to hold the clamp upside down (before attaching it to the cryostat insert) to screw in the thermometer holder in order to minimize torques on the thermometer wires. Check again to be sure that there are no ground contacts in the thermometry. Slowly lower the fiber optic cable into the hole in the thermometer holder. The male thermometer connection on the holder is then attached to the female counterpart on the insert. Thermometer contacts should then be checked again.

After the cell has been attached to the cryostat insert, the capillary line should be connected (tight enough to prevent a leak, but not so tight as to damage the connection and put you out of business for a week or more), and everything placed in the cryostat, make a final check of all the contacts. Also, make sure that the cell (both sample and ruby) can be seen clearly through the eyepiece when illuminated via the fiber-optic cable. If the diamonds are dirty or there is too much grease, the sample and/or ruby may be obscured. If this is the case, it is better to remount the sample after thoroughly cleaning the diamonds. (You should really should have taken care of this earlier, but if not, it is better to do it now than to start an experiment and not be able to measure the pressure.) Make sure the ruby fluorescence line is visible at

room temperature and of a sufficient magnitude to be useful.

There are two valves on the He gas membrane system. One of the valves opens the He to the membrane and the other opens the system to the outside to remove pressure from the membrane. The diaphragm line should be pumped on for at least 15 minutes with P $\sim 10^{-2}$ torr on the Pirani gauge on the turbo pump. The valves may need to be opened slowly for the pumping to work properly. It is a good idea to flush the membrane at least once with one bar of pressure. (Note that the zero of the analog diaphragm pressure gauge has a zero of -2 bar.) As of 2009, we have a new digital gauge for the membrane. With an excitation voltage of approximately 20 V, the membrane pressure (in bars) is given by

$$P = 1 + 20.59(V - 1.002)$$

where V is the voltage output of the gauge.

1.4.3 Cooling Down

An Oxford "flow" cryostat, shown in Figures 1.7 and 1.8 is used to cool the DAC down. First, the cryostat insulation (aka OVC or "outer vacuum chamber") should be pumped down using a turbo pump or diffusion pump. In the meantime, the transfer tube is placed in the liquid He dewar. With the current transfer tube from Cryo Industies of America (seen in Figure **??**), the cold valve should be opened 1/2 a turn before inserting the transfer tube into the dewar. This is to allow helium to flow through the transfer tube and prevent it from getting clogged. The transfer tube should be lowered into the dewar slowly ($\sim$5 min) so that the pressure in the dewar remains less than 1 psi. Make sure that the transfer line is clean and dry. Make sure that the transfer tube is at least half an inch off the bottom of the dewar so that it doesn't freeze to the bottom. After the transfer tube has been inserted, the cold valve may be closed. Always note the value on the more sensetive of the two He gas meters and the He level in the dewar before and after an experiment. Using the fact that 27 ft^3 of He gas corresponds to 1 liter of liquid, keep track of the He recovery rate (it is usually in the 80 % range). It takes 1.5-2 hours to cool from room temperature to helium temperatures. These values were obtained using the maximum vacuum reading possible without causing fluctuations in the VC 30 vacuum gauge needle. The actual cooling rate can be adjusted by supplying less

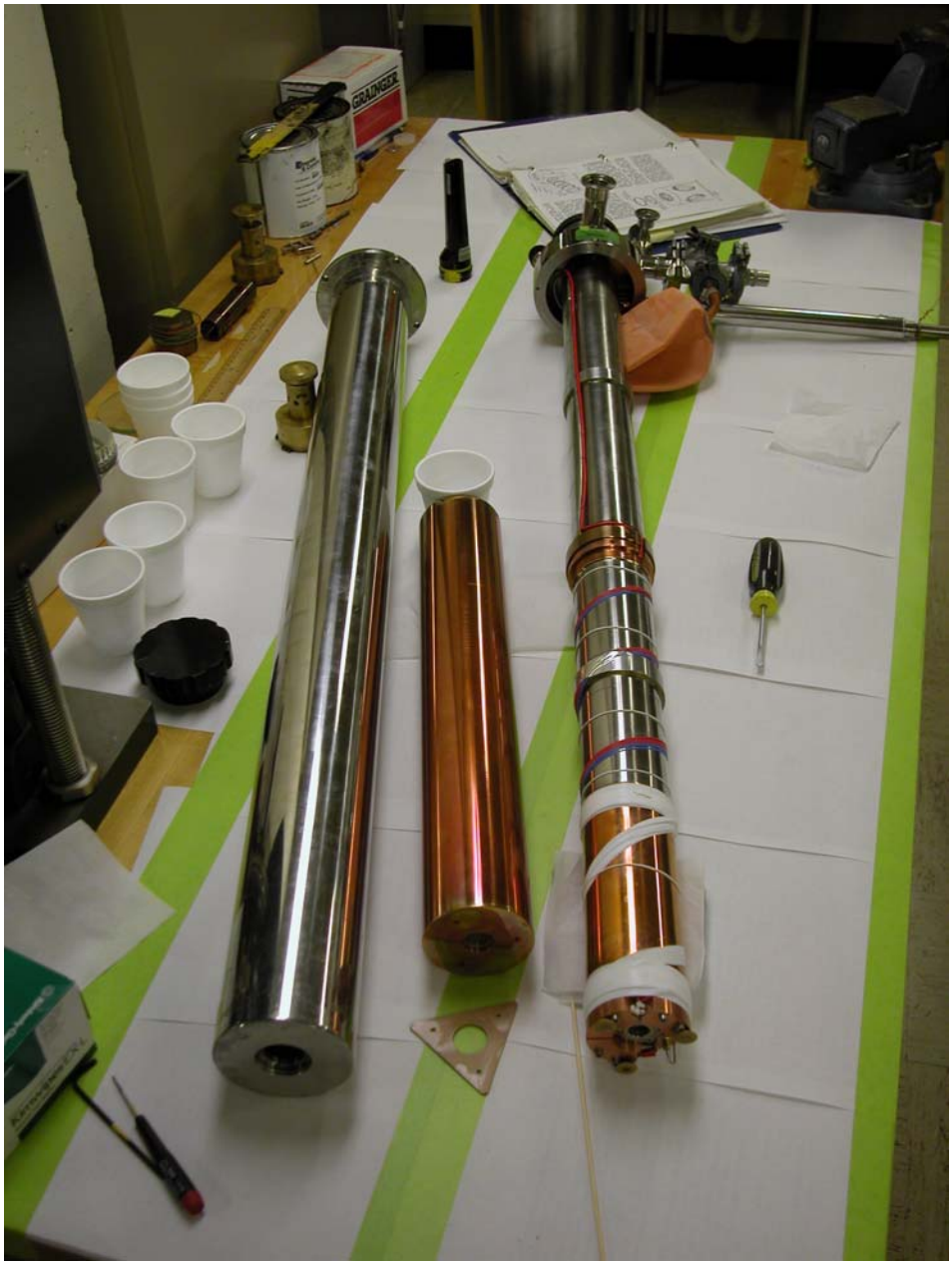

Figure 1.7: Oxford "flow" cryostat. (left) outer case with quartz window; (middle) heat shield with quartz window; (right) sample tube with sapphire window and copper gasket seal. Heater and Rh-Fe thermometer are in holes in two of the three copper blocks on the bottom of the sample tube.

Figure 1.8: Oxford "flow" cryostat. (left) outer case with quartz window; (middle) heat shield with quartz window; (right) sample tube with sapphire window and copper gasket seal. Heater and Rh-Fe thermometer are in holes in two of the three copper blocks on the bottom of the sample tube.

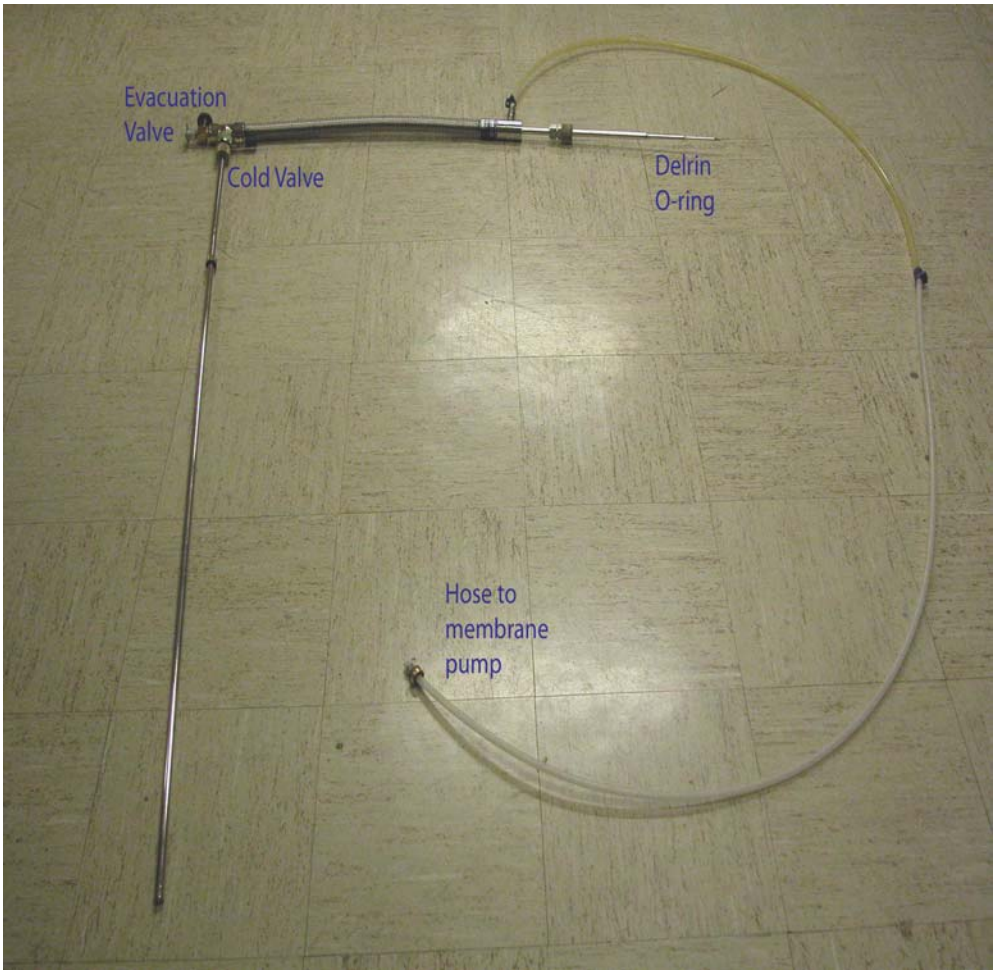

Figure 1.9: New liquid He tranfer tube from Cryo Industries of America.

underpressure with the membrane pump or by adjusting the needle valve on the stem of the transfer tube shown in **??**.

Original Cooldown Procedure: (old transfer tube)

In the "original" cooldown procedure (as described in the Oxford cryostat manual), the old transfer tube, seen in Figure 1.10, is inserted all the way into the cryostat arm but the connector is only tightened a couple of turns (it is NOT tightened down). This will allow the helium to escape to the recovery line without going into the cryostat, so that the transfer tube can be rapidly and efficiently cooled when the membrane pump is turned on. (Consult the cryostat manual, a copy of which can be found in the blue binder, for details on the path taken by the helium.) When a sufficient vacuum has been attained, open the transfer line valve on the wall and turn on (that is, plug in) the He (membrane) pump when P$\sim$5 x 10^{-2} torr. The He needle on the flow control box will read about -0.7 bar (on the outside scale) until He starts to go through the transfer line. When the reading goes down, the noise that the He pump makes will change due to He coming through the line and going through the plastic tubing. When this happens, the transfer tube connection to the cryostat should be tightened firmly by hand to make the He go into the cryostat. The tube should be closed but not too firmly. Only a Teflon seal is used and it could be damaged if the tube is closed too tightly. *Note: the small Teflon piece marked in Fig. 1.10 sometimes comes off (when the transfer tube is pulled out of the cryostat) and needs to be removed from the arm of the cryostat.* Continue pumping on cryostat with the turbo pump until the cryostat begins to cool. Close the valve to the cryostat and remove the turbo pump. As the temperature decreases, the transfer line should be tightened as the Teflon contracts. If this is not done, the arm to the cryostat will excessively frost up. Note that when the cooldown is started from lower temperatures (<70 K), the system will generally warm up a bit (as much as 30 K if the cooldown is begun at 20 K) before cooling begins. When you have cooled the system to the desired temperature, unplug the pump and make sure that the helium gas can escape from the sample tube to the recovery line. At the time of this writing (July 2003), the gas can escape through an internal (not visible from outside) relief valve, installed so that the "Alternate Cooldown Procedures" described below could be used. Previously, it was necessary to open a valve by hand to connect the sample

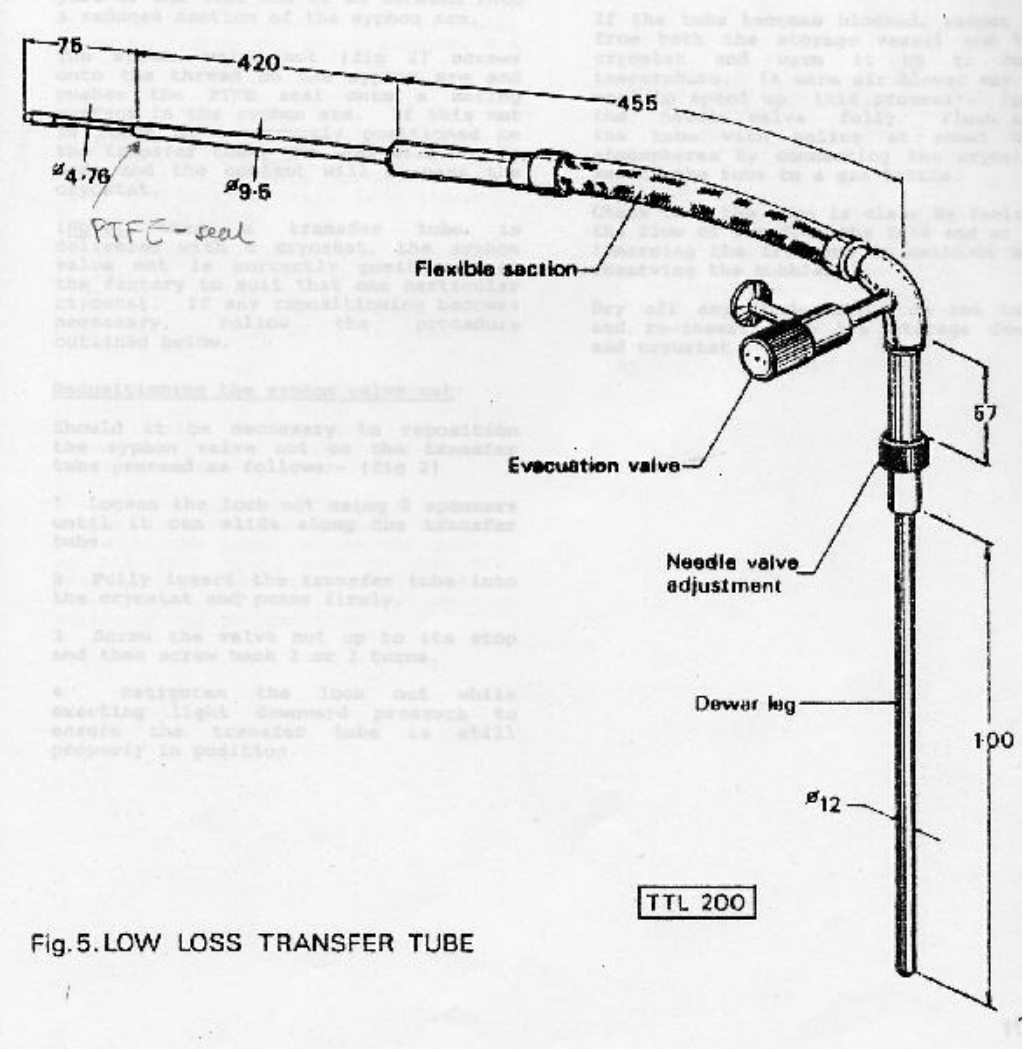

Figure 1.10: The old liquid He transfer tube from Oxford

tube to the Helium recovery line.

To make the system more self-contained, Andy Cornelius performed tests to see if a rotary pump was sufficient for pumping on the OVC. The pump was allowed to run for a minute or so with the valve closed. The valve was then opened and allowed to run for 5 minutes. The same procedure as used for the turbo pump was then used. To use the rotary pump to cool down, simply follow this procedure. Open the ball valve to pump on the OVC line. Make sure that the ball valve for the cryostat is closed. This is important because the OVC line is used to pump on vacuum, while the cryostat line will be pumping on He. Do not allow the vacuum line to become contaminated with He. Always make sure that vacuum parts are labelled vacuum or He and never use one marked He to pump on a vacuum.

Alternate Cooldown Procedure: (old transfer tube)

This cooldown procedure (ca. 2003) has been devised to prevent potential problems associated with a small leak in the membrane pump that could result in blockages due to frozen air (presumably the water vapor component) during cooldowns that begin at lower temperatures. In this procedure, the cryostat insulation (OVC) and the diaphragm capillary are pumped down as before. The flow valve on the VC30 flow control box is completely closed and the sample tube is evacuated and backfilled to slight over pressure with Helium gas, so that no air gets in when the He can is connected. The transfer tube (already fully inserted into the He can) is then inserted into the cryostat arm and (here is a major difference!) the connection is fully tightened. The cooldown is then initiated by pumping on the sample chamber with the large rotary pump; this pulls helium out the dewar through the transfer tube and into and through the cryostat. When the temperature starts to go down (the Rh-Fe thermometer in the heating element will react before those in the DAC), the rotary pump is valved off and shut down, the membrane pump is turned on (by plugging it in), and the flow valve on the VC30 box is opened (in that order). [If the VC30 valve is opened before turning on the membrane pump, air could leak into the system.] As the system cools, you should check to make sure that the transfer tube connection is still tight (it generally needs to be tightened eventually). When you have cooled down to the desired temperature, close the VC30 valve and then unplug the membrane

pump.

New Cooldown Procedure: (new transfer tube)

A new transfer tube (see in Figure **??**) was purchased from in early 2005 from Cryo Industries of America. This transfer tube has less problems with sealing, and is superior in that there is more pre-cooling of the Helium gas. The cooldown procedure is completed as follows. Place the delrin o-ring on the end of the transfer tube arm. Make sure that the wheels on the dewar are aligned so that it rolls in a straight line, making it easy to insert the transfer tube into the cryostat arm. At this point, the (already flushed) sample space should be filled with helium, and the balloon will be inflated. Right before inserting the transfer tube into the arm of the cryostat, plug in the membrane pump and open the flow valve for the membrane pump by turning the knob on the VC30 flow control box counter-clockwise all the way. You do not want to pump on air for longer than absolutely necessary. If you put your finger near the hole on the arm of the transfer tube, you will be able to feel that it is pumping. Remove the rubber stopper from the cryostat arm, and quickly insert the transfer tube. To prevent air from getting into the sample space, have somebody gently squeeze the balloon so that there is a flow of helium out of the sample space. If noone is available to help, it is possible to slightly open the helium bottle (that was used to flush out the sample space) to obtain a flow of helium out of the sample space when the stopper is removed. The transfer tube should be inserted all the way into the cryostat arm. Make sure that it goes in properly. Tighten the connection between the cryostat and transfer tube. Once the transfer tube is in the arm of the cryostat, the balloon will rapidly deflate, since the sample space is now being pumped on by the membrane pump. If you had the helium gas bottle open, close it now. Close the black valve at the top of the cryostat that connects the sample space to the line from the helium bottle in order to prevent leaks and because the rubber hose line is very dirty. Open the cold valve on the transfer tube 4 turns to allow cold helium gas to flow into the sample space. After $\sim$3 minutes, you should see the indicator on the membrane pump flow gauge start bouncing up and down. After $\sim$12 minutes, it should stabilize at a rate of $\sim$1.5-2 L/hr. At this point, the cell should start cooling down. Note that if cooldown is begun at lower temperatures (say less than $\sim$70 K), the cryostat will generally warm

up a bit (perhaps as much as 20-25 K) before it starts cooling. As the cell cools, and the teflon o-ring contracts, periodically check that the connection between the transfer tube and cryostat is tight so that no air can get into the sample space. If it takes longer to start cooling, or seems to be cooling more slowly than normal, it is possible that the transfer tube is clogged. In this case, it will be necessary to turn off the membrane pump, disconnect the transfer tube from the cryostat, and then romove it from the dewar. It should be allowed to reach room temperature to remove any clogs. It is possible to speed up this process by blowing COOL air from the hot air gun on the transfer tube. Do NOT use hot air, as it could melt some of the rubber parts in the transfer tube.

Using LN_2 as Cryogenic Liquid

If the transition being measured occurs at high enough temperatures, liquid nitrogen (LN_2) can be used as the cryogenic liquid. The same general procedures used for liquid He are used for LN_2. A few differences do exist. First, the turbo pump or a diffusion pump must be used to pump on the OVC (LN_2 does not cryopump as well as He). Also, the transfer tube needs to be pumped on weekly. This is done by pumping on the evacuation valve shown in Fig. 1.10. The evacuation valve is removable and is kept with the DAC parts. If using He, the transfer tube needs to be pumped every six months or so. The transfer tube is then placed in the LN_2 dewar and everything proceeds in a similar manner to using He.

1.4.4 Controlling Temperature

Sophisticated temperature control is generally not necessary for routine measurements. The cooling rate can be reduced or increased by adjusting the vacuum at the VC30 gauge. For slow cooling (during the measurement of magnetic susceptibility) the vacuum should be reduced so that the needle lines up with the black pen mark. For warming measurements, turn off the membrane pump (pull the plug) at a temperature 10-30 degrees below the temperature of the expected transition and open the system to the helium recovery line.

Measurements below 4 K (both warming and cooling) are made by first loading several inches of LHe into the cryostat (see Section 1.4.6) and then

pumping on the sample space to reduce the pressure, and hence the temperature. To take measurements while cooling, turn on the rotary pump and very slowly open the two-stage valve to bring the temperature down slowly. Keep an eye on the temperature while you open this valve. It will probably take a bit of practice to get used to doing this so that you have good control over the temperature. Using the rotary pump, temperatures as low as $\sim$1.55 K can be reached in this way. To reach lower temperatures (as low as $\sim$1.3 K), it is necessary to turn on the large roots pump. This should be turned on at $\sim$1.7-1.8 K. After it has been running for about a minute, open the ball valve betweeen the pump and the cryostat. Be sure to hook up an exhaust hose (we generally connect a additional blue hose to the hose which is usually connected to the pump for the glove box. For slow, controlled warming measurements in this temperature range, slowly close the two-stage vavle while monitoring the temperature. By $\sim$2 K (or at least before the λ-point of LHe at 2.19K), the valve should be completely closed. At this point, you may close the ball valve to the roots pump (if it was being used) and turn it off. Be sure to immediately vent the roots pump after turning it off - if you don't, you will hear it start to make noises. The temperature will continue to increase naturally as LHe gradually boils off and the pressure increases. Make as many cooling and warming measurements as desired, or until you run out of LHe in the cryostat. It is important that the LHe be above the top of the cell for measurements at low temperature so that there is good thermal coupling between the sample and the thermometer.

