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Design Construction and Use of a Thermomechanical Detwinning Apparatus: Detwinning of Orthorhombic $\text{Nb}_2\text{BaCu}_2\text{O}_3$

A Thesis Presented for the Masters of Science Degree in
Physics from the University of Tennessee

Chad J. Bircher
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Abstract

In this thesis I will describe a device designed and built for thermomechanically detwinning crystals. Twinning is a process in which a crystalline structure with two different axes of nearly equal length alternate directions throughout the material, effectively generating a single bulk material with two or more distinct internal crystals. This structure defect can lead to many other effects in the crystal, and it is useful to eliminate twinning in many materials in order to determine which properties are fundamental properties of the crystal, and which properties are artifacts of the structural imperfections. The apparatus described in this thesis applies forces as large as 150 lbs along a single axis directions at temperatures as high as 650 C in a controlled gas environment. Under these conditions many crystals will align so the short axis of each unit cell aligns parallel to the axial pressure. This process reduces or eliminates twinning in many crystals leading to clearer interpretations of data from neutron scattering, and other experimental techniques. The initial results on NBCO are encouraging, although not ideal. The NBCO test sample is less twinned than originally, although the detwinning is not complete.

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Part I

Detwinning Process as carried out on NBCO

1 Background information

1.1 Detwinning

Many crystals have structure packing faults commonly referred to as twins or twinning. Twinning occurs when two separate crystals share the same lattice points. A one dimensional example would be the chain $abcb\mathbf{C}bacba$. In this case the first 6 letters are one crystal, while the last 6 letters are another identical to the first, but with a rotation of 180 degrees. In two dimensions there are more options for how the zones align. For NBCO the twinning occurs along the 1,1,0 and 1,-1,0 directions. In crystals that naturally twin, neutron scattering experiments do not give the results of a single crystal, but rather an average across all crystals in the sample. In a sample that twins along two orthogonal axes there is a total of up to 4 different crystals, as shown in figure (1).

In Figure 2, the four boxes are commonly referred to as twin zones. The lines between the boxes are referred to as twin boundaries, and the directions of the boundaries are called the twin axes. If twinning is reduced to one axis there are only two crystals, and if it is removed altogether the experiment is reduced to a single crystal. Three dimensional twinning is much more complicated, and beyond the scope of this paper. This paper focuses on detwinning NBCO, which only twins in two dimensions.

In YBCO and NBCO incommensurate scattering yields four symmetric peaks around the $\pi\pi$ position[1]. The twinned structure of YBCO and NBCO complicates the interpretation of the data. In YBCO, however, a detwinned sample clarified the data, and gives a little more evidence for stripes. NBCO is the closest relative to YBCO, but it has never been detwinned in a bulk sample large enough to measure magnetic flux pinning, or other useful neutron scat-

a	b	c	a	c	b	a	c
b	c	a	b	a	c	b	a
c	a	b	c	b	a	c	b
a	b	c	a	c	b	a	c
c	a	b	c	b	a	c	b
b	c	a	b	a	c	b	a
a	b	c	a	c	b	a	c
c	a	b	c	b	a	c	b

Figure 1: Two Dimensional twinning example



Figure 2: Stress Picture by Transmission Polarization

tering experiments. With a detwinned NBCO sample it might be possible to strengthen the stripe argument presented by Mook, or some other picture might be presented.

NBCO is a single example of a crystal that has never undergone neutron scattering after twinning, but several others exist as well. This paper will specifically discuss efforts to detwinn NBCO in the newly developed detwinning apparatus I designed, built and described in detail in Part II. However, the methods are relevant to many other bulk samples as well.

For further discussion of twinning check out Ashcroft, Kittell and Wikipedia [2, 3, 4].

1.2 Polarizing microscope

A typical first year undergraduate demonstration of polarizers shows that one polarizer cuts incoherent light intensity in half, and can remove glare due to reflection. If an additional polarizer is aligned with its polarization axis parallel to the first polarizer, nothing changes. If the two axes of polarization are perpendicular the transmitted light drops to near zero. If the two polarization axes are perpendicular and a third polarizer is placed between them, some light comes through depending on the direction of the center polarizer according to the equation $T = (I/8) * \sin(2\theta)$ where I is the original intensity of the light, and θ is the angle of the polarization axis with respect to either of the orthogonal polarizers. If a transparent object is placed between two polarizers, internal stress visualizes, due to the polarizing nature of the stress lines[5], as shown in Figure (2) from the department of agriculture [6].

This setup is similar, in that it uses two polarizers, although instead of transmitting through a transparent surface, the light is reflected off the crystal. A crystal is identical everywhere, and so we would expect very little information from a stress picture of the surface of an ideal crystal. However, twinned crystals are not ideal crystals. Twinned samples contain two orthogonal crystals

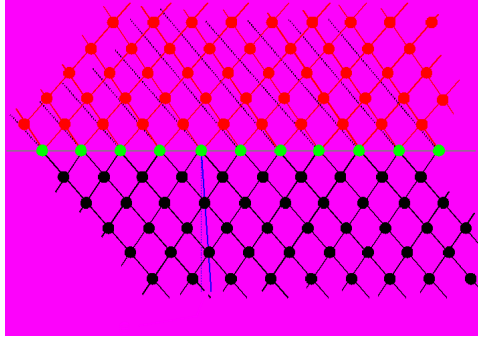


Figure 3: Orthorhombic Twinning in two dimensions along 110 direction

throughout the bulk sample, and if the crystal has a slightly different stress in one direction twinned samples begin to have a polarized structure. In NBCO the a and b translation vectors are slightly different. Specifically, $a=0.3916(1)$ nm and $b=0.3858(1)$ nm in bulk NBCO[7]. Since there is only a 2% difference between the axes the crystal can twin relatively easily. The difference in the axis lengths gives rise to a stress at the twin boundaries, which then allows viewing of the twin zones through a polarizing microscope. Figure (3) is an example of twinning along the 110 direction in an orthorhombic crystal, identical to the situation in NBCO. The black dashed lines are where we would expect the crystal axes to lie if the sample were not twinned.

The stress picture depends greatly on the angles between the polarizer, the analyzer, and the axes of the crystal. If the crystal rotates with respect to the polarizers the stress picture reduces, and completely disappears at some angles. This shows that the stress induced by twinning exists only along two axes. If the stress existed along other axes as well, the stress picture would always show some amount of twinning.

The quality of the picture also depends on the surface condition of the crystal. When the twin zones are far apart and the stress levels are high, the surface only needs to be somewhat polished. In NBCO however, the surface must be highly reflective in order to have any stress picture show up in the microscope. Imperfections in the surface condition scatter the reflection, and blur the zone boundaries significantly.

2 NBCO Characteristics

2.1 Superconducting Properties

The NBCO used in this experiment was under doped initially. The doping placed a fairly sharp transition temperature at approximately 35 C. The dewinner designed for this work controls both the gaseous environment, and the temperature, so the conditions can remain the same as during the annealing

process, leaving the transition temperature the same before and after detwinning.

The T_c of NBCO can reach as high as 95 K when properly annealed [8]. These samples were annealed to the lowest T_c possible for future experiments. The transition temperature is clear in Figure (4).

2.2 Twinning structure

NBCO twins along the (110) and (1-10) directions [9] similar to its closest relative YBCO [10]. The twin domains of YBCO are spaced around 20 μm apart, and move relatively easily [11]. The mobility of the twin zones allowed Segawa to detwin bulk YBCO at temperatures below 220 C and 100 MPa [12]. The twin zones in the NBCO are much more compact, generally reaching densities of a boundary every 0.5 to 2 μm [13]. Pradhan has thermomechanically detwinned a small sample of NBCO previously, but his detwinning took place between 400-600C, much higher than needed for YBCO, and very close to the tetragonal-orthorhombic transition temperature of between 620 and 660C as reported by Oka [14]. However, Pradhan cut a small piece off of a larger bulk crystal, and the small section had twinning only in one direction. This simplified the detwinning process. The doubly twinned structure is clear in Figure (5).