Currently (2011), most measurements above 4K are performed while warming naturally, i.e. by turning off the membrane pump and allowing the temperature to drift upwards through the transition. For temperatures $\sim$3.5-5 K, however, any LHe that is boiling off can cause anomalies in susceptibility measurements which could be mistaken for transitions. One way to avoid this is to perform slow cooling measurements by using simultaneous heating and cooling. Heating is provided by applying a current to a heater in the cryostat (40 V, maximum). A power supply is connectedt o the current ramping device designed by James Hamlin (see Section 6). Adjust the cold valve (open $\sim \frac{1}{2} - 1$ turn) so that the temperature is stable, at say 10 K, when a set current is applied. As the current is decreased using the current ramping device, the temperature will slowly decrease. For slow cooling measurements in the temperature range 3.5 - 10 K, it has been found that using

setting $I_{\max} = 0.27$ A (V = 5.7) and using rate #8 on the current ramping device gives good slow cooling measurements (the current will decrease at ~ 5.4 mA/min).

In some cases, it might be necessary to hold the system at an exact temperature. Rough control can be obtained by reducing the underpressure (at the VC30 gauge) and/or by throttling the transfer tube needle valve. For stability, however, it is necessary to have the potential for both heating and cooling. The Oxford ITC4 (or some other temperature controller) can be hooked up for this purpose. Previously, the temperature sensor monitored by the temperature controller was a Rhodium-Iron resistor, which was very closely coupled to the cryostat and to the heater. Note that the thermometers in the DAC are not closely coupled to the heater and thus should NOT be used for temperature control. The temperature controller applies power to the heater when necessary to maintain a constant temperature. The temperature controller can also be used to "sweep" over a temperature range at a programed rate by steadily increasing the heater power. In 2011, the Rh-Fe resistor was replaced with an Allen-Bradley carbon resistor. The ITC4 temperature controller does not contain the calibration for this resistor, so it cannot easily be used for temperature control.

The temperature controller or a manual heater can be used to rapidely warm the system to room temperature. *However, it is crucial that the Allen-Bradley resistor (previously Rh-Fe) be used for monitoring the temperature.* (Since the cell thermometers can lag the Allen-Bradley (Rh-Fe) thermometer by 50 K or more when heaters are applied, using them to monitor the temperature during rapid warm-up can result in serious overheating of cryostat components.) Before using the ITC4 (or any other temperature controller) with the system, be sure to read the manuals for both the temperature controller *and* the cryostat.

1.4.5 Preparing the Cell for Loading with Liquid He

To use liquid He as pressure medium in a hydrostatic pressure experiment, it is necessary to successfully bring superfluid He into the sample chamber of the gold-plated metal gasket. To achieve this, the following procedure should be followed once the sample and ruby spheres have been properly loaded inside the gasket hole onto the culet of the diamond anvil on the

piston. The body of the DAC should be carefully lowered onto the piston which is propped up by a Teflon cylinder. Rotate the two small, vertical safety screws so as to prevent the diamond anvil in the DAC body from touching the gasket. Slowly loosen the safety screws to lower the DAC body over the piston until both diamond anvils just touch the gasket as viewed under the stereo microscope. Do not loosen further! Now turn the DAC body upside down, insert the membrane with capillary, the top plate, and retaining ring. Rotate the retaining ring until it is finger tight. The safety screws will keep the anvils from pressing into the gasket, thus possibly preventing the liquid He from entering the sample space. This is an important procedure to ensure that liquid He can flow into the sample space when the entire DAC is submerged in liquid He in the Oxford flow cryostat.

After the cell has been attached to the cryostat insert, make sure the protection screws are slightly loosened (0.13 mm corresponds to 1/4-turn of screw) so that the opposing anvils can push into the gasket and generate very high pressures. In case one diamond anvil fails, the safety screws will prevent or reduce possible damage to the other anvil and/or the coil system (This precedure was modified March 2014 by N. Forouzani).

1.4.6 Loading the Cell with Liquid He

Liquid He is used as a pressure medium in the DAC. He is the best possible pressure medium to ensure a hydrostatic or nearly hydrostatic environment. The liquid is allowed to accumulate in the cryostat where it is trapped in the cell by applying pressure to the membrane.

The cell is cooled as normal. Make sure the transfer tube is kept tightened. Use the Cernox resistor (or whatever low temperature sensor happens to be installed) for the low temperatures, with the appropriate excitation current (consult thermometer calibration chart). The membrane pump should give a sufficient under-pressure in the cryostat to allow liquid to accumulate. If no liquid is accumulating or if it is very slow, it is possible to use the rotary pump to pump directly on the cryostat (this is the method previously used regularly). This is not ideal, however. The preferred method (if necessary) is to add a slight overpressure ($\sim$2 psi) to the dewar. Using only the membrane pump is best, as the vacuum in the cryostat can be read directly from the VC30 gauge and a slight drop in the height of the flow rate indicator signals

that liquid has started to accumulate. The sample should become blurred once He starts to accumulate in the cell. At this point, continue to pump at the same level, adjusting the flow control valve if necessary, until a sufficient amount of He has accumulated; generally this takes about 20 minutes, but it can take longer. It is best to keep an eye on the helium recovery line while loading LHe. Originally, it was necessary to pump for half an hour and "hope for the best", but during Shanti Deemyad's tenure on the apparatus, a He level meter was installed. As LHe starts to accumulate above the top of the cell, the voltage across the level meter will decrease as the resistance decreases. Using an excitation current of 70-75 mA, calculate the change in the resistance in order to determine the LHe level. The resistance decreases by 11.6 Ω/in. 5-6 inches of LHe should be sufficient. Do not leave the current on the level meter on for longer than necessary, and be careful not to turn it on when the sample space is under vacuum, as this can damage the sensor. Turning it on while pumping on the sample space is alright as long as LHe is present. For more information, read the manual that came with the level meter. Note that, with an excitation current of 75 mA, the voltage is approximately 10.8 V when no LHe is present. Once the desired amount of He has accumulated in the cryostat, the temperature is lowered by pumping on the helium until the temperature is below the λ point (2.19 K at 0.0508 atm and 1.8 K at 30 atm) where He becomes superfluid and will easily flow into the sample chamber. In preparation for this, the VC30 valve is closed, the membrane pump is shut off, and the rotary pump is valved off if it was being used. (The rotary pump may be left on, because it will soon be used again.) Let a slight overpressure build up (or hasten the process by supplying helium gas), and then remove the transfer tube rapidly and steadily. Don't stop or the tip is likely to freeze in the arm from the cryostat. (It is highly recommended that you get somebody to help you move the helium can so that you can focus on removing the transfer tube.) Place the appropriate plug in the arm of the cryostat to seal it off. If the transfer tube will not be used again for cooling that day, remove it from the dewar. Pump on the cryostat again by opening the 2-stage valve from the roatary pump. Pump on the He for about 10 min while watching the temperature. When the He becomes superfluid, it will be possible to see the sample clearly.

Once the He is superfluid, slowly open the valve to let the He gas enter the membrane (do so slowly because room temperature gas is being allowed to

enter a region at 1.7 K). Apply the appropriate membrane pressure one bar at a time while monitoring the gasket. [Roughly 10 bars for TaW and 16-17 bars for Rhenium]. While superfluid He is flowing into the sample space, you may see some movement of the sample. As pressure is being applied, the hole size should shrink somewhat. When the hole size stabilizes and the sample stops moving, the gasket should be sealed. Take pictures with the digital camera every few bars or so to record the changes in the gasket hole. Monitor the pressure in the sample space using ruby fluorescence (see 2.2). Once the sample space is sealed, the pressure should start to increase. After you are done applying pressure, valve off and shut off the rotary pump, and make sure the He has a path to the recovery line. The system will take a while to warm up with all the liquid He that needs to boil off. It is probably not a good idea to use the heater to warm up the system while there is still liquid He in the cryostat because there could be a large pressure buildup.

1.4.7 Changing Pressure

The pressure in the DAC is increased by *carefully* opening the valve between the gas bottle and the diaphragm capillary. One bar on the capillary gauge will correspond to different pressures in the DAC depending on the culet size. You can estimate the pressure by taking a ratio of the membrane area to the culet area. While performing an experiment, it is always always a good idea to keep a plot of membrane vs. sample pressure, as this can give and early indication of problems. Figure 1.11 shows a plot of typical membrane vs sample pressure for various culet sizes. It is vital that the pressure always be increased at temperatures above the melting curve of the helium in the membrane.

At the end of an experiment, the pressure is released slowly by opening the venting valve. Andy typically released the pressure at room temperature at a rate of 1 bar of diaphragm pressure every 15-30 minutes. However, in certain circumstances it may be desirable to release the pressure at low temperatures. Craig has released pressure at 6K at the rate of 1 bar of diaphragm pressure every 10 minutes. *When releasing pressure at low temperatures, be sure that the temperature is high enough that the helium in the diaphragm line is not frozen.*

It is possible to partially release the pressure by releasing some, but not all,

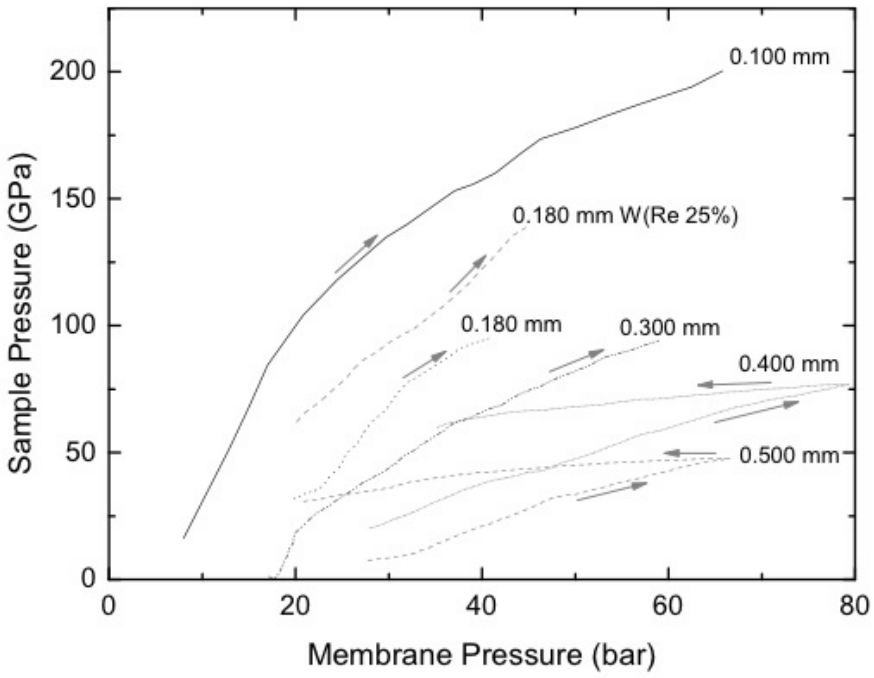

Figure 1.11: Plot of membrane vs. sample pressure for various culet sizes. Data in each case, excluding that marked W(Re25%) was taken with Re gaskets. The 0.180 mm culets are beveled from 0.350 mm, and the 0.100 mm are beveled from 0.300 mm. Taken partially from James' Hamiln's thesis.

of the pressure in the diaphragm line. There will very likely be no pressure change in the DAC until a couple of diaphragm bars have been released. Note that releasing pressure in this manner requires the escape of some of the pressure fluid from the gasket bore. Thus, there will be less space for the sample/ruby for a given pressure.

2. Measurements

2.1 Measuring Susceptibility

A Stanford Research SR830 Lock-In Amplifier is used in conjunction with the coil system (field and pick-up coils) and the measurement program to measure AC susceptibility as a function of temperature. A sinusoidal alternating current is supplied to the primary coil by the SR830's internal oscillator (via the "SINE OUT" located right below the big knob), or by a high quality external oscillator or function generator. If an external source is used, it needs to be connected to the "REF IN" terminal (which is next to the "SINE OUT" terminal), so that the SR830 can phase-lock to it. The secondary (pick-up) coils are connected to the inputs (terminal A or B or both, at the bottom left of the front panel). As of this writing (2003), the secondary coil output is actually fed to a preamplifier (SR554), and the output of the preamplifier is connected to terminal A. However, there are many ways to use the lock-in amplifier, and the setup may be modified several times before the next revision of this manual. [Just the other day (7/25/03) a signal subtracter was removed from the measurement circuit because it was found to attenuate the sample signal and cause temperature dependent phase shifts. If I had included THAT device in this description, it would already be out of date...] The best way to arm yourself is to dive into the lock-in amplifier manual and familiarize yourself with the lock-in's specifications and principles of operation. In particular, you should read the section of the SR830 manual entitled "What is a Lock-in Amplifier?" (See also section 7.1 in Andy's Cornelius's dissertation for discussion of the theory of a lock-in amplifier.) You probably won't understand everything at first, but you should periodically return for more study until you understand why the system is set up the way it is. At that point, you will be able to make improvements if necessary.

Without being overly specific, here is a general outline of how to make a measurement. First, you need to set the oscillator voltage (internal or external) to generate the appropriate magnetic field with the coil system. Actually,

what is really important is the CURRENT in the primary coil, but generally it is the VOLTAGE that is adjustable. An external resistor is generally placed in series with primary coil to reduce the temperature dependance of the current. You should measure the current that a given voltage produces at the desired measurement current, and you should check to see that it agrees with what you would have calculated based on the impedances in the circuit. Don't forget to include the output impedance of the source. Note: the current might be frequency dependent because inductive impedances are frequency dependent; whether or not frequency makes a big difference depends on the inductance of the coil ... calculate it and see! In the present configuration (2011), with a frequency of 1023 Hz, the inductance is not important, but the coil system might be changed substantially before the next revision of this manual. Of course, you will also need to know B as a function of I for the primary coil (which can be calculated using the short solenoid formula from the number of turns and the diameter of each layer), as well as the impedance of the circuit. Thus, with a knowledge of the actual current, and B as a function of I, you can calculate the current that gives you the desired magnetic field, and you can then adjust the source voltage (of whatever oscillator is driving the coil) to give you the desired current. [Note: be sure to include an external resistor in series with the primary coil that has a substantially larger resistance than that of the primary coil; otherwise the temperature dependence of the primary coil resistance will cause a large temperature dependence in the applied magnetic field.]

Next, set the frequency of the oscillator (SR830 or external, whichever you are using) to the desired value. Currently (2011) a ferequency of 1023 Hz is used for ac susceptibility. Choose an appropriate measurement range to give you the maximum sensitivity without the risk of going off-scale (overload will not damage the lock-in but it will ruin your measurement), and set the time constant to a value large enough to smooth out the bulk of the noise but small enough to keep from compromising the response of the lock-in to real changes in the signal. (Ranges between 1 sec and 10 sec have typically been used, for example.) In the region of interest, you may have to use the offset and expand functions of thelock-in amplifier to avoid digitization of the signal. See the lock-in manual for an explanation of how this works. Make sure the appropriate thermometer is selected for the temperature range in which the measurement will be made. Then start up the measurement

program (set it to take data every [say] 0.001 K in the temperature range of interest), cool down or terminate your cooldown (whichever is appropriate), and you are in business. To date, the best measurements have been made by warming "naturally" through the transition, no cooling or heating. You can speed things up by ramping with the temperature controller. You can also cool slowly through the transition by adjusting the flow control valve on the VC30 box. [There is substantially more noise during cooling, perhaps from the pump or maybe from the turbulence of the flowing helium, but if your signal is relatively large you still might be able to get a ballpark measurement that will help you locate the transition.]

Before starting the measurement, you should also set the "phase" (on the SR830) so that a ferrite sample (with only a "real" susceptibility part) placed in the coil system changes the voltage in only ONE channel of the lock-in (x or y, but not both). If the phase of your coil system has an appreciable temperature dependence, you will need to account for this. Note that you only need to do the ferrite test when you are installing a new coil system (or if you suspect that you have measured it incorrectly before, or if you have added a new component to the pick-up circuit, like a preamplifier or signal subtracter).

The measurement program currently being used was written in LabVIEW by Mathiewos Debessai. Previously, a measurement program written in QuickBasic by Jost Diederichs (with some assistance from Craig Looney) in 1992-93 was used. A description of how to use each of these programs may be found in the "Computer Programs" manual.

After an experiment, the raw data includes background from the clamp and the gasket that needs to removed to clearly see the desired transition. A description of the various background components is given in Andy's thesis. Andy used to fit a polynomial (usually quadratic or cubic) to the data, and then subtract it from the data, in the hope that only the signal from the sample's transition would remain. However, anything beyond a 1st order (linear) fit can introduce wiggles that can easily be mistaken for a transition. It is probably better to subtract a straight line from different regions of the measurement until the signal is found. (In Origin, one can perform a linear fit to a selected portion of the data, and then subtract this curvefit from the data.) Another method (developed by Shanti Deemyad that can work very

well is to subtract the data from two data sets obtained under similar measurement conditions (field, frequency, rate of warmup, etc.). The background contributions will largely cancel, leaving the two transitions (one from each measurement). This is fairly easy to do using the Origin plotting program, which has a built in algorithm for interpolating between data points. When practical, this method is perhaps the best of all.

2.2 Measuring Pressure using Ruby Fluorescence

(This section was modified December 2001 by J.S. Schilling and September 2011 by N. Hillier)

2.2.1 Ruby Fluorescence

At ambient pressure and temperature the R_1 and R_2 fluorescence lines of ruby are located at 6942.4 Å (Ångstrom) and 6929 Å, respectively (in both Stefan's and Andy's theses the order is reversed!). Although the R_2 line is comparable in size to the R_1 line at ambient temperature, at helium temperatures the R_2 line is very weak. Under pressure both lines shift initially at the rate of +3.646 Å/GPa to longer wavelengths. This pressure dependence remains constant to liquid helium temperatures, even though the lines themselves shift by approximately 8 Å to shorter wavelengths as the temperature is decreased from 300 K to 100 K; below 100 K there is little temperature shift. There are many references dealing with the nearly linear shift of R_1 with pressure (see Section 6.2 of Andy's thesis). One is excellent reference is a review paper by K. Syassen ("Ruby under pressure", *High Pressure Research,* Vol. 28, No. 2, June 2008, 75-126). For the pressure dependence of the wavelength of the R_1 line into the megabar region we initially used the following expression from p. 228 of a 1988 publication by Mao, *Simple Molecular Systems at very high density* (see enclosures in this manual). The following expression given therein is valid under hydrostatic pressure to about 50 GPa.

$$P(GPa) = \frac{1904}{7.665}\left[\left(1 + \frac{\Delta\lambda(\text{Å})}{6942.4}\right)^{7.665} - 1\right].$$

At low pressures this expression reduces to the above rate (+3.646 Å/GPa). From 2005 onwards, we have been using

$$P(GPa) = \frac{1876}{10.71}\left[\left(1 + \frac{\Delta\lambda(\text{Å})}{6942.4}\right)^{10.71} - 1\right],$$

which is valid to about 150 GPa [A.D. Chijioka (Silvera), J. App. Phys. 98, 114905 (2005)]. Currently, we are using ruby spheres obtained from Paris [J.C. Chervin et al. *High Pressure Research*, 21, 305 (2001)].

Because R_1 depends on temperature, it is necessary to measure pieces of ruby both inside the pressure cell (at high pressure) and outside the pressure cell (at ambient pressure) to obtain the pressure-induced shift $\Delta\lambda$(Å). Because of the differing optical paths and angle of incidence into the monochromator, the R_1 fluorescence lines from the sample and reference ruby pieces at ambient pressure are not measured at exactly the same wavelength; the R_1 line from the reference ruby is normally located at approximately 0.3 - 0.7 Å greater wavelength than that from the sample ruby, but this shift changes when the reference ruby is exchanged.

2.2.2 Measurement

The pressure is determined using a simple 1/4-wave monochromator (Jarrell Ash Monospec 25) to measure the shift under pressure of the R_1 fluorescence line of ruby. The monochromator has two holographic plates with 30,000 lines/inch (blazed to 500 nm) and 60,000 lines/inch (blazed to 280 nm); we use the 500 nm blaze. In front of the monochromator is a self-made 35 micron wide collimator which results in a resolution of 3-5 Å. A driving motor (SPEX 1673) set at 0.2 nm/s varies the wavelength of the monochromator.

Because the monochromator is not accurately calibrated, it is necessary to measure the well documented spectrum from a neon lamp together with the ruby lines using one of three recording methods listed below. The neon lines normally measured are located at 7032.413 Å, 7024.050 Å, 6929.467 Å, 6717.043 Å, and 6678.276 Å, respectively. The second line at 7024.050 Å is barely visible in our measurement (same result from Jodl's group in Kaiserslautern), and can thus be neglected. The separation between the R_1 ruby at ambient pressure and the neon lines and between the neon lines themselves should be regularly checked to insure that the pressure measurement system is operating properly.

The detector connected to the monochrometer is either a photomultiplier (PMT) or CCD detector. Currently (2011), the CCD is being used, but a description of how to use the PMT tube has been preserved here in case it ever becomes necessary to revert back. It is advised that you read this section even if you will be using the CCD

Photomultiplier

The photomultiplier used is model R128 from Hamamatsu Photonics which has a high sensitivity near 700 nm. A self-made two-stage preamplifier converts the output current (typically 100 nA) into a voltage of approximately 1 V. A 1 kV power supply (Knottelektronik) supplies the necessary high-voltage to the photomultiplier.

The output from the photomultiplier preamplifier may be recorded in three different ways: (1) using the Linseis X-t plotter on the time range 1 mm/s, or (2) using the chart recorder (Fischer Recordall series 5000) at the speed 9 cm/min, or (3) using the standard measurement program set to take data every 0.1 second. The third method avoids possible errors in the pressure estimate arising from variations in the mechanical plotter or chart recorder speed. The monochromator is set at the desired starting value and placed on the 0.2 nm/sec setting (note that this rate is not calibrated - look on the wall for the proper calibration measurement). Using the two neon lines above (7032.413 Å and 6678.276 Å), we obtain for the above settings that for the X-t plotter 1 mm = 1.74 Å, for the chart recorder 1 mm = 0.382 Å, and for the computer 0.667 Å between measurements taken 0.1 seconds apart.

After the two ruby lines have been observed, turn on the neon lamp and put the proper mirror in place. The power switch for the Ne lamp is near the microscope behind the optical setup board. Andy always made sure the height of the neon line was 0.4 V on the DMM (this value changes dramatically as the aperture is opened and closed - it will often need adjusting). Make sure that the lamp is off and the mirror is moved back during the actual measurement of the ruby lines because neon also has a line that coincides with the ruby lines and the mirror will cut down on the intensity of the ruby lines. After the ruby lines have been recorded, move the mirror (Halbdurchl, Platte 2 in Figure 2.1) into place to allow the neon line to be recorded.