Since there is no information on the thermomechanical detwinning of doubly twinned NBCO, the best starting points were the thermomechanical detwinning of doubly twinned YBCO, and of singly twinned NBCO. Summarily, neither conditions yielded the desired results. We have only one trial performed by Dr. Shiliang Li where the crystal detwinned, but that trial is undocumented.

3 Procedures

3.1 Difficulties

Many difficulties hampered the process of detwinning large single crystals. This section addresses the most stubborn obstacles that presented themselves during the process. Included are the treatment of wax inside the system, inexplicable surface changes, distortions due to heating, and an environmental problem that turned out not to be a problem.

3.1.1 Waxy deposits

Every sample must be flat in order to detwin properly. The flattening process involves attaching the sample to a flat metal mount with hot wax, and polishing on diamond paper both surfaces are parallel to the plane of the metal plate. Heat applied during detwinning melts and boils off any wax left on the sample. While the boiled wax does not interfere with the thermomechanical detwinning process, the wax does stick to any cool surface, including the view port. As wax builds up on the view port, it blurs the image to a point where the sample's twinning structure is no longer resolvable. The wax is transparent when thin

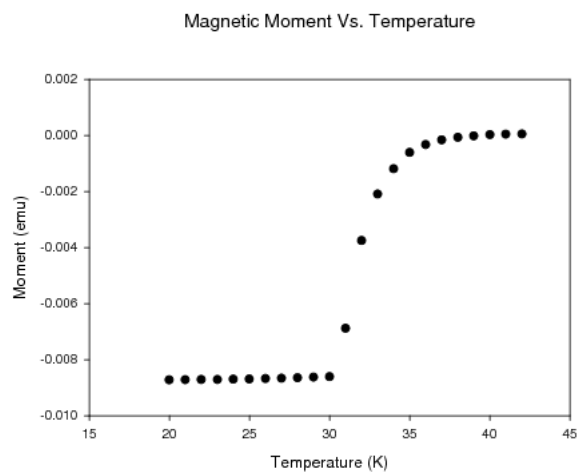


Figure 4: Initial superconducting transition temperature for NBCO

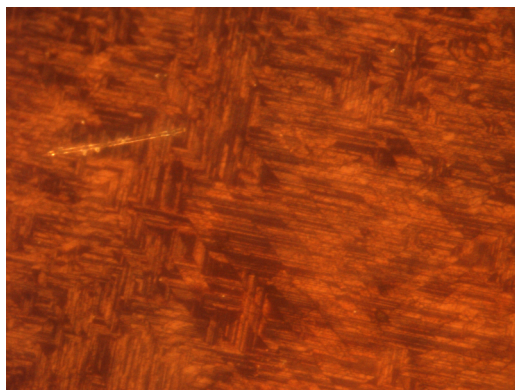


Figure 5: Initial Twinned structure of NBCO

enough, so even though it dissolves in acetone, it is difficult to tell when all the wax has been removed. Also, when placed in the ultrasonic bath for long enough parts of the sample start flaking off, so there must be some median between cleaning off the wax, and leaving the sample in tact. In the end the wax ends up not being a significant problem due to other viewing constraints.

3.1.2 Surface changes

Next, every condition I tried to detwin under led to the surface changing phases. The reason for these surface changes is unclear, specifically the link between the change in surface structure and the detwinning apparatus. When another oven provides the heat source the surface does not undergo any noticeable changes. Admittedly the sample does have a chance to cool down before viewing under the microscope again, so the change might occur, but change back upon cooling. Only the surfaces exposed to the gaseous environment change, the bottom of the sample, and the internal structures are unchanged. One initial possibility was that the insulation might be ejecting some substance that is changing the doping of the sample. However, even with all the insulation removed the same effect occurred, so it was not likely the insulation. Figure (6) is an example of a highly polished twinned surface once the surface structure changed. Once the crystal is repolished the twinning structure reappears.

The surface structure change means that the camera cannot observe the twinning ratio of the sample in real time. Ideally the microscope would record the the migration and disappearance of twin domains; however, by the time the twin zones would disappear due to detwinning, the entire surface has lost zones by switching to an orthorhombic structure rather than the tetragonal structure. So, the entire crystal must be removed from the detwinner, the surface repolished, and then placed under the polarizing microscope again. The biggest drawback of that process is that it is impossible to compare the same area before and after the detwinning process. Given the heat distortions in the

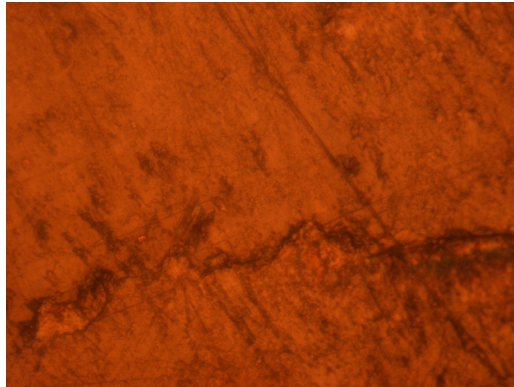


Figure 6: Untwinned Surface after warming

glasses used due to heating, the surface effects were unimportant.

Ideally we would watch the crystal as it heats up, and observe the actual transition. Then we would cool down, and watch the twin zones reappear. Finally we would heat up, apply pressure, watch the crystal detwin, and cool down again under pressure, and notice that twinning does not take place. Then the pictures could be played together to make a movie. However, with the changes in the surface structure this is impossible.

3.1.3 Heat Distortions of Glass

As the sample heats up, any nearby glass heats up as well. Hotter glass somehow distorts the twinning of NBCO, although non-polarized effects remain just as clear. The effect takes place on both the view port, and on the glass of the microscope objective. Adding a cooling tube around the microscope objective has reduced this problem significantly. Also, Shiliang Li noticed that the problem reduces further if the glass on the viewport is aligned in a certain direction with respect to the microscope objective. This means the glass of the viewport and the twin axes of the crystal must both be aligned to have the best possible picture.

3.1.4 Environment concerns

When initially checking the ability of the cartridge heaters to heat the molybdenum, the Mo turned brown, smoked, gave off a putrid odor, and practically fused together. All literature available from the manufacturing company suggested Mo was fairly inert and did not react with oxygen until very hot, and does not react with nitrogen at all. I assumed the Mo must have been interacting with something else in the atmosphere, and so all experiments took place in a closed environment after purging normal air from the chamber. However, I eventually postulated that it might not have been the Mo at all, but something on the surface of the Mo that had reacted. Frequently oils and lubricants are used when machining metals in order to make the process of machining easier on both the machined parts and the machining tools. Without a sample I slowly began to heat up the press in ordinary air. The stage did not react with the air in any noticeable way, and upon opening the sealed top it still did not interact with the atmosphere when heated. It was indeed some sort of machining oil that had smoked upon the initial heating.

3.1.5 Sample destruction

Several of the samples lost significant flakes of material after heating and pressing. Eventually Shiliang Li noticed that there was a small (approximately 100 micron) lip on the sample stage where the sample presses against the Mo wall. I machined this area flat by removing 0.005" from the top of the stage, and running the cut 0.01" into the pressing wall. The press tends to break crystals much less now. Additionally, if the pressure and temperature are both raised

very slowly the cracking is reduced. I find raising the pressure to full value over 5 minutes, and raising the temperature no faster than 5 degrees per minute reduces the crystal destruction as far as it can be reduced.

3.2 Initial trials

The detwinning process began by trying to detwin along the 110 direction, the natural twinning axis. In order to detwin along that direction I cut a sample parallel to one of the twin axes. Next I tried detwinning at pressures from 5 to 30 psi in both oxygen and nitrogen environments. Although we did not run any experiments on that sample, to the eye it was obviously still twinned. We tried to detwin this sample at temperatures up to 220 C. As we ran at higher temperatures and pressures along other directions we omitted this sample, since it was small, unreliable, and detwinning in general must take place along a crystal axis rather than a twinning axis.