This procedure is repeated for the reference ruby that sits just below the bottom diamond on the backing plate. If the clamp is always put in the same way, the reference ruby will also be in the same place. Since the reference ruby is much larger than the ruby pieces in the cell, it is much easier to find. The peak location for R_1 and the neon peak are determined by the location of the center of the half-peak (half of the distance from the baseline to the peak) and not the maximum of the peak.

Previously, when the driving motor was used, the ruby lines sometimes looked unusual. This was due to the fact that the motor sometimes drove at an incorrect speed. When it did this, the reverse button tended to make the wavelength go back very quickly. The problem could almost always be solved by turning the power off and on a couple of times on the driver. This is no longer a problem with the new stepper motor and controller. If the ruby lines are good, and if the driver is performing consistently, it is possible measure the pressure to an accuracy of ± 1.5 kbar by using the 0.2 cm/s setting on the plotter. Always take at least two spectra to make sure the data is reproducible.

CCD Detector

Measurements can also be made with a two-stage cooled CCD detector (Hamamatsu HC 236-1007) which is connected to the monochromator. To measure a spectrum, first turn on the CCD and open the LabVIEW program "Hamamatsu Demo Kit.vi". Select the correct camera/device from the drop-down list (32-1007) and choose the desired exposure time. Align the optics to maximize the ruby signal, and then save the spectrum. We usually save several spectra and then average them together. Be sure to also measure the reference ruby to determine the shift. Periodically check that the calibration, set using the Neon lamp, is correct. This is especially necessary if the monochrometor has been moved or if the center wavelength of the monochromator has been changed.

2.2.3 Lasers

Three lasers have been used as various times for optical measurements.

- An argon-ion Laser (Ion Laser Technology, Salt Lake City, model 5425A)

with a maximum output power of approximately 150 mW outputs light at two main wavelengths – 488 nm and 514 nm. This laser was initially used for measuring ruby fluorescence in the cryostat, but at high pressure the 514 nm line is too low in energy to sufficiently pump the ruby fluorescence, and the signal from the ruby will decrease. Currently (2011), it is used for Raman and ruby measurements outside of the cryostat (through the metallurgical microscope).

- A He-Cd laser (441.6 nm) was used for measuring ruby fluorescence inside the cryostat until it stopped working in December 2009. The lower wavelength (and hence higher energy) meant that ruby fluroescence could be measured to higher pressures.
- A diode laser with a wavlength of 445 nm (Coherent, CUBE laser) was purchased in February 2010 to replace the He-Cd laser. It has an output of 40 mW. It is currently (2011) being used for ruby fluorescence measurements in the cryostat.

2.2.4 Optical System

Over time, the optical setup used to make ruby fluorescence measurements inside the cryostat has changed as modifications have been made, but the basic idea is still the same – a laser is focused onto a ruby in the sample space using various optical components, and then the resulting spectrum is measured to determine the pressure.

Stefan's paper and thesis give diagrams of the original optical system setup, as shown in Figure 2.1.

1. Spiegel 1: a mirror that adjusts the Ar laser beam toward the DAC.
2. Polfilter 1: a polarization filter which serves to reduce the intensity of the linearly polarized laser beam.
3. Kantenfilter 1: this filter (BG 18, Schott) suppresses all light with wavelength above 550 nm. At high power the laser generates some unwanted radiation in this region.
4. Blende 1: a circular aperture to limit the beam diameter

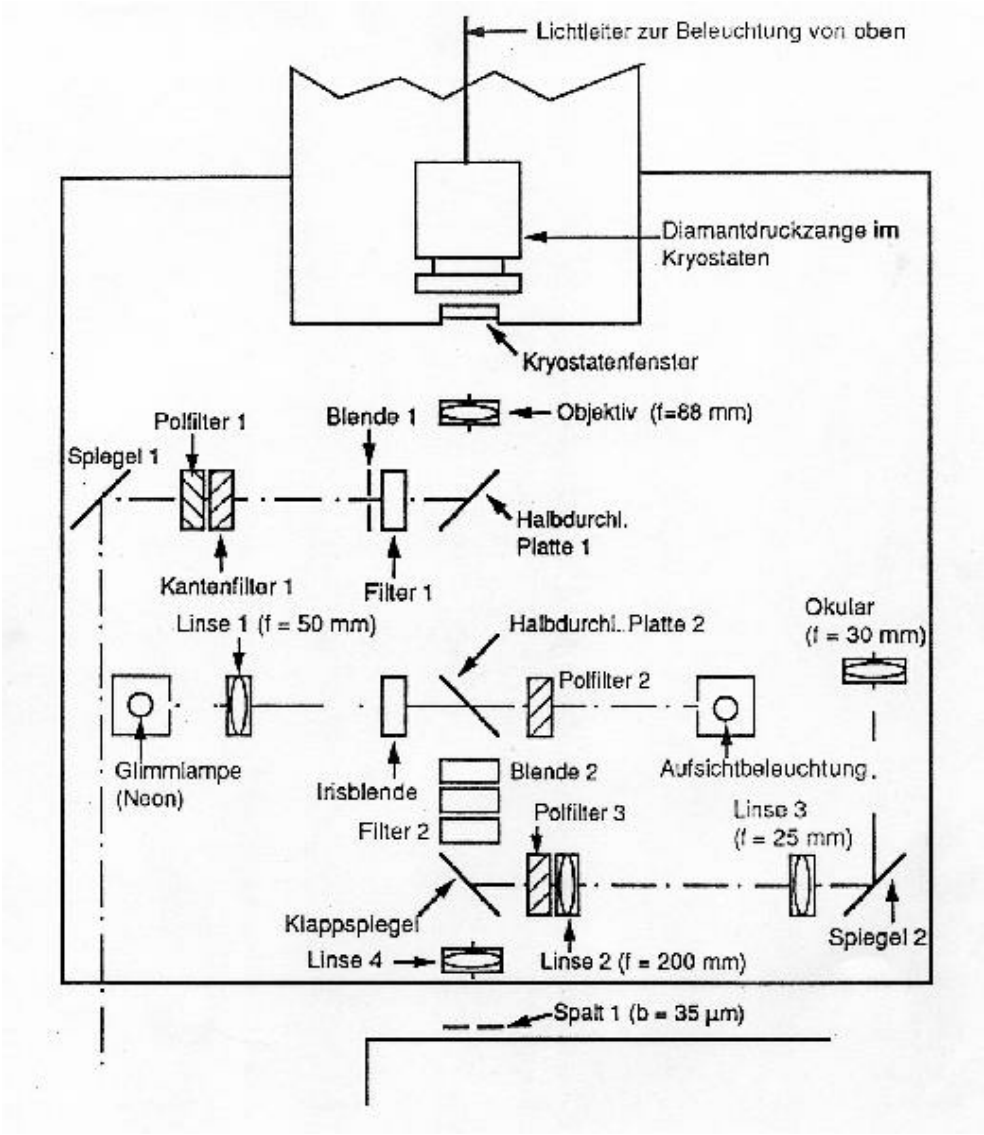

Figure 2.1: Original DAC optical setup.

5. Filter 1: function?
6. Halbdurchl. Platte 1: this was replaced in the year 2001 with a small 6 mm dia. to direct the laser beam upward into the cryostat.
7. Objektiv: an adjustable objective lens with 88 mm focal length to focus the laser onto the cell.
8. Filter 2: this filter cuts out all light with wavelength shorter than 665 nm, in particular scattered laser light, from entering the monochromator, thus allowing the measurement of the very weak ruby fluorescence lines.
9. Klappspiegel: this is a rotating mirror that allows light to be sent into the monochromator or the eye (the microscope setup includes Linse 3 Spiegel 2 and Okular).
10. Glimmlampe: the neon lamp, which is used as a reference line. The movable mirror (Halbdurchl Platte 2) is used to allow the neon light into the monochromator.

Improvements to the optical system were made by James Hamlin, who modified the optical setup slightly. Figure 2.2 shows a schematic of this modified optical setup. More details can be found in his thesis.

In 2010, the 6 mm mirror (M3) was replaced with a dichroic filter.

The following two paragraphs contain a description of how Stefan's setup could be used to measure ruby fluorescence in conjunction with the PMT, but much of the description is valid even when using the CCD.

First, the equipment is turned on and allowed to warm up. Make sure that the low pass filter (Filter 2 cuts out wavelengths below 665 nm) is in place to cut out the laser light from entering either the microscope or photomultiplier!!!!!! Next, focus on the sample with your eye using light through the fiber optic cable and center the gasket hole in the field of view. Make sure that the laser light is centered on the circular aperture. Note the gasket hole diameter to insure that the experiment is going properly. Lower the laser intensity as much as possible with the polarizer and remover the filter to allow the laser into the field of view. Move the laser spot so that it coincides with the gasket hole. Put the filter back

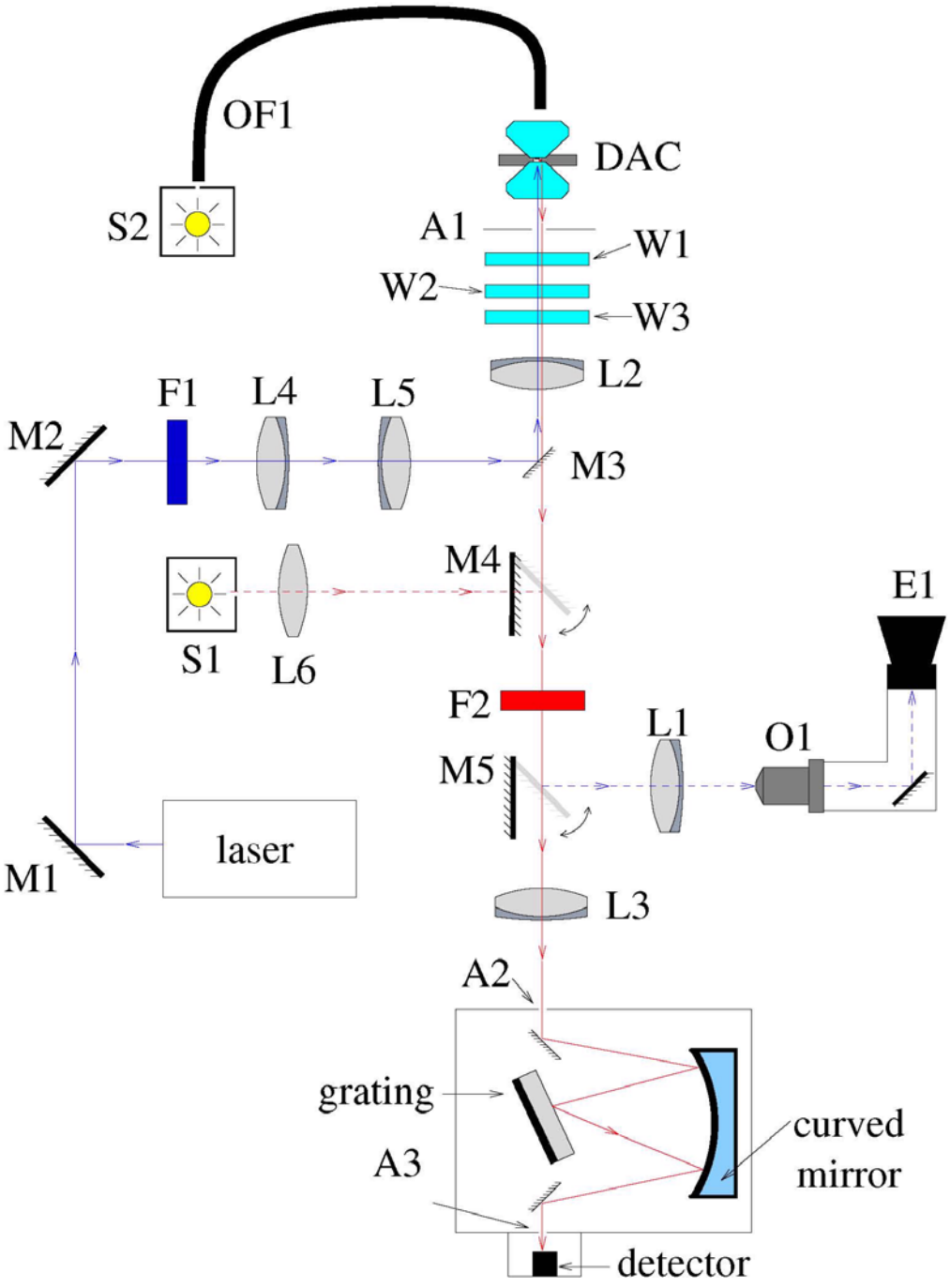

Figure 2.2: Schematic of modified optical setup.

in place and turn the intensity to its maximum. Find the maximum in the spectra using the monochromator (note the readings on the monochromator for different temperatures and pressures). Readings are taken on the digital multimeter in the DC volts setting. GENTLY move the cryostat with the screws to better center the laser spot and maximize the voltage. Adjusting the polarizer can also increase the signal. You should always try to obtain 0.4 V for the maximum (this value might be unobtainable at room temperature for the ruby in the cell if a small ruby is used).

When the pressure measurement is done, make sure that all of the equipment is turned off. Turn of the high power switch on the 1 kV power supply first and allow a couple of minutes for it to cool off before turning off the other switch. The laser cooling fan will continue to be operational even after the laser has been turned off. Make sure that the Ne lamp switch is turned off (this is easy to leave on).

2.3 Measuring Pressure using the Raman Diamond Vibron

At pressures above 100 GPa, measurement of pressure using ruby fluorescence becomes increasingly difficult (see James Hamlin's thesis for an explanation of why). For this reason, at Mbar pressures, it becomes necessary to find an alternate method for determining pressure. Raman measurements of the high pressure diamond vibron provide an accurate measure of pressure. Raman scattering is the inelastic scattering of photons. When light is scattered from an atom or molecule, most of the scattered photons have the same energy as the incident photons (Rayleigh scattering). A small fraction (approximately 0.1 ppm), however, are scattered back inelastically with a slightly higher (Anti-Stokes) or lower (Stokes) energy, as seen in Figure 1.11. Under pressure, the vibrational levels in diamond shift, so measuring the Stokes scattering from the diamond allows pressure of the sample to be determined. Presently (2011), Raman measurements are performed at room temperature outside of the cryostat with the metallurgical microscope using the setup seen in Figure 2.4. Light from the laser is directed via a beam steerer to a lens that focuses the laser onto the sample space. It is then focused onto the back focal point of the objective and travels to the spectrometer via a fiber optic cable. There are two filters - the first eliminates all of the laser lines other than the 514 nm line from the laser, while the second filter, di-

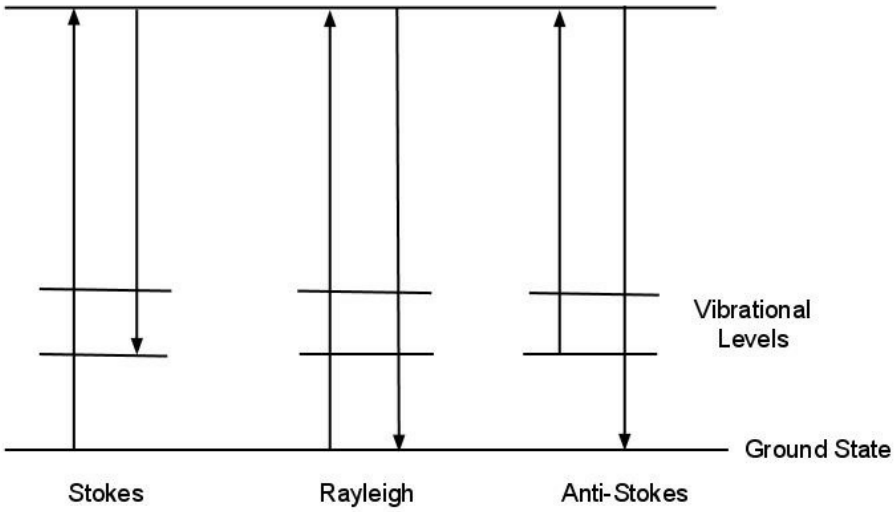

Figure 2.3: Most of the light scattered back from an atom or molecule has the same energy as the incident photons (Rayleigh scattering), but a small fraction (approximately 0.1 ppm) is scattered back inelastically with a slightly higher (Anti-Stokes) or lower (Stokes) energy.

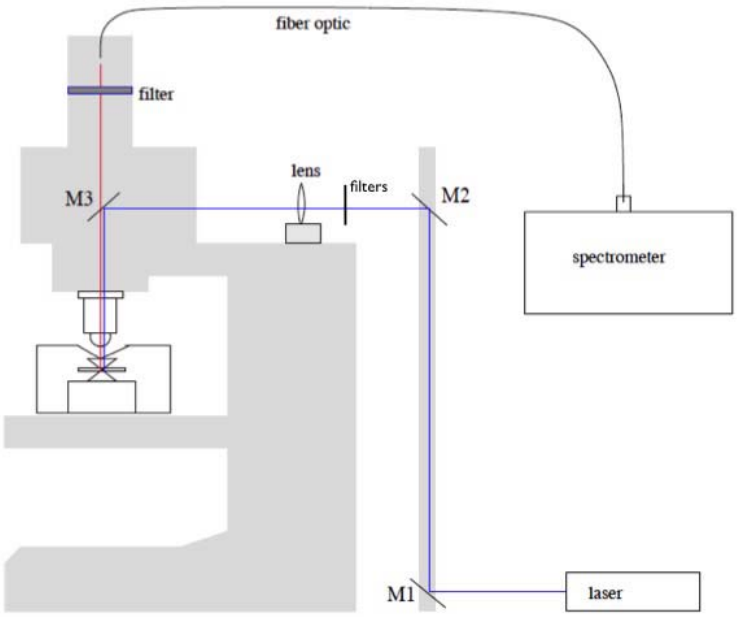

Figure 2.4: Optical setup for measuring Raman diamond vibron using the metallurgical microscope.

rectly before the fiber optic cable, is an edge filter that cuts out the laser line so that only the Raman signal reaches the spectrometer. An Ocean Optics spectrometer (QE65000), purchased in 2009, enables measurement of the Raman spectrum. Since the position of Raman lines is determined by a frequency shift, the wavelength at which the diamond vibron line appears will depend on the laser being used. The Ocean Optics spectrometer can measure wavelength in the range 515 - 615 nm and is specifically designed to be used with the Argon-ion laser, which has a wavelength of $\sim$ 514 nm. The Ocean Optics spectrometer should be used in conjunction with the Spectra-Suite software on the computer to measure the Raman spectrum. See the SpectraSuite manual for more details.

Pressure is determined using the calibration by Akahama and Kawamura (*J.*

Appl. Phys. **100** 043516, 2006), where the pressure is given by

$$P(\text{GPa}) = K_o \frac{\Delta\nu}{\nu_o}\left[1 + \frac{1}{2}(K_o' - 1)\frac{\Delta\nu}{\nu_o}\right].$$

Here $\frac{\Delta\nu}{\nu_o}$ is the relative frequency change, $K_o = 547(11)$ GPa, and $K_o' = 3.75(20)$. At ambient pressure the diamond vibron Raman edge frequency is $\nu_o = 1334$ cm^{-1}. Most of the diamond is at or near ambient pressure, so even when the laser is focused on the sample space, a large ambient pressure peak will be seen. This peak broadens, however, as the pressure at the diamond culet (and hence the sample pressure) increases. The high frequency edge of the Raman line (seen in Figure 2.5) will correspond to the maximum pressure where the laser beam is focused.

2.4 Measuring the Diamond Separation

Using the knowledge of the gasket diameter, pressure and compressibility of He, it is possible to estimate the diamond separation (or gasket thickness) at a given pressure. The density of He is shown in Fig. 2.6 from Poolian & Grimsditch, Europhys. Letters 3, 849 (1986). The initial density is the liquid value (0.125 g/cm^3 at 4.2K, 0.145 g/cm^3 at 2.2K) since liquid is trapped in the gasket for pressure medium. This is done by knowing that the ratio of the volume of the DAC to that of its original volume is the same as the ratio of the volume of the He at the given pressure to its zero pressure value, and assuming mass conservation of the pressure fluid. So the height h at a given pressure is

$$h = h_o \frac{D_o}{D}\frac{r_o^2}{r^2}$$

where the o subscripts denote the zero pressure values, D is the density of He, r is the radius of the gasket hole and h is the height of the gasket hole (the diamond separation). Always check that this number is greater than the sample thickness or you might be pressing directly on the sample with the diamonds.

Alternately, the diamond separation can be measured optically, using the optics provided by the ruby fluorescence measurement apparatus. This can be done by observing the interference spectra produced by white light

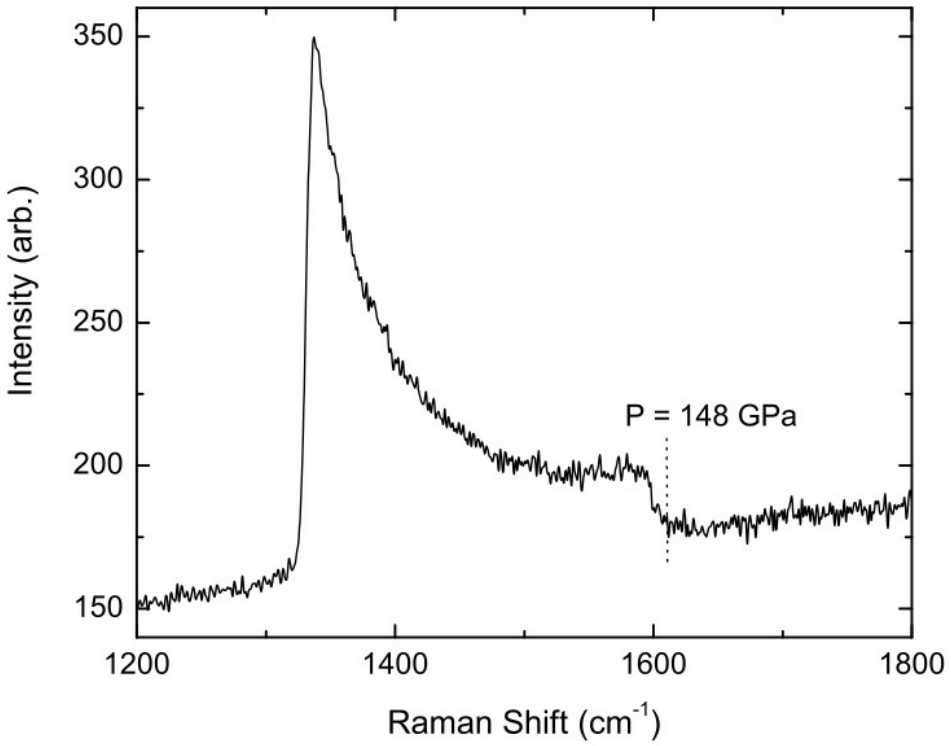

Figure 2.5: Raman diamond vibron spectrum. When the laser beam is focused on the sample space, the high frequency edge gives the pressure.

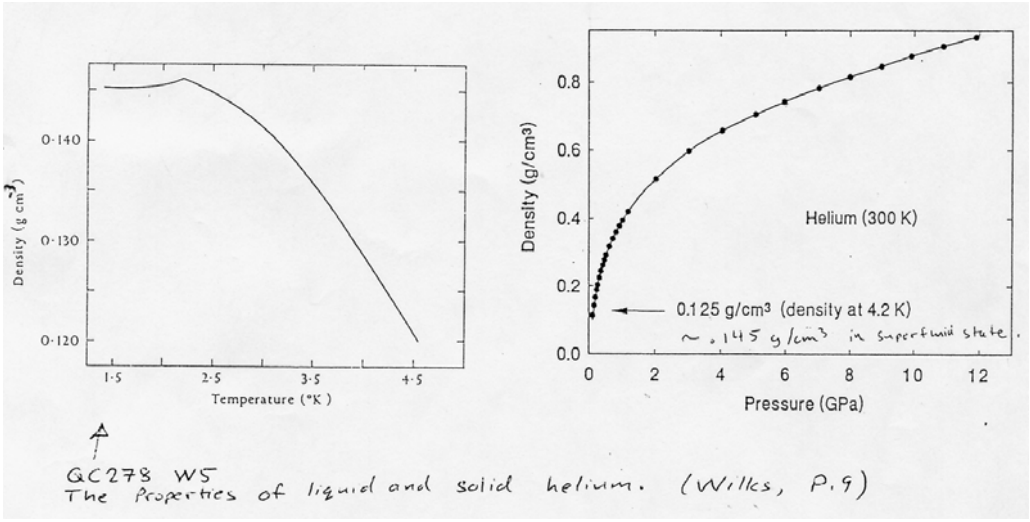

Figure 2.6: The density of He at 300 K. The initial density in the DAC measurement is the liquid value of 0.125 g/cm^3.

illumination of the hole in the gasket. For a detailed explanation of the method and theory, see James Hamlin's thesis, pages 60-63.

2.5 Problems with Ruby Measurements

This section is meant to be a review of the problems that can arise when doing a measurement and the precautions that need to be taken. The following is a troubleshooting list giving the problem and possible solutions.

Ruby lines look bad

1. Ensure the filters and apertures are in place.
2. Make sure that the separation of the R_1-line and the neon line of all of the scans agree.
3. The cryostat or optics may have moved and realignment is necessary.
4. After measuring the neon line, turn off the neon lamp (this is very easy to overlook, so check twice).

5. Always make sure the filter is in place when light is allowed into the photomultiplier (if not, bad things can happen - see Problems With the System section).

6. When the aperture near the cutoff filter is not in place (Blende 2 in Fig. 2.1), multiple reflections may occur, causing the reference line be unusually broad and causing an abnormally large difference between the neon line and R_1-line. To make sure a "good" value is obtained, always check it with a known value for the temperature.

7. If the fluorescence signal is not big enough, make sure that the optics are clean, there is no visible grease in the cell and the laser specs. The Ar-ion laser power can be measured on the laser directly. Two jacks give a DC voltage than can be converted to the laser power with the relation 0.05 V_{DC} = 1 mW laser power. Typically, values near 60 mW were obtained.

3. Problems with the Equipment and Maintenance Info

This chapter gives a running log of problems (and solutions) until 1997.

3.1 Laser Power Supply Board

3/27/92. The power supply board for the Ar-ion laser cooling fans malfunctioned. The same problem occurred to Stefan and was fixed by getting an identical board for replacement of the faulty board. The service representative said the board was incorrectly designed and was completely redesigned nine months earlier. A replacement board was sent free of charge. The company is:
Ion Laser Technology, 263 Jimmy Doolittle Road, Salt Lake City, UT 84116.
Tel: (801)537-1587 or (800)272-7375

3.2 Photomultiplier Tube

4/2/92. The Ar-ion laser was allowed to enter the photomultiplier without the cutoff filter in place for 1 to 2 minutes. Afterward, the photomultiplier tube appeared to have no dark current. All connections were tested and no problem was found. A new tube was ordered for $353 on Friday afternoon and arrived Tuesday morning. That same morning, the dark current was checked and found to be about normal (1-2 nA). John Gilmour, the technical representative, said this behavior was possible, but it was extremely coincidental that the dark current was the same afterwards. The problem could have simply been a bad connection. The tube has functioned properly ever since, and the replacement tube was sent back to Hamamatsu on 4/21/92. The company is: Hamamatsu, 360 Foothill Road, PO Box 6910, Bridgewater, NJ 08807. Tel: (908) 231-0960 or Tech: (800) 524-0504.

3.3 Pickup Coil

7/–/92. The pickup coil stopped functioning in the middle of an experiment (P ∼30 kbar). When the coil was checked, the inner coil was found to not work. The exact reason for the failure could not be determined. The coil was made by Stefan in 1/92 and went through approximately 50-100 thermal cycles.

2003 update: The last of Andy's coils (based on Stefan's design) broke around 1998. Several different designs have been used since then. The most recent design (courtesy of V. Tissen) sports a side-by-side design in which both the primary and secondary coilsystems are each wound from 60 micrometer (diameter) copper wire and have a resistance of approximately 23 Ohms.

3.4 Capillary Line Connection

9/26/92. The capillary line on the diaphragm has a piece on the end that is hard soldered on. This piece is the right size to accommodate the Swagelock fittings (∼1/16 in.). The piece was originally made by Stefan. Andy found that a 16 gauge hypodermic needle has a 65 mil outer diameter and a 45 mil inner diameter and is made of stainless steel. He obtained some of this material from the engineering workshop in the Cupples II basement (they are moving to the medical school very soon). He talked to: John Kreitler, 5-6186, Engineering Technical Services Shop. He was very helpful.

Andy also tried the cyclotron shop: Jim Linders, 5-6559, Cyclotron Shop. They had some stainless steel tubing but none of a useful size. Andy used the pointer sharpener to reduce the outer diameter so the Swagelock fitting of 63 mil diameter would fit on the end. The shop hard soldered this piece on. This sanding, however, was unnecessary as the fittings did fit on without sanding (a small edge on the end of the hypodermic needle piece originally did not allow the fittings to slide on).

10/5/92. Andy: "I thought there would be enough room on the line to put the new fitting on so the capillary line was flush with the fitting. I was wrong, however, and the fitting had to be removed and a new longer one was put on. Todd (head of the physics machine shop) said that there is a good chance that, when a hard solder joint is removed on the thin capillary line, the line

will fail. An extended fitting, however, was successfully soldered on."

3.5 Thermometers

10/10/92. The wires to the Pt3 thermometer, one voltage and one current, broke. They were replaced by soldering the broken wires together with the non-superconducting solder. Epoxy was then put on the solder joints for mechanical stability and to insulate the connection.

10/95. It was observed that the calibration data for the K24 carbon resistor had some "lumps" (calibration points off by a couple hundred milliKelvin) that caused wiggles in the spline interpolation that can often look like a transition. (The calibration errors have been traced back to typographical errors of the original calibration table of the N2G germanium resistor, which was used to calibrate the K24). A polynomial function, used by Stefan Klotz, was used for calculating temperature and satisfactorily removed the wiggles.

6/96. A quick and dirty recalibration by C. Looney showed that the calibration of K24 had changed by over a degree at 40K from the original calibration.

2003: The K24 resistor was subsequently replaced with the N2G resistor. The 1995 recalibration by C. Looney should be used, which corrects a couple of (probably typographical) errors in the original manufacturer's calibration.

2011: The Pt and Ge (N2G) resistors were replaced with a Cernox resistor that can be used over the entire temperature range

2011: The Rh-Fe thermometer in the cryostat was replaced with an Allen Bradley thermometer.

3.6 Laser Power

1/18/93. The Ar-ion laser was found to only be giving off $\sim$10 mW. The mirrors were aligned (alignment procedure is given in the laser manual) and the power returned to 60 mW.

2/19/93. The laser power could not be increased above 5 mW. It was sent to the company: Ion Laser Technology, 3828 S Main St., Salt Lake City, UT 84115.

2/26/93. The repaired laser was returned. A seal between the laser tube and the optics was found to be broken and the optics were cleaned. Total cost was $144.14. A new tube runs $2680, while a refurbished one costs $1800. The tech was: (Don Zanelli, 1-(800)-272-7375).

10/–/07. New laser tube purchased for Ar-ion laser from Midwest Laser for $4,300. Should readjust periodically and not use more power than necessary.

4. The Coil System

4.1 Winding the Tissen Side-by-Side Coil System

The side-by-side coil system shown in Figure 4.1 is currently being used for AC-Susceptibility measurements. It was developed and brought to our lab by Vladimir Tissen during one of his visits from Russia.

We have developed a simple sprig, shown in Figure 4.2, for winding the coils. Before starting, be sure that the stainless steel tube and Teflon disks are cleaned thoroughly. To assemble the sprig, first insert the brass pin through the Teflon disks, stainless steel tube, and brass holder in the order shown in Figure 4.3. After assembling the sprig, spray it once with a lubricant such as *TFL 50 dry lube*. Once the pin is inserted, simply thread the wire through the slits in the side of the brass holder and then pass it through one of the slits on the Teflon disk to start winding it on the stainless steel tube. Depending on the desired inner diameter of the coil, either a 2.7 mm or 3.0 mm outer diameter tube may be used. The free end of the wire should be wound around the brass holder several times and held in place using scotch tape (see Figure 4.4). The brass holder should be placed in the lathe to start winding the coil manually. Do not use the lathe motor! The winding should be very tight - a small loop of solder may be placed on the wire between the coil and the spool to pick up the slack. After each layer, place some "Russian glue" diluted with methanol (about 50-50) on the coil. The "Russian glue" is a brown, semi-transparent glue similar to the "Gorilla glue" sold in America. The secondary coil, which is wound first, should contain 6 layers of 60-64 μm copper wire with 30 turns per layer. Note that the newer Russian glue (since 2009) does not work as well, so instead GE varnish diluted with methanol has been used.

After winding the secondary coil, pass the wire through the same slit on the Teflon disk through which the wire was first inserted. Wind it for a few turns on the brass holder, cut it, and hold it in place using a piece of scotch tape.

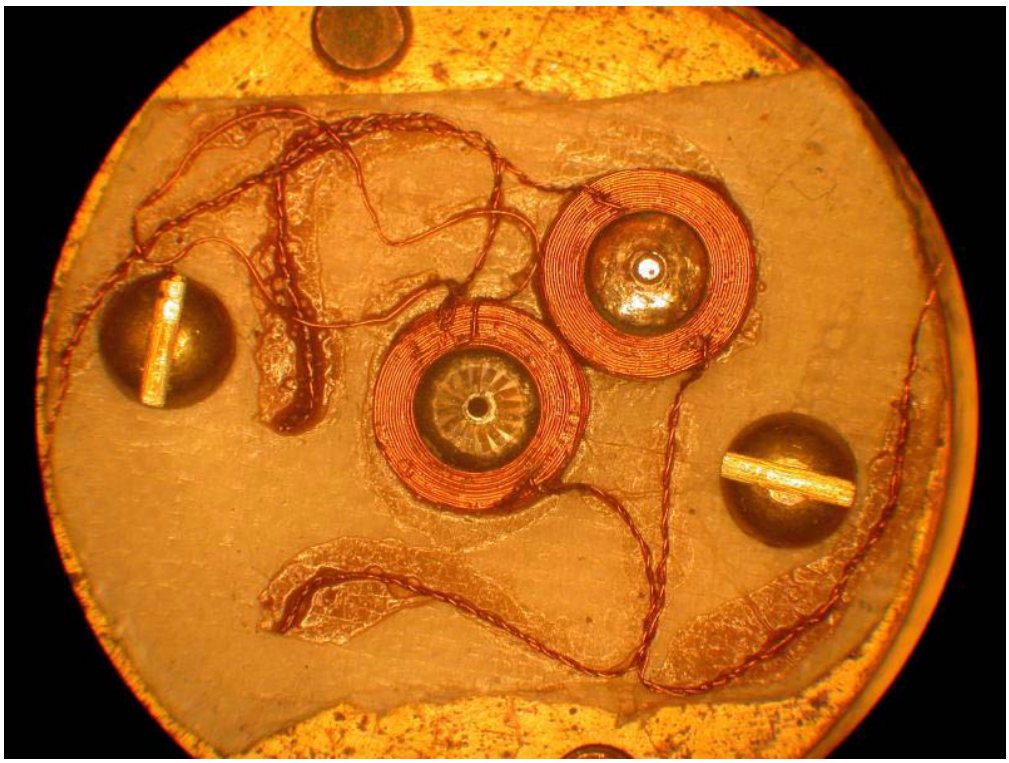

Figure 4.1: The Tissen side-by-side coil system, pictured mounted in the DAC.

Figure 4.2: The coil form used for winding the side-by-side coil system.

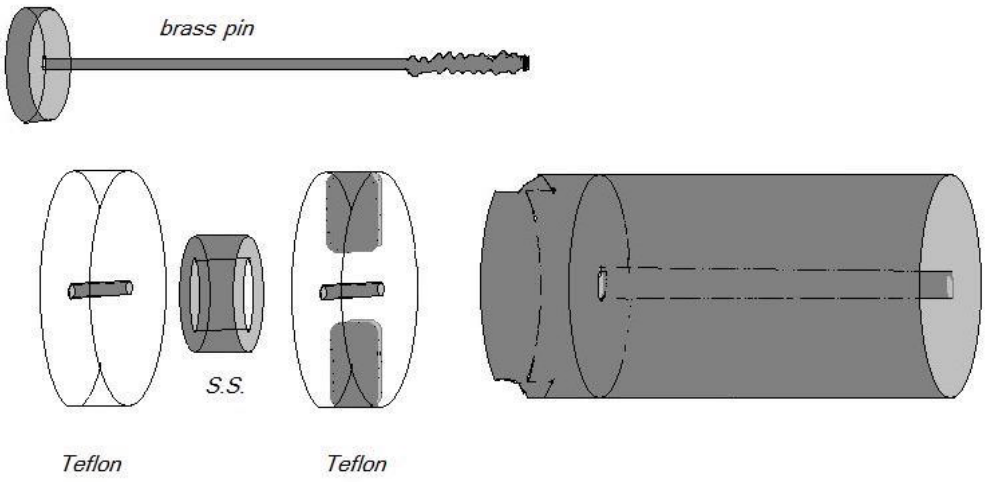

Figure 4.3: Exploded view of the coil winding sprig.

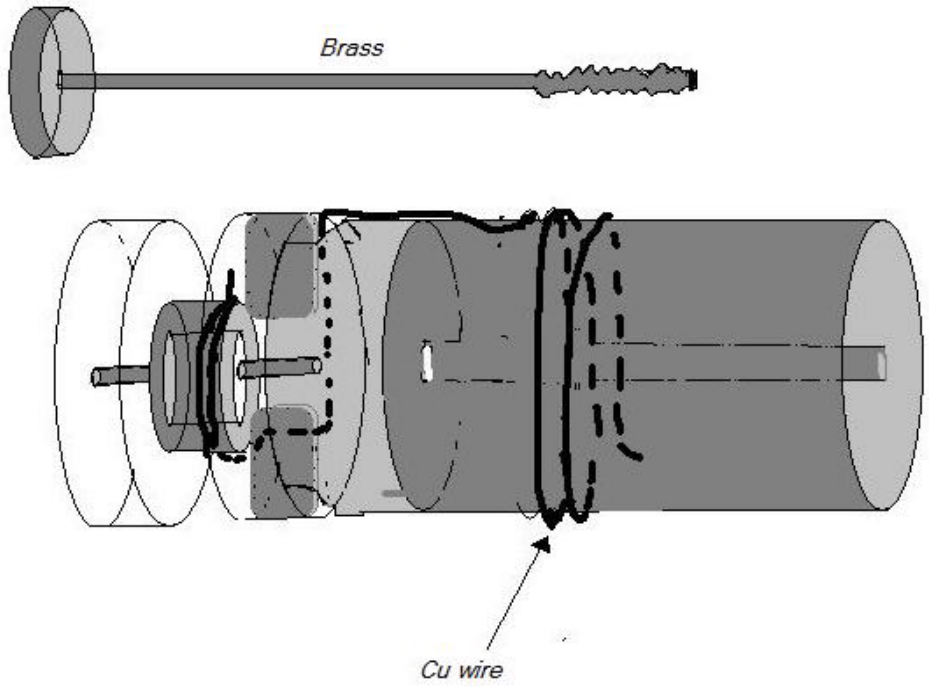

Figure 4.4: Configuration for winding a coil for the Tissen side-by-side coil system.

To wind the primary coil directly on top of the secondary coil, repeat the above procedure. The primary coil also uses 6 layers of wire with 30 turns per layer, and should be wound in the same direction. It is best to start winding the primary coil from the opposite slit in the Teflon disk.

Once the winding is complete, leave the coil for about 2 days so that the glue dries inside and the coil becomes firm. Alternately, one can anneal the coil at 70°C for a few hours. Remove the scotch tape and unwind the wire from the brass holder. Unscrew the brass pin and gently remove the Teflon disks. When removing the disk with slits, be sure that the wires are not inside the slit, otherwise they can be cut or damaged. Look at the coil under the microscope and put some glue on top and bottom of it without letting the glue touch the tube in the middle. Wait another day for the glue to dry.

Hold the stainless steel pin from the DAC box vertically in a vise - the pin's diameter is larger than the inner diameter of the stainless steel tube, but smaller than the outer diameter of the tube. Place some tissue around the pin and put the coil on top of the pin. Using a Teflon plate that has a hole with exactly the same diameter as the outer diameter of the tube and which is very well centered with respect to the coil and pin, push carefully but firmly on the coil so that the coil is removed from the tube. This procedure is best performed under a microscope or a big lens so that you can make sure that no wire is on the tube and that you do not damage the coil. Under a microscope, put a thin layer of glue on the inner side or the coil and let it dry for one day (or anneal it). Unwind the first and last turns of the coil - one belongs to the pick-up coil and the other to the field coil. Place the coil in a safe location and repeat the procedure to make a second coil.

When both pairs of coils are ready, cut the extended wires on each coil to the same length (about 5-6cm), and connect the primary and secondary wires of one of them to two coaxial cables. Connect the primary side to the generator of the lock-in amplifier and the secondary side to Channel A. Adjust the phase angle so that one channel reads 0V, while the other reads some constant number. Record that number and its sign. To test whether the coil is working properly, you can insert a stainless steel bar inside the coil and observe the signal changes. Now connect the other coil in series to this coil and the coaxial cables. The lock-in amplifier channel that used to read 0V should still display 0V, while the other channel, assuming the coils

are identical, shows either a smaller number close to 0V with respect to the previous measurement or a number twice the previous measurement.

1. If the number is close to 0V, the coils have been connected correctly and compensate for each other. The final connections should be made in the same way.

2. If the number is twice the previous measurement, then either the secondary or primary wires of one of the coils should be reversed.

3. If the number is neither closer to 0 nor twice the previous measurement, then you will need to decrease the number of primary turns on one of the coils. In order to determine which coil to do this on, again insert the s.s. bar inside both coils. Turns should be unwound on the coil whose signal has the opposite sign of the original signal. You can alternatively add a few windings on one of the coils and see whether the signal gets better or worse. To determine the number or turns that should be unwound, simply calculate the total number of turns for the primary coil. Then, the total number of turns to unwind is simply given by (total # of turns on primary x original signal x 2) $\div$ (difference in signal from original and both coils). Always decrease the turns from the last turns of the primary. The reason for this is that these turns are farthest from the sample. It is best to recheck signal after removing a few turns rather than removing them all at once realizing that you have unwound too many.

Typically, for the side-by-side coils, you should be able to balance the coils to about 1% of the signal from a single coil. After the coils have been balanced – the total signal from the coils should now be close to zero (about a few μVs) – it is time to connect the coils.

Mark the ends of the primary and secondary coils that should be connected together (you can do this by cutting these wires 1cm shorter). On each coil, gently twist the primary wires together counter-clockwise for 5mm from the coil. Repeat with the secondary wires. Hint: to twist the wires tightly together you will need to keep them about 180 apart while you are twisting them. Measure 1.5cm on all the wires that have been marked and cut the rest of these wires off. Remove 5mm of insulation from the end of the shorter wires. Twist two shorter secondary wires of the coils together for 5mm from

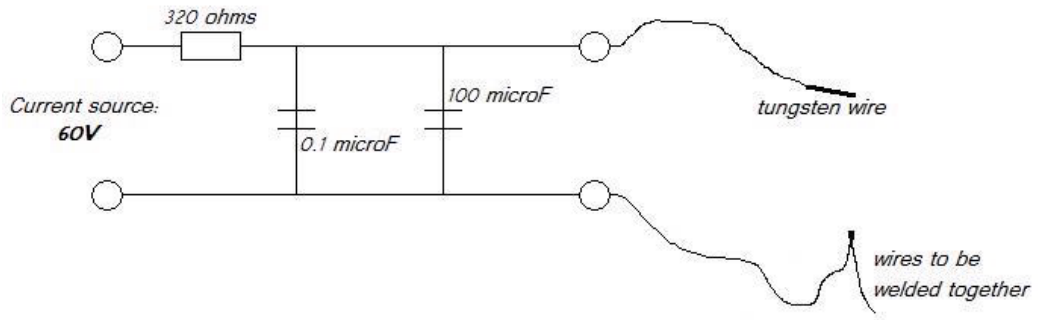

Figure 4.5: Circuit for spark welding wires together on side-by-side coil system.

the end and spark weld them together at the end. Repeat the same procedure with the shorter primary wires. Twist the longer secondary wires together and leave 5mm of the coils untwisted so that the coils have 1cm distance between each other. Then twist the twisted long and short secondary together and primaries together.

Spark welding the wires together provides a strong, durable connection between the wires and also avoids the need to put any solder (with possible magnetic signal) near the coil system. The circuit for spark welding the wires together is shown in Figure 4.5.