Next we tried detwinning along one of the crystal growth axes. Since the crystal naturally grows along the a and b axes, minimal cutting was needed. One of the samples was heated to 180C and detwinned at 25 psi overnight in a closed oxygen environment. For this sample the detwinning did show up to the eye, that is there was still twinning, but it looked to be less abundant. Neutron scattering showed that this sample is fully twinned. The other sample I heated to 220C and pressed at 40 psi for three days in pure nitrogen. On this sample the pressure equated to approximately 800 MPa. In this sample the twinning disappeared completely, and does not show back up upon polishing the surface. However, the neutron scattering data was not as clear on this sample. It appears that the oxygen deficient environment allowed some of the oxygen to leak out, leading to a structure change. Possibly the twinning disappeared not because the crystal structure is actually detwinned, but because a change in the crystal has lead to indistinguishable a and b axes. This hypothesis stands in stark contrast to the information given informally by Rodney Hunt of ORNL which suggests that an oxygen deficient environment is preferred at these temperatures. A summary of the results follows in Figure (7).

Figure (7) shows that the crystal pressed overnight had no noticeable change from the twinned crystal. The crystal pressed for 3 days did have a change, but the structure changed noticeably as well. Finally I pressed the piece at 40 psi and 575 Celsius for 3 hours. This lead to appearances very similar to those of the piece pressed at 220 degrees for three days. However, the crystal was determined to still be twinned by Dr. Shiliang Li.

3.3 Results

In addition to the few attempts described above, Dr. Shiliang Li and I have tried to detwin under many other conditions. These include varying the temperature, pressure, size of the crystal, gaseous environment, and amount of time the crystal is pressed. So far we have only managed to detwin a single small flake, but that particular experiment was not recorded. The approximate pressure was several

GPa on the flake, and at a temperature of 270C. No other papers seem to suggest such a high pressure being needed, so the detwinning might work at a lower pressure.

4 Acknowledgments

I would like to thank my adviser Dr. Pengcheng Dai for all his help with this project. I would also like to thank Dr. Shiliang Li for his work with the NBCO crystals.

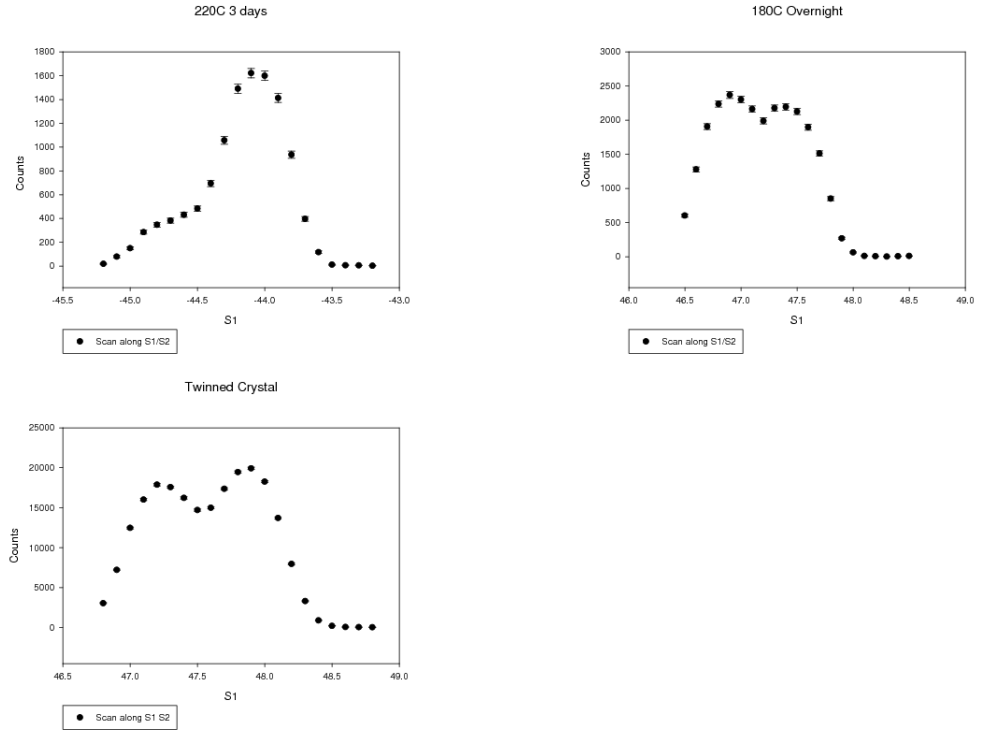
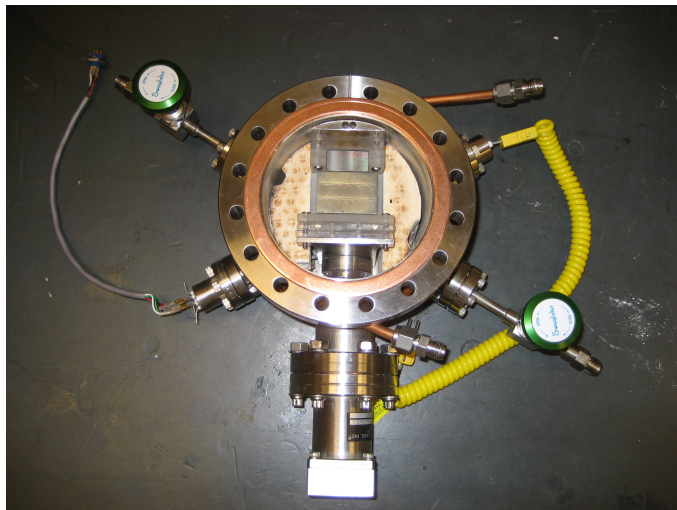