In 2010, we acquired a power supply from UCSD that is specifically designed for spot welding. More details can be found on the power supply itself and in the manual (Chapter 5). The power supply can also be used for spark welding Cu wires. The basic procedure is the same as for our home-made spark welder. Isaiah Lim found that the best settings for spark welding Cu wires are as follow:

- 60-100 μm Cu wire ...80 V and an 8 x 300 μs pulse with a 250 μm tungsten wire
- 30 μm Cu wire ...70 V and a 7 x 300 μs pulse with a 100 μm tungsten wire
- Welding 16 μm and 60 μm Cu wire together ...70 V and a 7 x 300 μs

pulse with a 100 μm tungsten wire

4.2 Winding the Cornelius/Klotz Coil System

This coil system was used by Stefan Klotz and Andy Cornelius. Stefan developed the winding technique, which was further refined by Andy Cornelius. This coil system, which utilizes tiny 16 μm wire for the concentric pickup coils and 30 μm wire for the field coil, has since been replaced by a side-by-side design wound from 60 μm wire for both primary and secondary coils. The theoretical loss of sensitivity (larger diameter wire means fewer turns are possible) has been largely countered by the improved compensation afforded by the side by side design. The use of a more reliable (and easier to use) lock-in, the Stanford SR830, has further countered any loss of sensitivity. Finally, it is much easier to wind coil systems with larger wire. [60 μm wire is relatively easy (anyone could learn to do it) and 30 μm wire is more difficult and tedious but still within the capabilities of most experimentalists. 16 μm wire, on the other hand, is horrible; it took Andy one whole summer to wind his first 16 μm wire coil, and another student spent a whole summer trying to wind such a coil without success.] Nevertheless, it may be necessary for somebody in the group to wind a coil system with very small diameter wire, so the sections relating to winding the Cornelius/Klotz coil systems are provided below. Andy's description of the winding technique follows.

The pick-up coils consist of two compensated coils wound concentrically in an opposite manner so that the amount of flux (Φ = NBA, where Φ is the flux, N is the number of windings on a coil, B is the magnetic field, and A is the area of the coil) in each coil is the same. A typical secondary coil-system has 1000 turns ($\sim$3 mm i.d.) on the inner coil and $\sim$442 turns (4.6 mm i.d.) for the outer coil. The inner pickup coil needs to be at least 3.0 mm is diameter to avoid the gasket (3.2 mm gives reasonable clearance). It is best that the field coil has an inner radius less than $\sim$5.2 mm to easily fit on the coil holder in the clamp.

4.2.1 Cornelius/Klotz Secondary (Pickup) Coils

The pick-up coils were wound with 16 μm and the field coil with 30 μm wire, using the non-motorized coil winder. The coils used in the theses studies

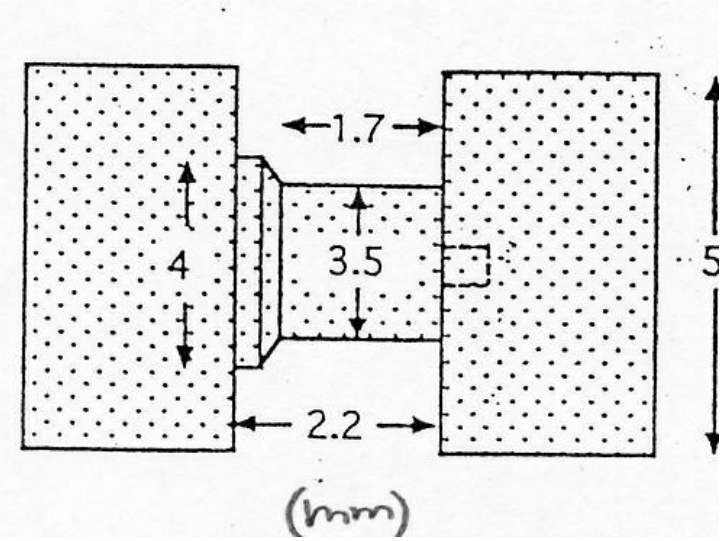

Figure 4.6: A typical delrin coil form used for winding pickup coils. The dimensions are in mm.

were all wound on delrin that was machined to the desired dimensions. The square edged lathe tool was used for the final cuts to ensure that the surfaces were smooth. A final delrin form that would be used is shown in Fig. 4.6.

Before the coil is wound, Teflon spray is applied to the coil form. A *very* thin layer of epoxy free of bubbles (to get epoxy without bubbles it is necessary to pump on the epoxy with the small Balzers pump) is applied to the coil form. Make the layer as thin as possible to just coat the form. The epoxy serves two purposes: (1) It gives a safety coating to the delicate wires on the inside of the coil which may get touched by the gasket and (2) It ensures that there is no gap between the coil form and the cap that is put on the coil form. If there is a gap the 16 μm wire will go into it. The coil form is then turned automatically by the motorized turner to allow a thin even coating of epoxy to harden. Let this go for at least 3 hours.

A thorough layer (30-50 μm thick) of epoxy is then applied to the coil form before winding. Note the starting counter # on the coil winder. The coil is then wound onto the flowing epoxy. Make sure that the first layer of the

coil is done especially well (the turns of the wire should be touching at all points). Making the first layer good makes the entire winding process easier. At the end of each layer, make sure the epoxy is spread over the top of the entire layer. It may not be necessary to add epoxy as it usually builds up at the end of a layer and can be spread back over the top of the windings. When the proper number of turns have been achieved, the first coil is finished. The ends of the coil are then soldered to 64 μm diameter wire. It is necessary to remove the lacquer from all wire ends. This is accomplished by dipping a pipette into concentrated nitric acid (HNO_3). Be careful with this acid - it can burn you and discolors the tables (just look at the subbasement tables). If you look at this process under the microscope, you will eventually (15-30 sec for 16-30 μm wire and $\sim$1 min for 64 μm wire) see the lacquer coming off the wire. Use tweezers to remove any remaining bits of lacquer. Carefully wrap ends of 16 μm wire around 64 μm with the tweezers. Apply some of the acid flux to the area to be soldered and let sit for a minute. Make sure that no extra flux can drop on the coil or coil form. The non-superconducting solder is then used.

After the 64 μm wires have been soldered to the 16 μm wires, it is necessary to secure the solder connections. I found the best way to do this was to use a couple of turns of 16 μm wire to hold the connections in place. A large amount of epoxy is then applied and the coil is rotated until the epoxy is hardened.

After the epoxy has dried, the end cap is removed. The epoxy is then hardened. After hardening, the outer layer is machined to the desired ending diameter. Make sure the coil form is centered properly on the lathe. Using the square edged lathe tool, cut the epoxy where the other coil is desired. Very slowly cut the groove (the width of the lathe tool is exactly the desired width of the coil. Periodically stop and measure the radius until the final radius is reached. If there are any visible air bubbles on the epoxy where the outer coil will be wound, fill them with epoxy and let it harden.

The second coil (outer secondary coil) is wound in a same manner as the first. Add about 20 more windings then you expect to need. Measure the value of the susceptibility for this configuration. Remove two windings a few times and you will be able to estimate the total number of turns necessary for minimizing the background signal. When finished, put a thorough layer

of epoxy on top of the coil and rotate while drying.

Reharden the epoxy. The coil is removed from the delrin form by placing the coil and form in LN_2. Let them get to LN2 temperatures and remove the coil form. Use a pair of tweezers to gently push coil off delrin. If it does not come off immediately, rotate the delrin piece any try again.

4.2.2 Cornelius/Klotz Primary (Field) Coil

The primary coil should be wound before the secondary coil. This is because the primary coil is needed in order to do the compensation of the secondary. The process of winding the primary coil is very similar to winding the secondary coil. First, a coil form is machined to the desired dimensions. (I, Andy, used 5.2 mm i.d. for my coil and a height of 2.0 mm.) I first put a thin layer of glue on the form as mentioned in the secondary coil section. After this hardens, the primary coil is wound with 32 μm wire. I used 600 turns. The 32 μm wire is strong enough to use the motorized winder, if desired. The 32 μm wire is then soldered to 64 μm wire as was done for the secondary coil. Enough epoxy is then applied to cover the coil. The coil is rotated to ensure even setting of the epoxy. The epoxy is then hardened as it was for the secondary coil. The primary coil that is to be put in the DAC with a given secondary coil should be used for the compensation process. If the coils are machined well enough, the secondary should fit snugly inside the primary. If one of the two coils should become damaged, it would then only be necessary to replace the damaged coil. However, I found it necessary to epoxy the coils (primary and secondary) I used together because the secondary coil did NOT fit snugly inside the primary. This led to a great deal of noise since the secondary could move inside the primary.

4.2.3 Miniaturized Coils (Andy)

The miniaturized coils were wound very similarly to the coils just mentioned. Instead of the coil form being all delrin, a piece of tungsten wire is placed in a hole drilled onto delrin. A cap is then slid onto the tungsten wire. Teflon is sprayed onto the tungsten wire and epoxy is placed on the wire. Andy started winding the coil immediately onto the flowing epoxy. The rest of the winding of these small coils is done in a manner similar to those already

Temp (°C)	Time	Hardness (kg/cm^2)
20	12 hours	120
40	3 hours	180
70	45 min	200
100	10 min	250
180	5 min	300

Table 4.1: Physical properties of UHU epoxy used in the winding of the Cornelius/Klotz coil system.

described. See Andy's thesis for the details of the performance tests of the miniaturized coils.

4.2.4 Epoxies

The UHU Endfast epoxy was used for all of the coils prior to implementation of the side-by-side coil system. The hardness of the UHU epoxy after it has been heat treated at various temperatures for a given time is given in Table 4.1.

A drawback of the UHU epoxy is the very large difference in the thermal contraction of the epoxy relative to the copper wire used for the coils. During a thermal cycle, there will be stress on the copper wire due to the difference in the thermal expansion coefficients. This makes the coils have a finite lifetime as the stress will eventually cause the coil to break. Andy Cornelius attempted to use glues from Tri-pac which had coefficients of thermal expansion very near that of copper. The "black" glue was the least viscous and flowed for the longest period of time. It machined easily and dried well. The epoxy contained small particles (up to $\sim 10\ \mu m$ in size) that made winding a coil with the 16 μm wire very difficult. Because of these problems, this epoxy was no longer used.

4.3 Common Pitfalls

Some of the many problems that occurred while winding coils that ruined them are listed in this section. The main things to worry about are:

1. The acid used to strip the lacquer off of the copper wires and the acid flux used for the non-superconducting solder quickly destroy the delrin forms.
2. Try to use short delrin forms as longer ones bend easily when machined.
3. Ensure that the coil form is well centered when machining the epoxy down. This is very important for a good coil system.
4. Do not overheat the coil on the coil form when hardening the epoxy. The delrin warps at temperature of 90°C and above.
5. Do not let the soldering iron get too close to the delrin coil form.

5. Micro Spot Welder Guide

(James J. Hamlin, April 14, 2010)

5.1 Overview

The micro spot welding power supply was designed and built by George Kassabian at the University of California, San Diego, Department of Physics electronics shop, and is based on designs described in several articles [1, 2, 3, 4]. The power supply was designed such that it can roughly reproduce the pulse shapes shown in reference [3]. The principles of making small spot welded joints are described nicely in the above papers, which should be read before attempting a weld. Note that it takes some practice to make successful weld joints and even with practice it is possible to destroy the sample while attempting to weld. Therefore, extreme caution is recommended in cases where you do not have some extra pieces of sample to practice on. It is very helpful to have a logbook in which to record the "voltage level", "pulse width", sample, sample size, and wire diameter which led to successful welds.

5.2 Components

The micro spot welding system is made up of the power supply, a thin tungsten wire electrode, a micromanipulator for positioning the tungsten electrode, and a copper plate which forms the lower electrode. The leads that connect the power supply to the two electrodes should be composed of rather thick braided copper wire (see right of Figure 5.1) since they will carry a significant amount of current. The micromanipulator has a magnetic base which is mounted onto a steel plate. The surface of the steel plate should be smooth so that the micromanipulator does not wobble around when you adjust the dials. Of course, a sample and some small diameter wire to attach to the sample is also necessary. A multimeter connected to the "Puls & Voltage"

Figure 5.1: (left) Micro spot welding power supply and foot switch. (right) Micromanipulator with magnetic base, mounted on a steel plate. The tungsten wire electrode is taped to the micromanipulator.

terminal is useful for determining whether a good electrical connection exists between the electrodes and sample prior to welding. In general, the red output connector on the power supply should be connected to the tungsten electrode and the black output connect to the copper plate electrode. However, in certain cases, better weld joints can be obtained by reversing this polarity.

5.2.1 Power Supply

The power supply can be adjusted to control the pulse voltage level and the pulse width. Figure 5.2 shows a schematic view of how changes in these settings effect the voltage profile across the internal capacitor.

5.2.2 Tungsten Electrode

The requirements for an effective electrode and possible designs are discussed in detail in Reference [1]. We built an extremely simple tungsten electrode assembly by soldering a thin tungsten wire (70 μm diameter) onto the end of a braided copper lead which attaches directly to the red "output" connector on the power supply. The copper lead was then taped in place on the tip of the micromanipulator and then bent to a convenient angle (see right of Figure 5.1). The tip of the tungsten wire can be ground to an appropriate angle using a Dremel tool.

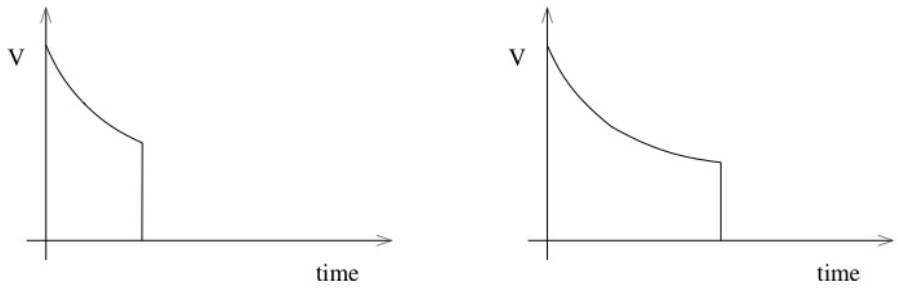

Figure 5.2: Pulse profiles for two different pulses with the same voltage level but different pulse widths.

5.2.3 Lower Electrode

The lower electrode is simply a piece of copper with a rubber sheet glued to the bottom for insulation. It is important that the surface of the copper be smooth and at so that it can be easily polished. A torch is probably necessary in order to get the copper plate hot enough to solder the lead onto it. I put a piece of tape (scotch or masking) across half of the copper plate in order to insulate the gold wire from the copper plate (more about this below).

5.2.4 Gold/Platinum Wire

I have had the most success with gold wire with diameters 0.5, 1, or 2 mil (1 mil = 0.001 inch). In principle, platinum wire would be preferable because it can be flamed (heated with a lighter to make it very malleable). Flaming makes it much easier to bend the wires into position after they have been welded. However, platinum wire seems to stick to the tungsten electrode very strongly (sticking to the electrode is discussed in more detail below).

5.3 Preparations

A key to successful welds is that the sample, wire, copper electrode, and tungsten electrode all be extremely clean. I usually wash the sample in acetone, then ethanol, after which I sand the sample using blue 9 μm sandpaper.

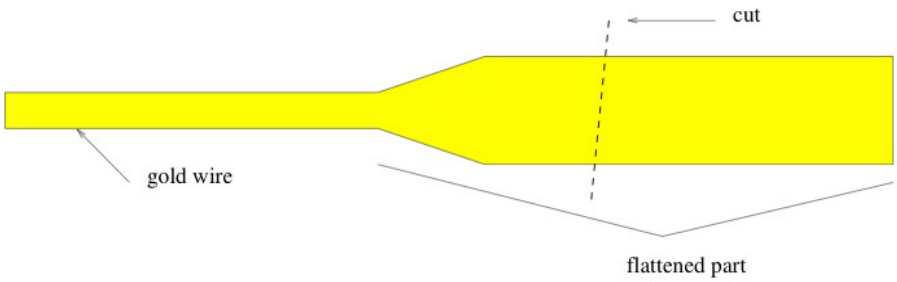

Figure 5.3: The tip of the gold wire is flattened with a metal rolling pin and then cut to length.

Both the surface that bonds to the wire and the surface that touches the copper plate should be sanded. Reference [3] describes an alternative way of cleaning samples when sanding is not practical. I also sand the copper plate before each use (using the same type of sandpaper). After sanding the copper plate I clean the surface with acetone followed by ethanol. The same treatment is given to the tungsten electrode. I also make sure to grind down the tip of the tungsten wire enough that there is no chance of contamination from the previous sample. Note that Apiezon N-grease (which is often used to mount samples) can only be washed off with xylene (which is highly toxic and should only be used in a hood). After washing with xylene be sure to wash away the xylene with ethanol so that you don't inhale xylene fumes during the welding process. Acetone, unlike ethanol, nearly always leaves a thin film of dissolved material behind and therefor should never be used as the final wash.

I found that attening the tips of the gold wire results in stronger weld joints. I flatten the tip by placing the wire on a glass slide and then rolling with a smooth metal cylinder. Be sure that the cylinder is also clean so that is doesn't dirty the wire. After rolling, I cut the flattened part of the wire to a length appropriate to the sample I will weld to (see Figure 5.3).

Now you can put together all of the components as shown in Figure 5.4. First the sample is placed on the copper plate near to the tape. Next the gold wire is layed on top of the crystal. The other end of the gold wire

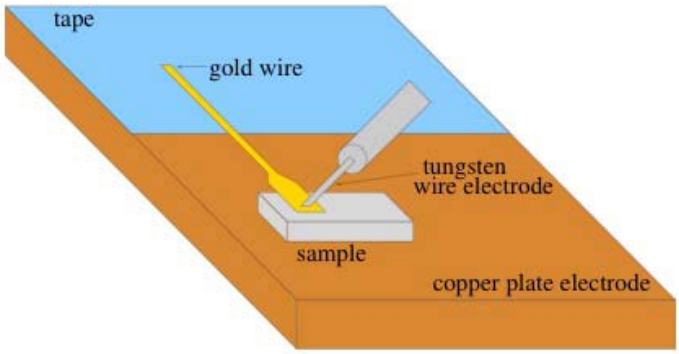

Figure 5.4: Layout of components for bonding wires to small samples. Note that the gold wire does not touch the copper electrode, so that the current must travel through the contact point between the sample and the gold wire.

should rest on the tape rather than the copper plate. This prevents the current from traveling through the length of the gold wire into the plate and either breaking the gold wire or bonding it to the plate. Rather, you want to make sure that the current travels through the junction between the gold wire and the sample. Next bring the tungsten electrode down on top of the gold wire and sample so that it presses frmly down on the assembly and touches only the gold wire. Make sure that there is some tension on the tungsten wire by continuing to lower the electrode a bit after it touches down on the sample. This is important so that when the current pulse melts the junction, the electrode springs down to maintain contact. Too little tension in the tungsten electrode can lead to undesired spark formation.

At this point, you can check if you have a good contact by reading the voltage on a multimeter connected to the"Puls & Voltage" terminal on the power supply. If this voltage is fluctuating then you do not have good continuity and will not be able to obtain a good weld joint. With good continuity, you will measure a stable voltage which is proportional to the "voltage level" dial. Attempting to weld with poor continuity can result in spark formation which will often vaporize part of the sample. If you can't get a stable voltage on the multimeter by pressing down rmly with the electrode, then you may need to further clean/sand the sample, electrodes, or wire.

5.4 Welding

Roughly, one can think of the voltage level dial as determining the maximum temperature that the junction will reach, while the pulse width dial determines the spacial extent over which this temperature obtains. A longer pulse width will lead to a larger portion of the sample being heated. It is best to use the minimum necessary pulse width so that the sample is not modi ed in any signi cant way. A switch provides two pulse width ranges, 30 μs and 300 μs. The pulse width dial allows one to multiply the range by a factor from 1-10. This provides a total range of pulse widths from 30 μs up to 3 ms.

The safest way to determine the required voltage and pulse width is to start from the minimum settings and gradually increase them until bonding occurs. The pulse is triggered with the foot switch. After each pulse is triggered, wait until the voltage on the multimeter returns to the set value before triggering the next pulse. Note that the capacitor takes some time to charge so you may have to wait several seconds between each pulse. A good sign that bonding has occurred is that the sample/wire will move slightly when you trigger the pulse. If you don't observe any movement on triggering the pulse then it is likely that no bonding has occurred and you can increase the voltage and or pulse width and try again. If the voltage and/or pulse width is set too high, the sample or wire may vaporize, or in the case of single crystals, the crystal may cleave.

Often, the tungsten wire will bond to the gold wire. I found that this issue is more problematic when using platinum wire. Separating the tungsten electrode from the gold wire without separating the gold wire from the sample is probably the trickiest part of the process. If you simply pull on the crystal, usually the crystal will break away from the gold wire and the gold wire will remain stuck to the tungsten electrode. I found a simple procedure that usually can separate the tungsten from the gold. After applying a pulse, lift the micromanipulator tip a fraction of a millimeter. If bonding has occurred and the gold wire is stuck to the electrode, both the sample and gold wire will be lifted. Now, by pushing on the edge of the sample that is furthest from the electrode, rotate the sample around (see left of Figure 5.5). Often, rotating the sample a half a turn is sufficient to break the weld between the gold wire and the electrode, though sometimes I have to do 2-3 full rotations. When the voltage level and pulse width dials are set exactly correctly, the

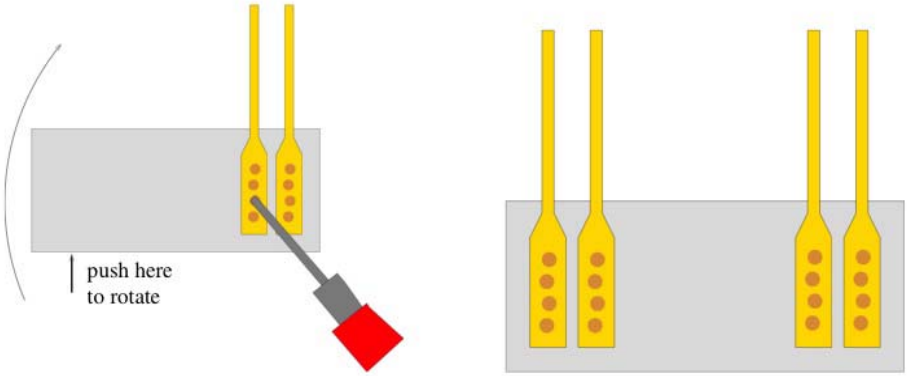

Figure 5.5: Figure 5: (left) When the gold wire becomes bonded to the tungsten electrode, the bond can usually be broken by gently rotating the sample. (right) Completed sample. The dark circles on the gold wires represent positions where the wire has been welded to the sample.

bonding between the electrode and the wire is greatly reduced. Reference [1] contains a discussion of how to reduce bonding between the electrode and wire. If any gold becomes stuck to end of the tungsten electrode, it is best to sand this away before welding again since this will increase the chances of a bond between the electrode and wire.

When the sample is large enough that there is room, I often tack the wire down in several places to insure a mechanically strong joint (see right of Figure 5.5). On some single crystal samples, I have found that there is an extremely narrow window of voltage and pulse width settings which is sufficient to bond the wires without being so high as to cleave the sample. On certain polycrystalline samples, we have found that it is easy to obtain a very low contact resistance but the welds are not mechanically strong and break off easily. In those cases we spot weld the wires on and then apply a small amount of silver paint. The silver paint provides a mechanically stronger joint while the spot weld provides a much lower contact resistance than is possible with the silver paint. I've also used the power supply to join together copper wires (as for ac susceptibility coils where you don't want any superconducting solder near the coil). Figure 5.6 shows some examples

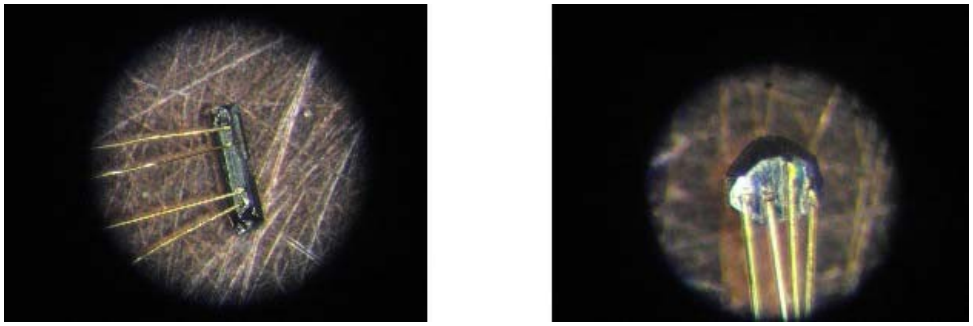

Figure 5.6: Examples of samples wired with the micro spot welding unit. (left) Asingle crystal of $Yb_2Fe_{12}P_7$ with length 1 mm. (right) A 370 m long single crystal of unknown composition grown with U, Fe, and P in a Sn flux.

of successful welds obtained with this system.

5.5 References

1. I. R.Walker and C. J. Moss. Spot welder for making small electrical contacts. Review of Scientific Instruments, 69(7):2747{2756, 1998.
2. Takeshi Hiraoka. The simple spot-welding apparatus. Review of Scientific Instruments, 69(7):2808{2809, 1998.
3. I. R. Walker and C. J. Moss. Further considerations on the preparation of small electrical contacts by spot welding. Review of Scientific Instruments, 71(5):2228{2232, 2000.
4. I. R. Walker and C. J. Moss. Erratum: "Spot welder for making small electrical contacts" [Rev. Sci. Instrum. 69, 2747 (1998)]. Review of Scientific Instruments, 71(11):4344{4344, 2000.

6. Quick Guide for using the Current Ramping Control Unit

6.1 Overview

This unit, seen in Figure 6.1 is designed to be connected to our Kepco ATE 55-20DM power supply. This unit will allow the output current of the ATE 55-20DM to be ramped from 0 to 20 amps at a variety of different rates. The remote control unit outputs 0-1 volts. The ATE 55-20DM has a control voltage input that allows the output voltage to be controlled by a 0-1 volt control signal. One volt at the control voltage inputs allows the power supply to output as much as 20 amps.

6.2 Connections

Make sure that both the ATE 55-20DM and the remote control unit are plugged into the wall for power. Remember that turning on the ATE 55-20DM may blow the circuit that the power supply is connected to. If possible, connect the ATE 55-20DM to a different breaker that the one that the measurement equipment is connected to. Connect the output voltage connector on the remote control unit to the control voltage inputs at the back of the ATE 55-20DM, making sure that red goes to red and black goes to black. Connect the current output of the ATE 55-20DM to a load such as our water-cooled electromagnet.

6.3 Start up tutorial

Before powering up the ATE 55-20DM turn the voltage dial all the way up (clockwise) and the current dial all the way down. Also, check that the switch on the connector block at the back of the supply is set to 'local', as seen in Figure 6.2. Power up the ATE 55-20DM.

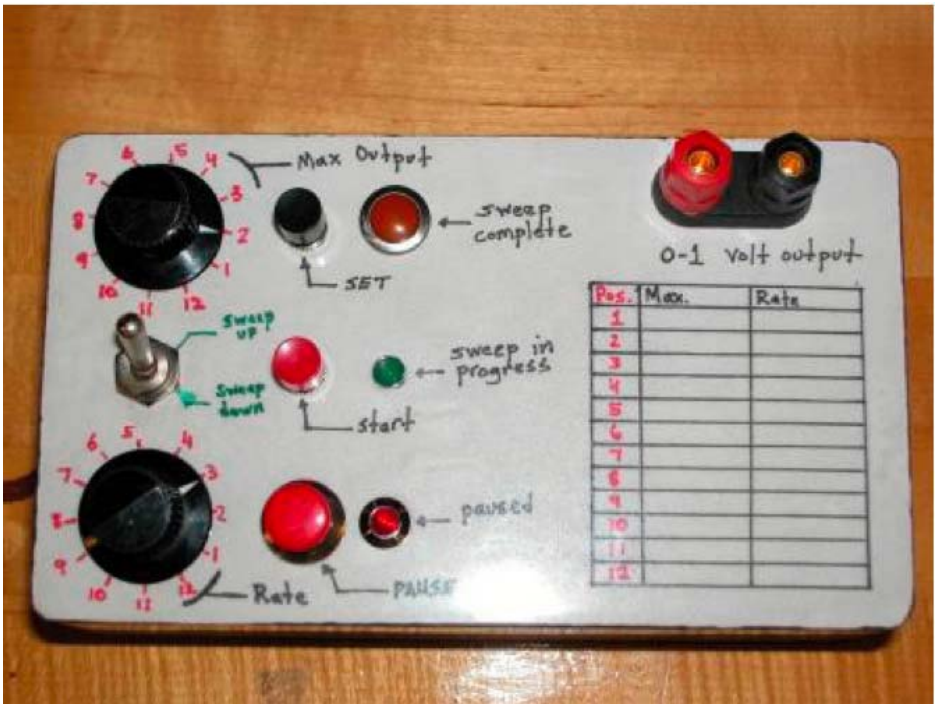

Figure 6.1: Controls for current ramping device

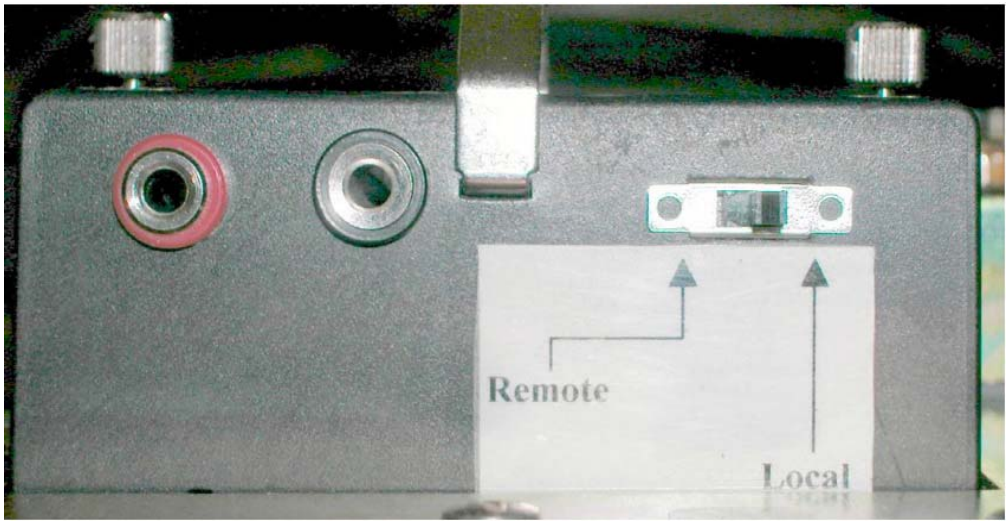

Figure 6.2: Back of one of the power supplies that may be used with the current ramping device.

When the switch is set to 'local' the dials on the front of the supply will control the output current. When the switch is set to 'remote' the output current will be controlled by the control voltage, which is provided by the remote control unit. However the dials on the front of the ATE 55-20DM still have the effect of determining the maximum voltage and current that the supply will allow at its output. For example, set the switch to 'local' and then dial the current up to 3 amps. Now turn the switch to 'remote'. Set the 'Max Output' dial of the remote control unit to position 1 which corresponds to 1 amp. Set the sweep toggle switch to 'sweep up' and press the 'set' button. Notice that the output of the ATE 55-20DM goes to one amp. Now set the maximum output of the remote control unit to 4 amps and press the 'set' button. Notice that the ATE 55-20DM does not allow the current to increase beyond 3 amps. This is a useful safety feature and should be used to make sure that mistakes in using the remote control unit do not result in too much current being sent to the load.

6.4 Controls

6.4.1 Max Output Dial

This dial controls the maximum voltage that will be present at the voltage output of the remote control unit. Sweeps will start or stop at the value determined by the position of the Max Output Dial (see Table on next page). Position 12 corresponds to 1V which will cause the ATE 55-20DM to output 20 amps (provided that the voltage and current dials on the ATE 55-20DM allow this high of an output; see tutorial). The values in the table on the right of the remote control unit are listed in amps (as produced when the remote control unit is connected to the ATE 55-20DM).

6.4.2 Rate Dial

This dial controls the rate at which the voltage output is linearly varied once a sweep has been initiated by pressing the "Start" button (see Table on next page).

6.4.3 Sweep Toggle Switch

This toggle switch controls whether the sweep will be an increasing or decreasing sweep.

If the Sweep Toggle is set to "Down" and the "Start" button is pressed, the voltage will sweep from the Max Output to zero. If the Sweep Toggle is set to "Up" and the "Start" button is pressed, the voltage will sweep from zero to the Max Output.

If the Sweep Toggle is set to "Down" and the "Set" button is pressed, the output voltage will be set to zero. If the Sweep Toggle is set to "Up" and the "Set" button is pressed, the output voltage will be set to the Max Output.

6.4.4 Set Button

This button sets the output of the remote control unit to zero (Sweep Toggle set to "Down") or Max Output (Sweep Toggle set to "Up").

6.4.5 Start Button

This button initiates a sweep from zero to Max Output (Sweep Toggle set to "Up") or from Max Output to zero (Sweep Toggle set to "Down"). While the sweep is in progress the green "Sweep in Progress" indicator will light up. When the sweep is finished, the orange "Sweep Complete" indicator will light up.

6.4.6 Pause Button

Pressing this button once while a sweep is in progress causes the sweep to pause and the red "Paused" indicator will light up. Pressing the Pause Button again will allow the sweep to continue.

It is important to note that settings such as the sweep direction, max output and sweep rate can be changed during a sweep only by first pausing the sweep with the pause button, making the changes and then un-pausing the sweep.

6.5 Dial Position Values

The values for each position of the "Max Output" and "Rate" dials are listed on a table on the right of the remote control unit. These values are written on top of the transparency covering the unit so that the values entered on the table may be changed (see the next section for information about changing dial values).

At the time of the writing of this guide (May 18, 2005) the values of the dials are those shown in the table below. The values are listed in Amps (Max Output) and Amps/minute (Rate) assuming that the remote control unit is hooked up to the ATE 55-20DM. Simply divide these values by 20 to get the values in Volts and Volts/minute at the output of the remote control unit.

Position	Max. (Amps)	Rate (Amps / minute)
1	1	20
2	2	10
3	3	8
4	4	5
5	5	2
6	6	1
7	8	0.5
8	10	0.4
9	12.5	0.3
10	15	0.2
11	17.5	0.1
12	20	0.01

6.6 Programming

The remote control unit is built around a Parallax Basic Stamp II. This is basically a tiny computer that can be programmed using a version of the language Basic. One might want to reprogram the unit if the currently programmed sweep rates or max output values are not suitable.

As of the writing of this document the program that is loaded into the remote control unit is contained in a file called Remote_OS2.bs2, which is stored on our Dell computer in C:\usr\Basic Stamp. This file is also backed up on the Gateway computer. Programs are loaded into the unit by connecting to the computer with a serial cable, loading the program you want to put on the basic stamp into BASIC Stamp Editor v1.33 (also located on the Dell computer or available for download at Parallax's website), and selecting the option to load the program onto the stamp. There is plenty of documentation available from Parallax on all the details of this process.

The contents of Remote_OS2.bs2 can be found in the "Computer Programs" manual.

7. Vickers Hardness Indenter

7.1 Background

The Vickers Hardness test can be applied to different materials across a very wide range of hardness. The Vickers test uses a square-based diamond pyramid with a 136^o point angle. The load (usually 50 kg, but could be 5, 10, 20, 30, or 120 kg) is applied via the pyramid against the smooth, firmly supported, flat surface of the test specimen for 30 seconds. The resulting hardness reading depends on the load and the area of the diamond pyramid impression, in accordance with the formula

$$HV = \frac{\text{Load (kgf)}}{\text{Area of the Pyramidal Impression (mm}^2\text{)}}$$

$$HV = \frac{1.854 * 0.5 \text{ kg}}{\text{diagonal 1 * diagonal 2 (mm}^2\text{)}}$$

The Vickers Hardness Indenter used is pictured in Figure 7.1.

7.2 Instructions

Before using the indenter, the specimen must be thoroughly polished; it is recommended to use one-micron 3M imperial lapping film. Then place the specimen level on the platform and raise or lower it until the pivot arm is parallel with the baseplate when the diamond tip rests on the specimen. Raise the pivot arm with the height adjuster so that the diamond tip no longer rests on the specimen.

Move the specimen horizontally to the desired position. Very slowly lower the height adjuster until the diamond tip contacts the specimen and takes up the full weight of the pivot arm. Let the load rest on the specimen for 30 seconds, and then slowly use the height adjuster to raise the diamond

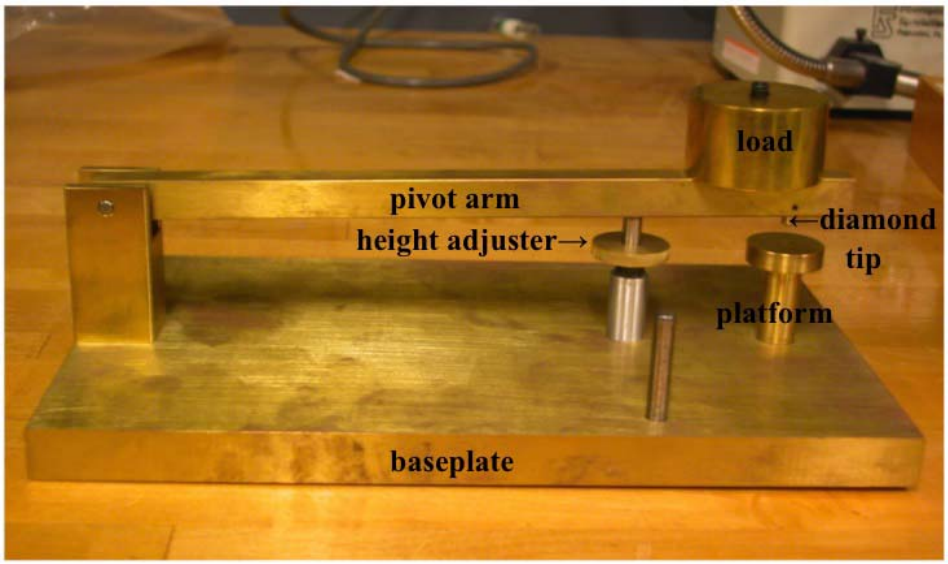

Figure 7.1: The Vickers hardness indenter used for measuring hardness. It uses a 0.5 kg load.

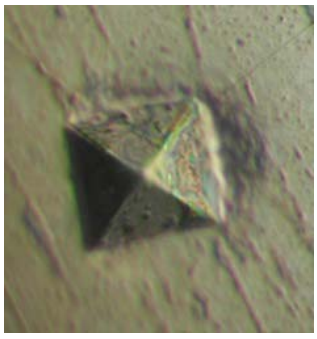

Figure 7.2: Diamond indentation. The depth of the indentation, as determined from the length of the diagonals, will indicate the hardness of the material being measured.

tip up off the specimen. Multiple measurements should be taken at different locations.

The specimen should then be placed under a microscope to view the indentations, seen in Figure 7.2. Under a high magnification, measure the lengths of the two diagonals. Use the equation above to determine the Vickers Hardness HV 0.5 (for a 0.5 kg load). The Vickers Hardness can be converted by going to *http://www.efunda.com/units/hardness/convert_hardness.cfm?cat=Steel&HD=HV* or by using the conversion table shown in Figure 7.3.

7.3 Results:

Non-magnetic Tungsten carbide:

- 1524 HV 0.5
- Standard deviation: 163

Rhenium:

- 514 HV 0.5
- Standard deviation: 9
- HRC: 50

Russian Steel (non-annealed):

- 469 HV 0.5
- Standard Deviation: 15
- HRC: 47 (near center 51, near perimeter 46, determined 5/11/05 Forrest)

Russian Steel (annealing # 1):

- Annealing procedure: 600^{o}C for $7\frac{1}{2}$ hours. Oven turned off and allowed to cool to 280^{o}C. Sample then removed from oven and air-cooled
- 565 HV 0.5

Vickers	Brinell		Rockwell		Shore	Vickers	Brinell		Rockwell		Shore
	10 mm	3,000kg	B Scale	C Scale			10 mm	3,000kg	B Scale	C Scale	
Hv		W.C	HRB	HRC	Hs	Hv		W.C	HRB	HRC	Hs
940				68.0	97	410	388	388		41.8	
920				67.5	96	400	379	379		40.8	55
900				67.0	95	390	369	369		39.8	
880		767		66.4	93	380	360	360	(110.0)	38.8	52
860		757		65.0	92	370	350	350		37.7	
840		745		65.9	91	360	341	341	(109.0)	36.6	50
820		733		64.7	90	350	331	331		35.5	
800		722		64.0	88	340	322	322	(108.0)	34.4	47
780		710		63.3	87	330	313	313		33.3	
760		698		62.5	86	320	303	303	(107.0)	32.2	45
740		684		61.8	84	310	294	294		31	
720		670		61.0	83	300	284	284	(105.0)	29.8	42
700		656		60.1	81	295	280	280		29.2	
690		647		59.7		290	275	275	(104.0)	28.5	41
680		638		59.2	80	285	270	270		27.8	
670		630		58.8		280	265	265	(103.5)	27.1	40
660		620		58.3	79	275	261	261		26.4	
650		611		57.8		270	256	256	(102.0)	25.6	38
640		601		57.3	77	265	252	252		24.8	
630		591		56.8		260	247	247	(101.0)	24	37
620		582		56.3	75	255	243	243		23.1	
610		573		55.7		250	238	238	99.50	22.2	36
600		564		55.2	74	245	233	233		21.3	
590		554		54.7		240	228	228	98.10	20.3	34
580		545		54.1	72	230	219	219	96.70	(18.0)	33
570		535		53.6		220	209	209	95.00	(15.7)	32
560		525		53.0	71	210	200	200	93.40	(13.4)	30
550	505	517		52.3		200	190	190	91.50	(11.0)	29
540	496	507		51.7	69	190	181	181	89.50	(8.5)	28
530	488	497		51.1		180	171	171	87.10	(6.0)	26
520	480	488		50.5	67	170	162	162	85.00	(3.0)	25
510	473	479		49.8		160	152	152	81.70	(0.0)	24
500	465	471		49.1	66	150	143	143	78.70		22
490	456	460		48.4		140	133	133	75.00		21
480	448	452		47.7	64	130	124	124	71.20		20
470	441	442		46.9		120	114	114	66.70		18
460	433	433		46.1	62	110	105	105	62.30		
450	425	425		45.3		100	95	95	56.20		
440	415	415		44.5	59	95	90	90	52.00		
430	405	405		43.6		90	86	86	48.00		
420	397	397		42.7	57	85	81	81	41.00		

Figure 7.3: Steel hardness conversion table for Vickers HV 0.5

- Standard Deviation: 23
- HRC: 53

Russian Steel (annealing # 2):

- Annealing procedure: 700^{o}C for 2 hours and 10 minutes and then immediately removed from oven and air-cooled.
- 547 HV 0.5
- Standard Deviation: 13
- HRC: 52

Comparison Measurements:

- Tungsten carbide: 1440 HV 0.5
  - With standard deviation: 50
  - Percent difference: 5.83 %
- Russian Steel (annealing # 1): 559 HV 0.5
  - With standard deviation: 14
  - Percent difference: 1.07 %

8. Copper Plating

An electrolytic solution was prepared with 200 grams of $CuSO_{4.5}H_2O$ and 25 mL of concentrated H_2SO_4. Distilled / deionised water was then added to make it the whole solution 1 L. The piston was cleaned thoroughly and the insulation of the Cu anode (1 mm thick) was removed by a razor blade. The wire was formed into a circle 1.5 inches in diameter and placed around the piston as shown in Figure 8.1. The electric connection was made as shown below and the current was 1.5A with net deposition rate of $\sim$1 μm/min. The deposition was made for four minutes and to secure uniform deposition the piston was rotated every minute by 90^o. If the system is working properly then a faint glow will be visible in the anode region.

The piston was sanded clean with 0.5 μm diamond powder and a Kim wipe to remove the softly adhered copper. The same powder was used to fit the piston in to the cylinder by applying a small amount to the piston and then moving the piston back and forth in the cylinder

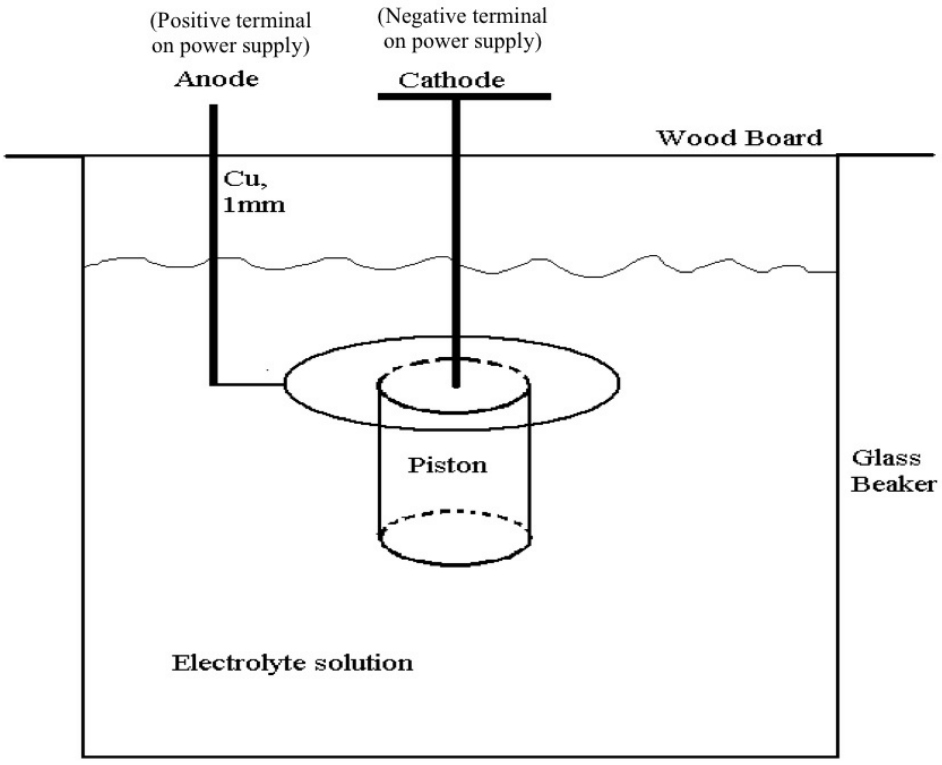

Figure 8.1: Setup for copper plating the DAC piston. Note that the circular wire should be placed around the center of the piston, making sure that it does not touch it.

9. Resistivity Measurments in the DAC

(M. Debessai, 14 March 2007)

This was documented when Takahiro was preparing his cell for a measurement of $CaLi_2$ with lithium 5% deficient in March 2007.

- Gasket - Rhenium, 10 mm by 10 mm by 0.25 mm
- Anvils - 0.3 mm yellow diamond (from Japan)

The gasket was preindented to 30 - 40 μm and a 150 μm hole was drilled using the EDM (see Figure 9.1).

Attach the gasket to the board with double sided tape. The board should be kept in place so that the gasket will be in the same orientation as it was during preindentation. Cover the whole gasket with scotch tape - this will act as insulation between the platinum electrode and the gasket outside of the sample chamber. Clean the surface of the tape using ethanol (ethyl alcohol). Cut the insulation tape on the preindented part as seen in Figure 9.2.

Make a very sharp tooth pick and use it to put a very tiny pit of diamond powder to almost cover the entire preindented area, as seen in Figure 9.3. If using Al_2O_3, be sure to use a bit more powder since it is more compressible than diamond.

Apply pressure up to 20 GPa so that the insulation material will be pressed against the diamond and takes the shape of the preindented gasket. Since the metal gasket is more compressible than the diamond, the preindented gasket material will further decrease its thickness. A circular ring across the culet edge through the perimeter (see Figure 9.4) is an indication that the system has been pressurized. If there is some diamond powder left around the culet which falls down after the diamonds are taken apart, use a sharp tooth pick to clean them up.

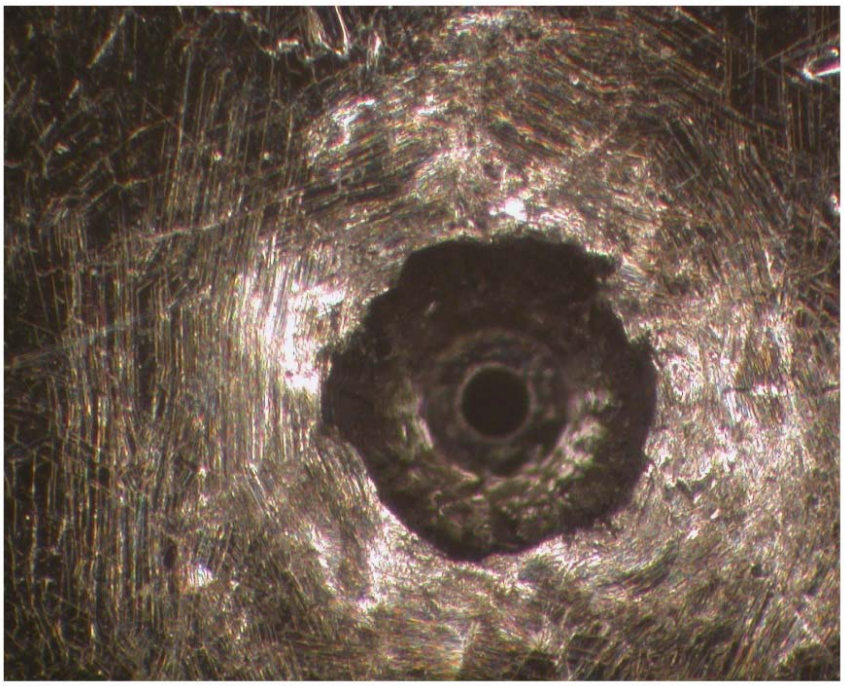

Figure 9.1: Hole drilled in Rhenium gasket with EDM.

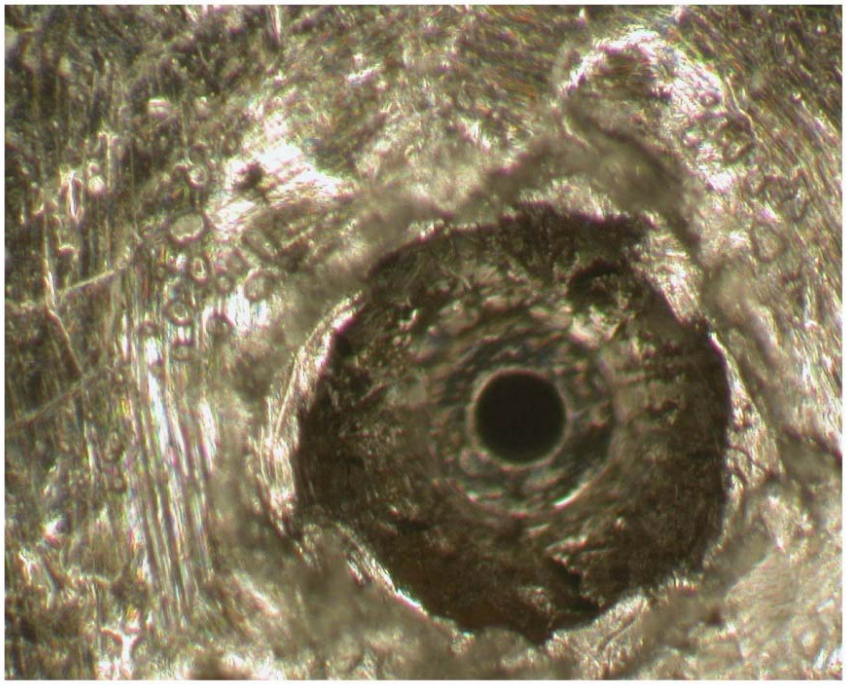

Figure 9.2: The insulation tape cur out in the preindented area.

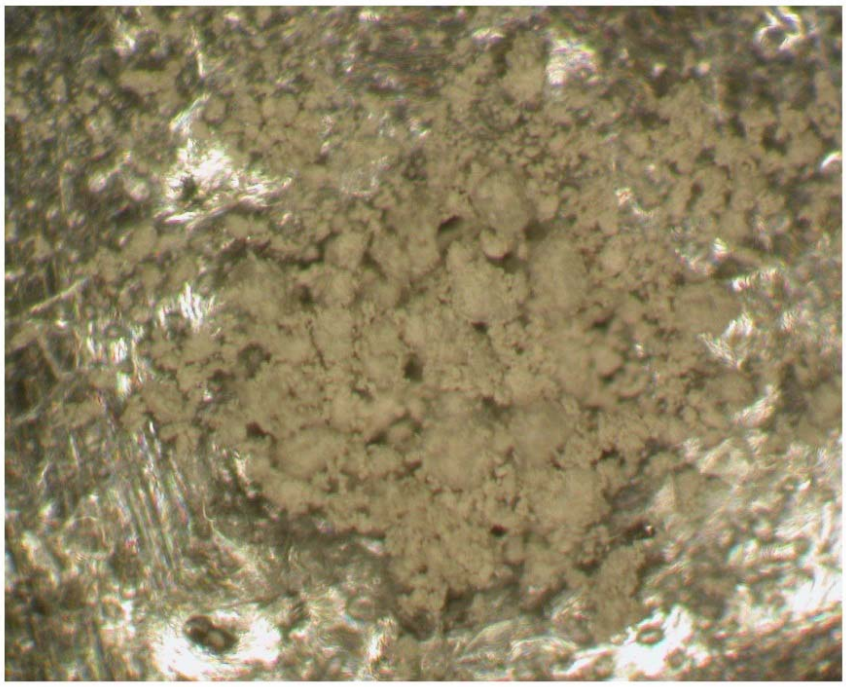

Figure 9.3: Diamond powder filled in the preindented area.

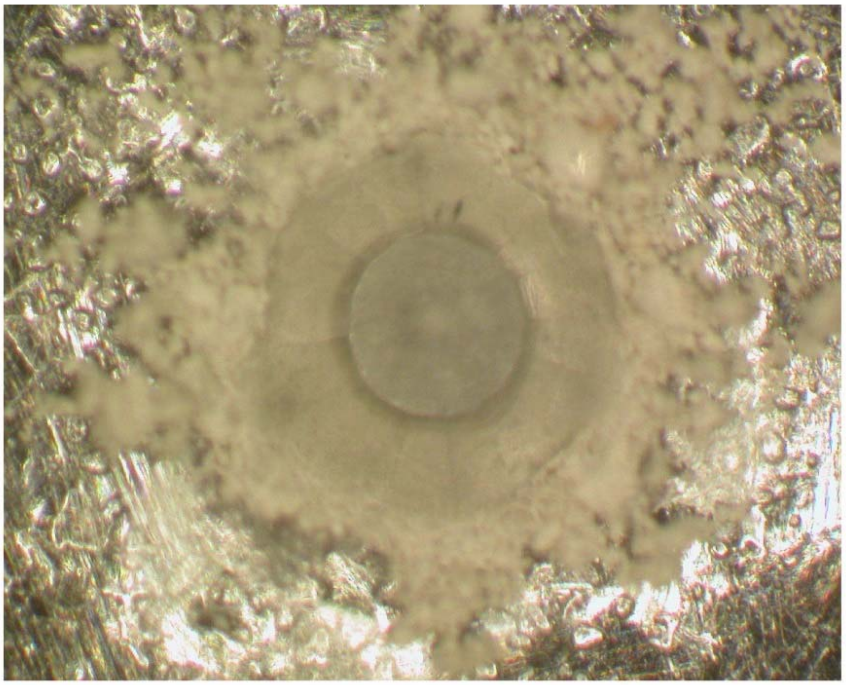

Figure 9.4: Insulation layer after being pressed.

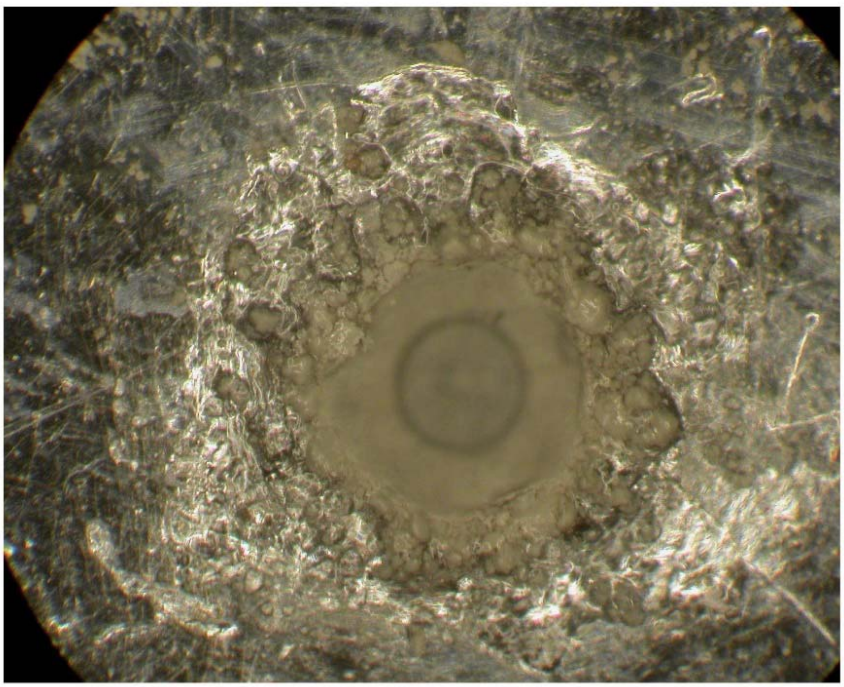

Figure 9.5: Crazy glue on the perimeter of the preindented circle.

To keep the insulation material in place, put a very tiny bit of crazy glue on the edges of the preindentation (see Figure 9.5)using a sharp kim wipe and let it dry for one or two minutes. Make sure that nothing gets toward the faces of the preindented area, specially toward the culet. If so, try to dig it out. Clean out the extra crazy glue that flows out of the preindented edge to the outside of the gasket that is covered by the tape.

At the center of the diamond insulation, poke a cylindrical 100 μm hole ($\frac{1}{3}$ of the culet diameter) using a very sharp blade, followed by a tungsten wire or needle (see Figure 9.6).

Prepare four platinum electrodes. Platinum has been found to be more stable as compared to copper at high pressures. Gold and palladium have also been used in Shmitzu's group. A new blade has to be used for cutting the electrode. To cut the electrode, use a cutting board. Clean the platinum with ethanol and make sure it is flat on the board. Hold the electrode with

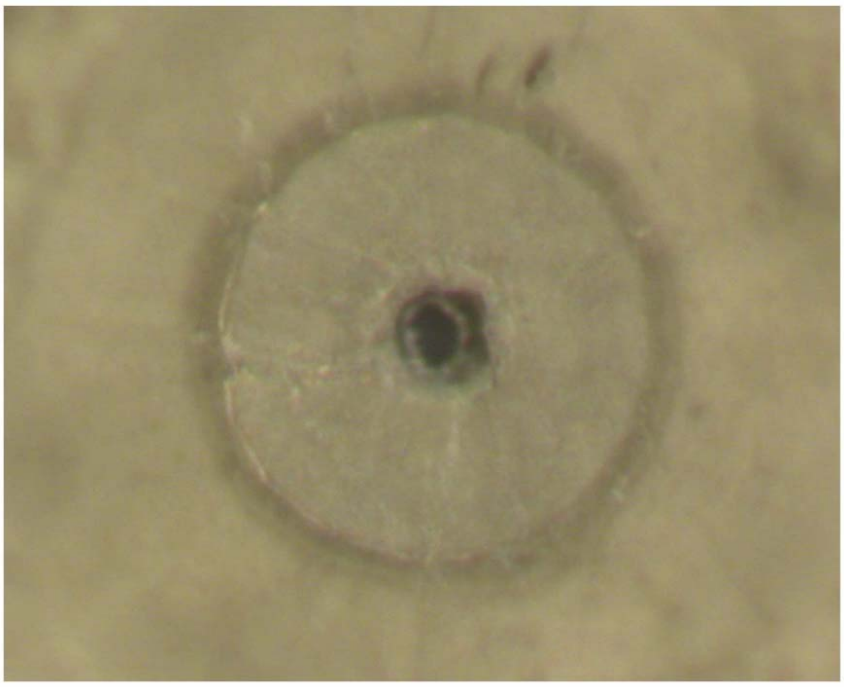

Figure 9.6: Sample chamber.

Figure 9.7: Initial electrode position.

a very small piece of scotch tape and place it on the gasket so that it passes over the preindented area and the tip is close to the edge of the hole, as seen in Figure 9.7.

Align and push the electrodes towards the center of the hole using a sharp tooth pick. Rearrange the electrodes by pulling them if they are too long or changing their orientation by positioning small pieces of scotch tape (see Figure 9.8). Care has to be taken in rearranging the electrode as they can be easily broken.

Push the electrodes with a diamond very slowly and watch the position of the electrodes very closely. If the electrodes seem to be out of order, then rearrenge them. As soon as you touch the insulation layer there should be a color change in the facets. The applied pressure shouldn't be too much to distort the shape of the sample chamber. This step might take as long as

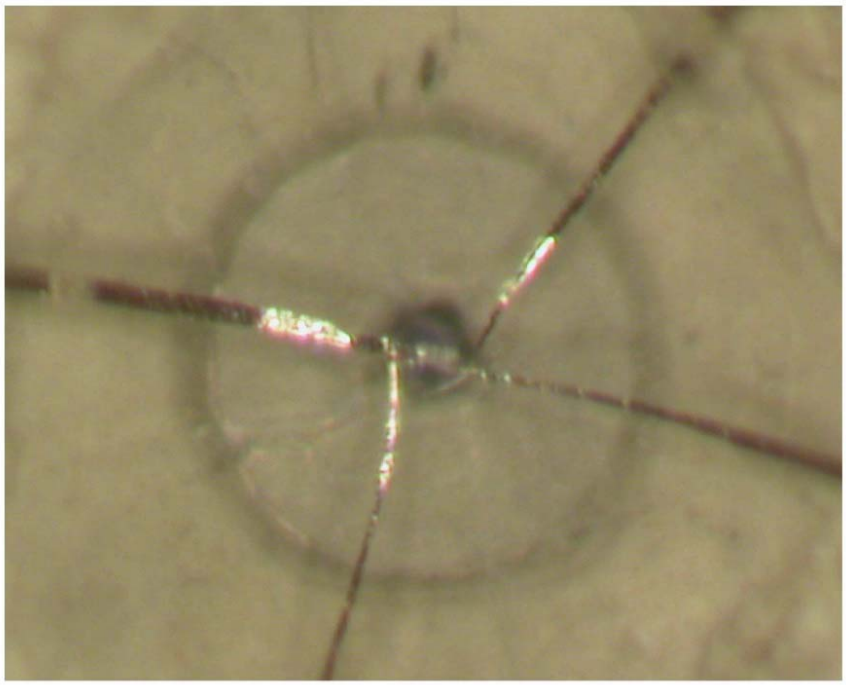

Figure 9.8: After the electrodes were arranged.

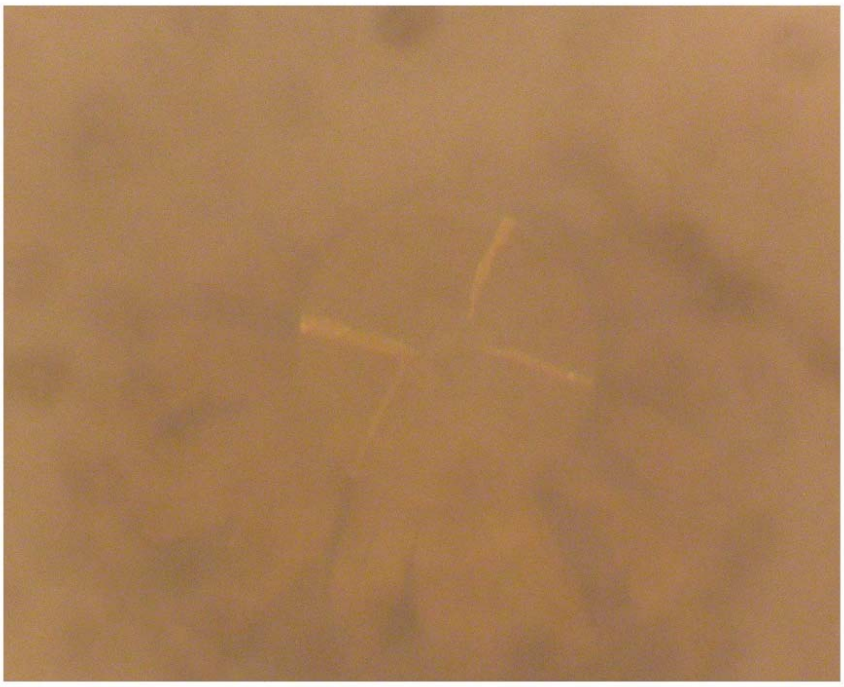

Figure 9.9: Picture taken through the top diamond when it was under pressure.

5 hours. Keep the clamp under pressure for about 30 minutes so that the electrodes take the shape of the diamond facets (see Figure 9.9)

Release the pressure. This experiment is set for a pseudo four point resistivity measurement. The final appearance of the sample chamber and the electrodes can be seen in Figure 9.10.

Put a small amount of crazy glue on the small pieces of tape that hold the electrodes on the gasket. Use 140 μm copper wire for extending the electrodes. Take the insulation out and solder it onto the board and then to the electrode at right angles. At the edge of the board, the copper wires should be taped and glued to the board. Figure 9.11 shows a final view of the clamp.

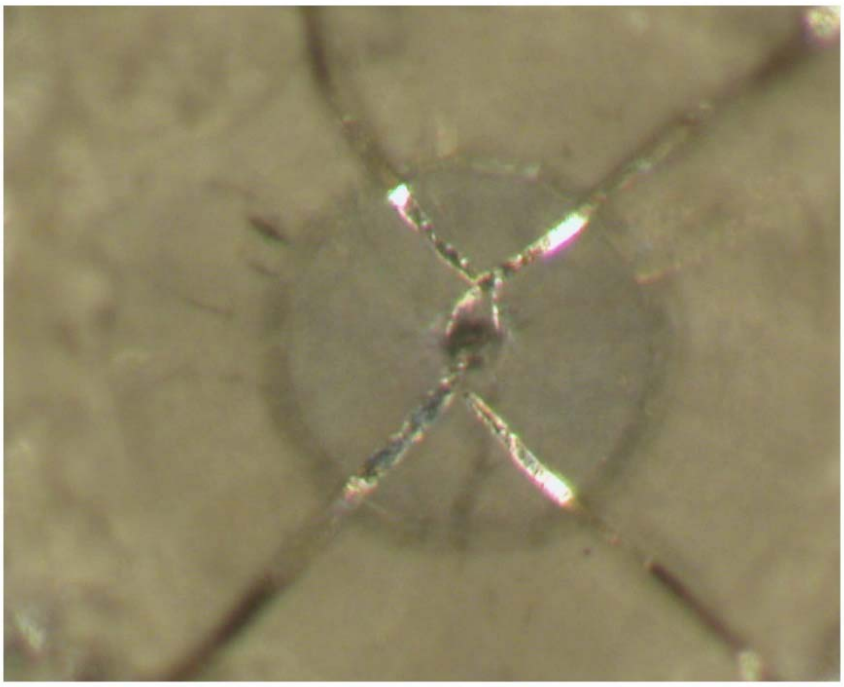

Figure 9.10: Final position of the electrodes over the hole.

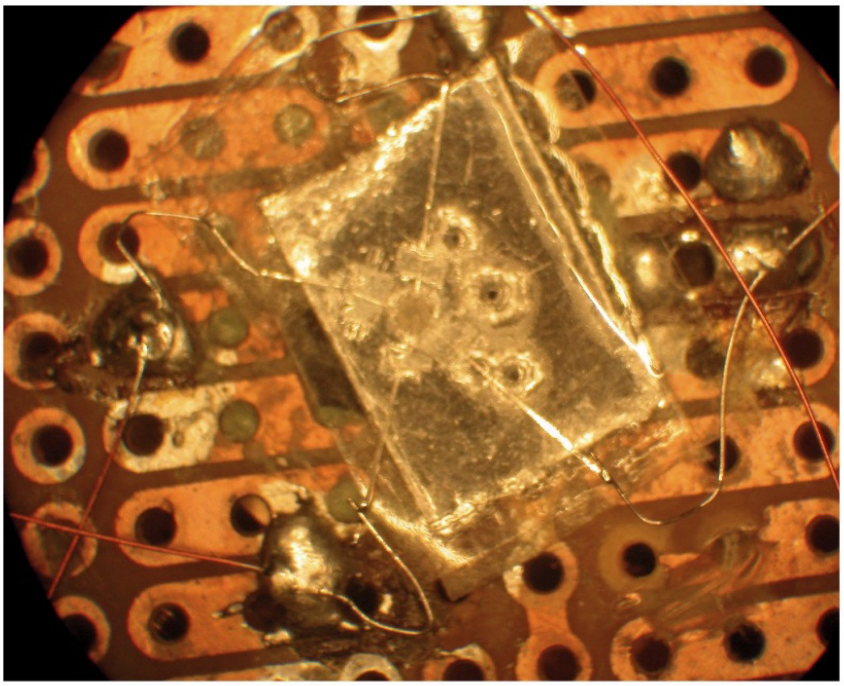

Figure 9.11: Final picture of the clamp.

10. Making an Insulated Gasket

(Takahiro Matsuoka, Feb 2007, 1^{st} edition, small modifications by M. Debessai)

Steps for making an insulated gasket (used for resistivity measurements in the DAC system) are given below.

10.1 Insulating the Gasket

1. Pre-indent a metal gasket and drill a hole. The thickness of the preindentation depends on the culet diameter of the diamond anvil being used. From previous experience, if A is the culet diameter, B is the preindented thickness of the metal gasket, and C is the diameter of the sample chamber (i.e. the hole drilled in the center of the gasket), then $B < \frac{1}{3}\ A$ and $C < \frac{1}{3}\ B$.

   Due to technical problems, for resistance measurements above 20 GPa, highly non-hydrostatic pressure conditions cannot be avoided.

   For instructions on drilling a hole in the gasket, see the EDM manual.

2. Cover the entire gasket with scotch tape and hollow out a region over the preindentation, as seen in Figure 10.1.

   Fill the pre-indent hole with powder insulation material, as seen in Figure 10.2.

3. Pressurize in a clamp to several GPa. After the anvils are released, a the layer is made on the culet surface and on the lateral side of gasket, as seen in Figure 10.3. Put crazy glue on the rim of the hole.

10.2 Making a Sample Chamber

1. *Case I* - Stiff crystal or powder sample with no pressure medium

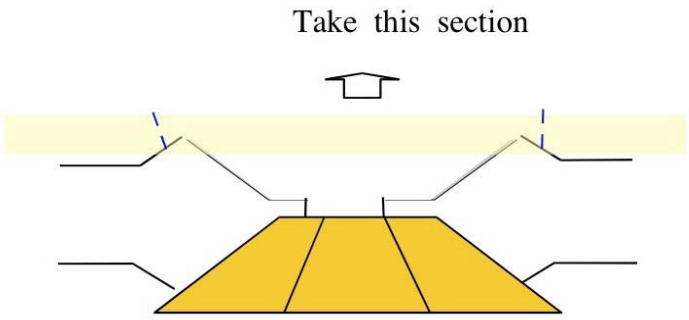

Figure 10.1: Cover the gasket with scotch tape (yellow in figure) and then remove a section above the preindentation.

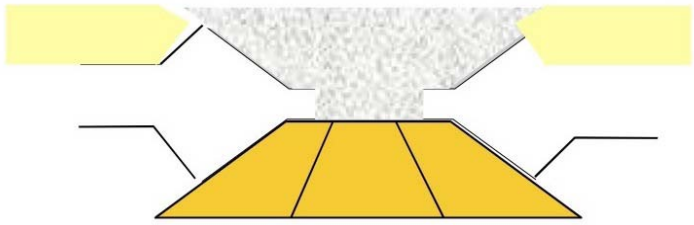

Figure 10.2: Preindentation region filled with an insulating powder.

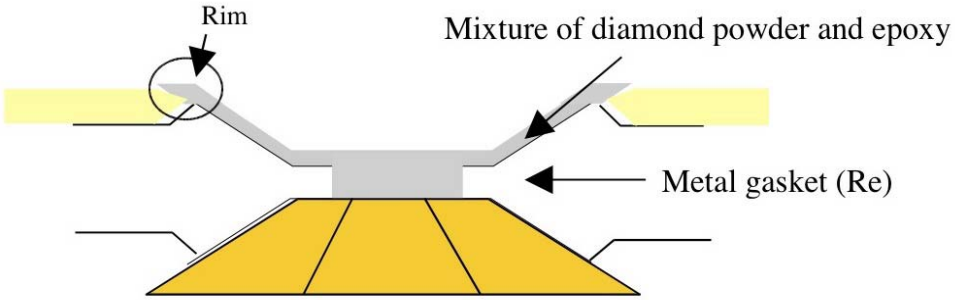

Figure 10.3: After being pressed using the clamp, the powder forms an insulating layer around the gasket.

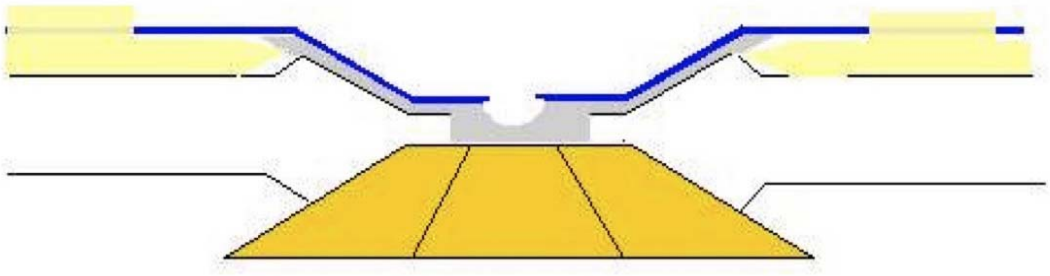

Figure 10.4: A shallow bowl-shaped hole in the insulation material is used for non-hydrostatic measurements. Electrodes are shown in blue.

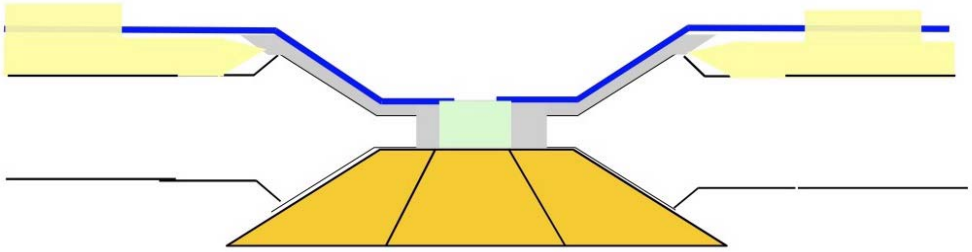

Figure 10.5: For quasi-hydrostatic measurements, a hole a sample chamber is made at the center of the culet. Electrodes are shown in blue. The sample chamber is filled with powder (green).

- Dig a shallow bowel on the insulation without making a hole (see Figure 10.4).
- Put on electrodes

2. *Case II* – For quasi-hydrostatic / less-non hydrostatic pressure medium
   - Make a sample chamber on the center of culet, as seen in Figure 10.5).
   - Fill the sample chamber with powder or NaCl crystal up to 80 - 85 % in volume.

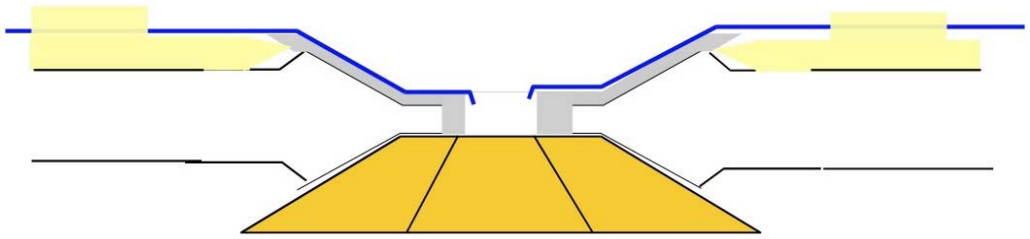

Figure 10.6: Sample chamber when using a soft or fluid sample with no pressure medium. Electrodes are shown in blue.

- Press the anvils together to press / fill NaCl in sample chamber.

  CAUTION: Using too much NaCl powder can be the reason for the NaCl and sample flowing out of the sample chamber during compression.

- Put on electrodes

3. *Case III* - Sample is soft or fluid and no pressure medium is used (see Figure 10.6).

   - Make a sample chamber at the center of the culet
   - Put on electrodes

     CAUTION: When putting on the electrodes, be sure to push the electrodes with the diamond anvil very gently so as not to destroy the sample chamber.

10.3 Making the electrodes

Cut platinum foil, using a shaving lather, on a cutting board which is made of something like circuit board. To obtain the sharp tip of electrodes, you can make small angle as seen in Figure 10.7). TRICK: The shaving lather must be very sharp each time you cut Pt foil, so change blades every 10-15 cuttings.

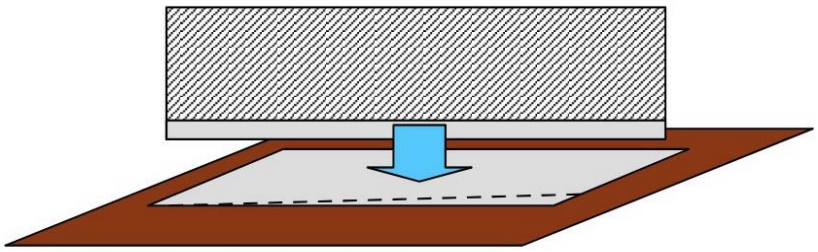

Figure 10.7: Cut electrodes from a piece of platinum foil, making sure to obtain a sharp tip.

10.4 Putting electrodes on the gasket

1. Cut a small (as small as the tip of tweezers), piece of scotch tape.
2. Stick the fore-section of an electrode using the small piece of tape.
3. Fix electrode on the gasket to make the tip directly at the center of culet, as seen in Figures 10.8 and 10.9.
4. Follow same procedure for other 3 electrodes.
5. Gently press electrodes onto the culet using the other diamond anvil, as seen in Figure 10.10. Make sure that the electrodes go to the desired positions when they are pushed onto the gasket. Prepare drawings of desired configuration of sample chamber and electrodes. Push electrodes down to the anvil and see the movement of electrodes. You can predict the configuration of electrodes with current geometry of electrodes and scotch tapes. If the predicted configuration is that which is desired, you can gently push them onto the gasket. If not, you must lift the diamond up and adjust the positions of electrodes by changing the angle and length of the electrodes. Repeat pressing and adjustment of electrodes until the desired configuration is reached. *Be patient!!!* Matsuoka spent a minimum of 3 hours on this procedure.

   After pushing electrodes on the gasket, leave it for several minutes, then lift diamond anvil up. If electrodes don't stay on the gasket, but

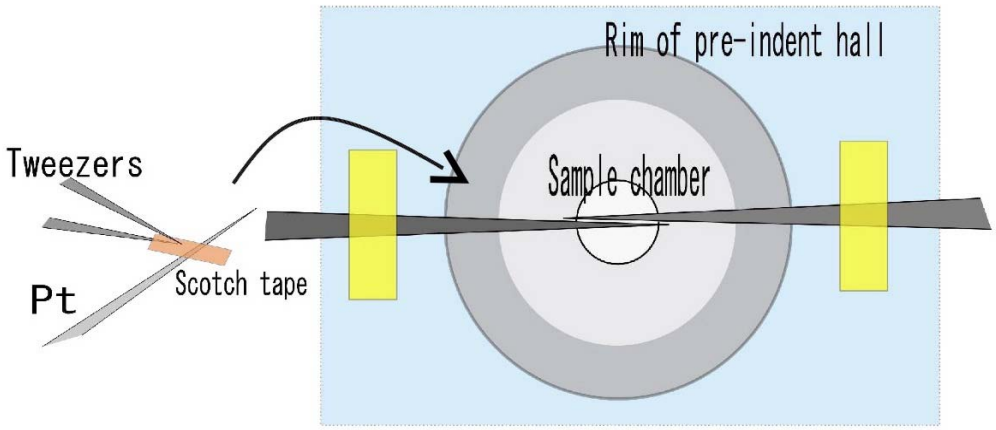

Figure 10.8: Top view of culet and preindented area as electrodes are put in place.

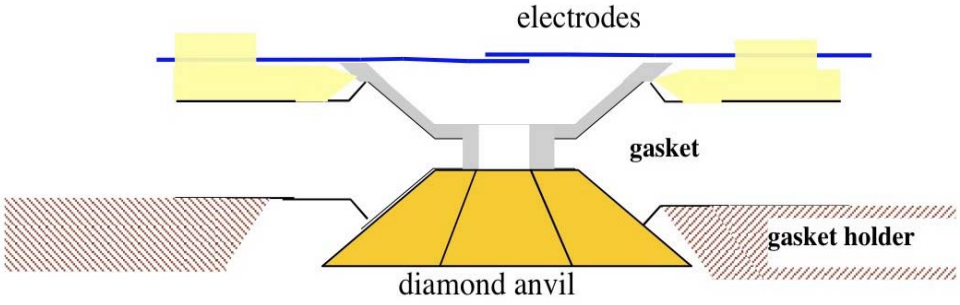

Figure 10.9: Schematic drawing of anvils gasket, gasket holder, and electrodes.

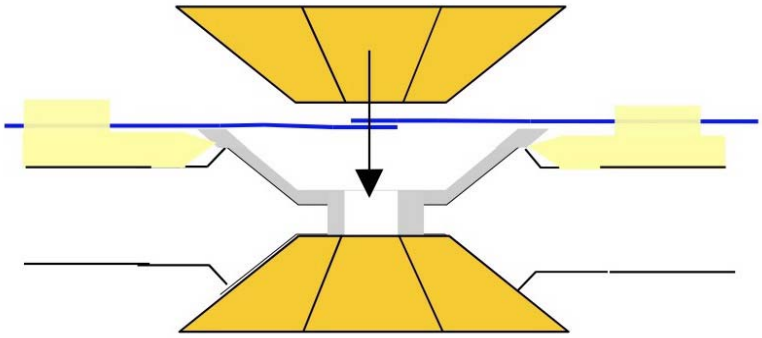

Figure 10.10: Gently press electrodes into place using the other diamond anvil.

rather float above the culet, gently press them down again.

6. Put glue on the scotch tape which fixes electrodes in place (see Figure 10.11).

   IMPORTANT: Never allow electrodes to touch the gasket in the vicinity of the sample chamber.

   Photos of the final configuration of the gasket, electrodes, and wires can be seen in Figures 10.12 and 10.13.

10.5 Making insulation materials for resistance measurements in DAC

10.5.1 Diamond powder + Epoxy glue

1. Mix diamond powder and Epoxy (Stycast 1266, A and B = catalyst are mixed), adding some ethanol as a solution to make mixing easy.

   - Average grain size of diamond powder Takahiro using is 1 μm dry diamond powder.

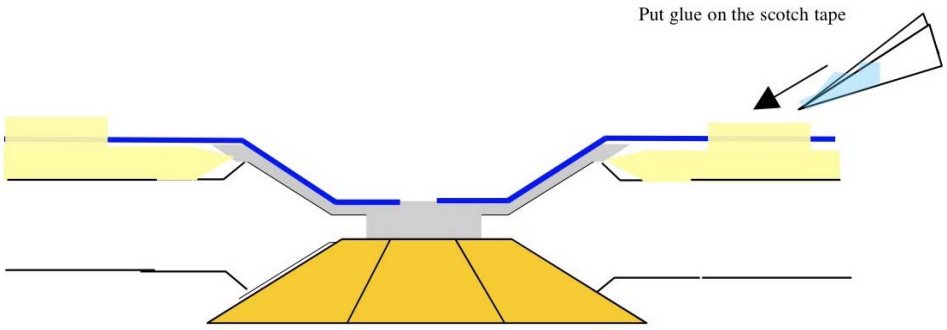

Figure 10.11: Place glue on the scotch tape.

- Diamond powder used for polishing backing materials contains some kind of solution, so it cannot be used ... want to avoid chemical reactions with reactive samples.
- Stycast 2850FT is black and mixed with Al_2O_3. This black stycast might contain some magnetic materials. (I'm not sure)

2. The ratio is diamond powder 4 : epoxy 1 in weight.
3. Wait until the epoxy dries.
4. Slice and grind the mixture to fine powder.

Epoxy glue works as a bonding material for powder. Think of it this way ... epoxy glue coats the diamond powder. By applying force, the epoxy spreads and fills the cavities between the diamond powder, as shown in Figure 10.14.

10.5.2 Other Materials

Other materials such as cBN and Al_2O_3 can be mixed with epoxy using the same procedure.

Al_2O_3 can be used without epoxy.

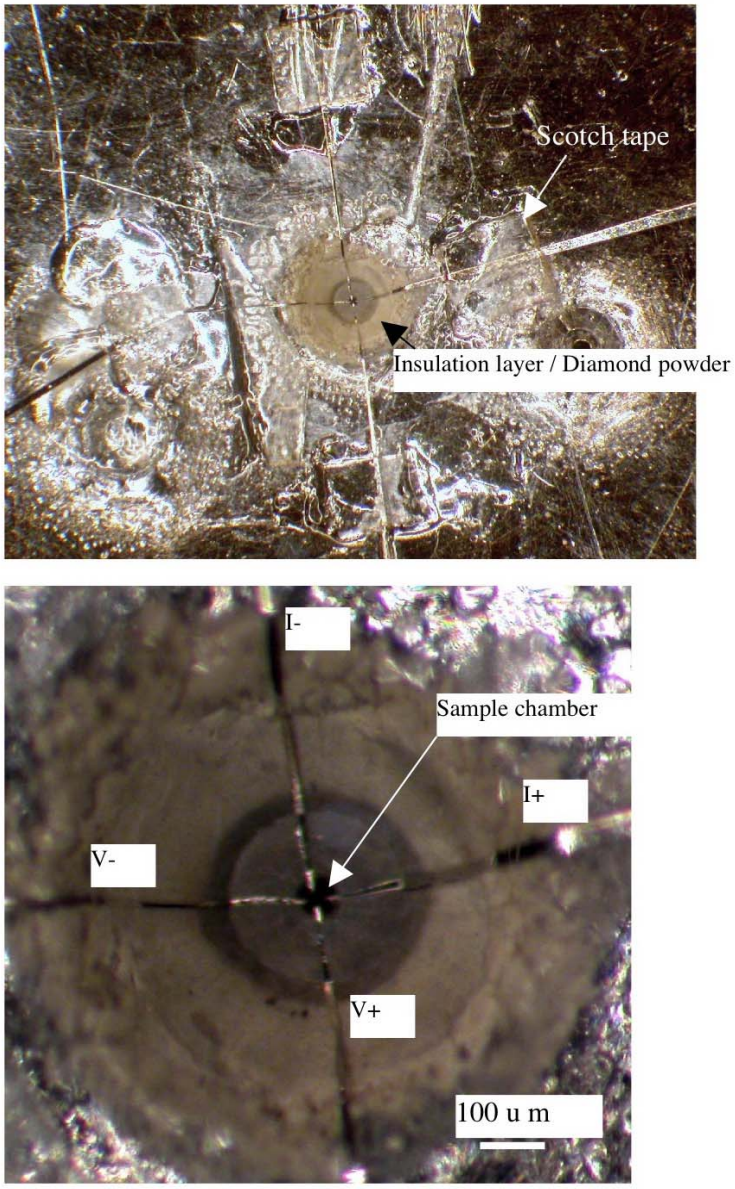

Figure 10.12: Gasket with electrodes in place. Anvil is 300 μm.

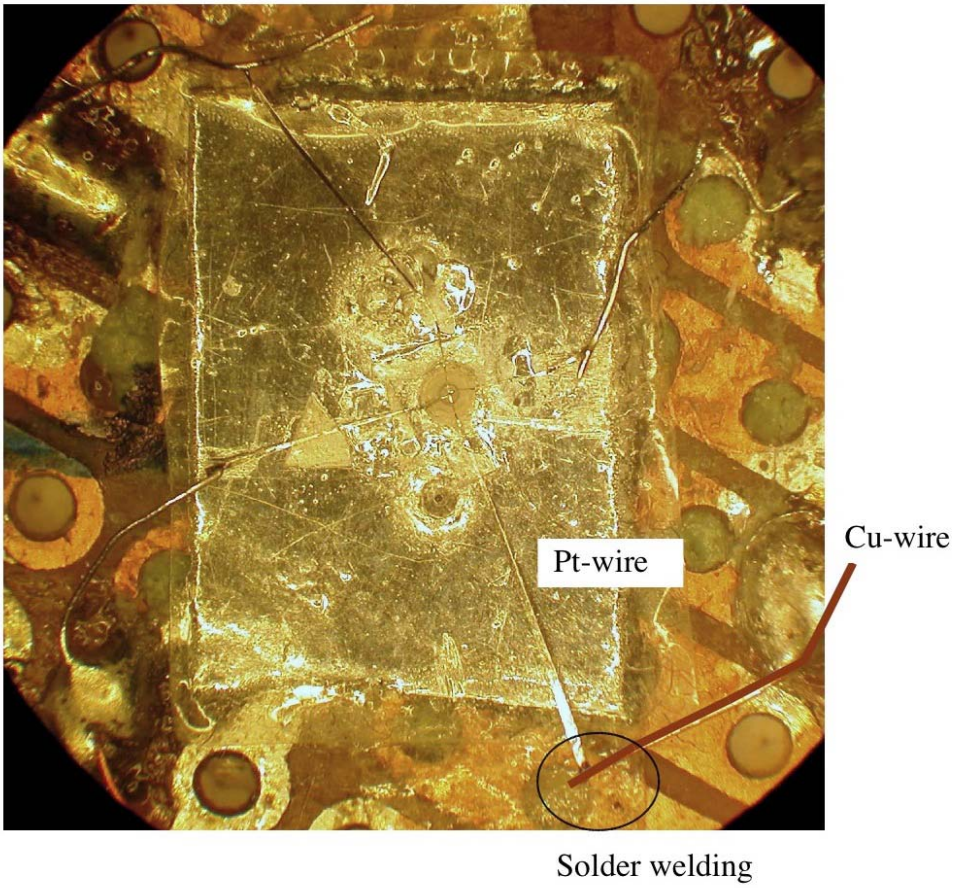

Figure 10.13: Gasket and wires on the gasket holder. The gasket is fixed to the gasket holder using double sided tape.

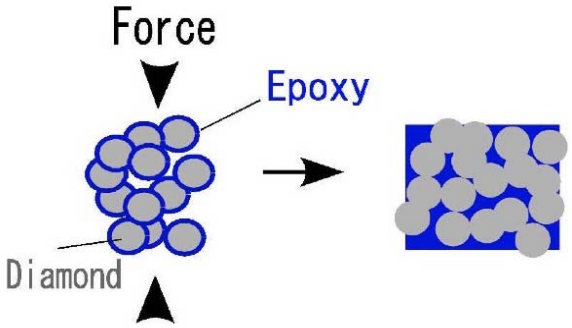

Figure 10.14: When force is applied to the diamond powder and epoxy mixture, the epoxy fills the interstitial regions, thereby acting as a bonding material for the powder.