Figure 7: Neutron scattering results for two test crystals and one control crystal

Part II

Detwinning Apparatus Manual



5 Introduction

Many crystals have two nearly, but not quite, identical axes. For example, Hendrixson found his YBCO sample to have axis lengths of $a=3.8187(14)$, $b=3.8692(14)$, and $c=11.620(21)$, a 1.3% difference between the a and b axes[15]. As the crystal grows the crystal might have the “long” a axis equally distributed in two perpendicular directions. As crystals are studied through scattering techniques there are some effects that may arise due to the “twinning” of the two axes. Some effects are ion channel splitting [16], incoherent scattering, alterations of the Laue symmetry [17], and a strong correlation between magnetic structure and twin structure [18]. The twinning effects make bulk properties difficult to analyze, since it is frequently difficult to determine if a property is a fundamental property of the crystal, or an effect of twinning. Two methods are available to eliminate the effects of twinning:

1. Cut the crystal into areas the same size as the twin zones. The pieces can then be coaligned, and the result is a sample where all a axes are perpendicular to all b axes.
2. Thermomechanically detwin the crystal. That is, apply heat and pressure until the crystal coaligns on its own.

Both methods have their drawbacks. It is often difficult to find a naturally detwinned area large enough to cut. While it might be possible to cut a piece large

enough for x-ray scattering, or SQUID measurement, usually it is infeasible to cut and align enough pieces for neutron scattering. Similarly, it is extremely difficult to thermomechanically detwin a crystal as well. The pressures needed can exceed 100 MPa, and the temperatures can exceed 600 Celsius. The stress can break the crystal, or cause stress fractures that break later. Also, due to the high pressures needed, thermomechanical detwinning is usually only possible on small samples. For instance, a sample with a one square centimeter cross-section needing 100 MPa of pressure would require over a ton of force (2248 lbs) applied along the axial direction in order to detwin.

This manual describes a device that thermomechanically detwins bulk crystals that can be detwinned with under 120 lbs of uniaxial force. This includes large crystals that can be detwinned at low pressures, and smaller crystals that need very high pressures. This setup requires 16.1 psi of inlet pressure to reach 100 MPa on a 1 square millimeter sample, and has an upper limit of 120 psi inlet pressure. The largest crystal needing 100 MPa that this apparatus can detwin is 7.5 mm².

Finally, the temperatures required for detwinning overlap with the annealing temperatures in many cases. In order to detwin, and still keep the same doping properties, the detwinning process must have an atmospheric environment identical to the environment used for annealing. Conceptually the process is fairly simple, heat the sample, apply pressure to a single axis, wait, and then cool down in a controlled atmosphere. This manual describes how to build a mechanism that does just that.

6 Detwinning Component Descriptions & Schematics

6.1 Inside box

The box is composed of the tubing and flanges, and the central “chair.” The term chair is appropriate since the piece looks much like a classic 4 leg chair without seat or back cushions, and without a cross brace where the seat meets the back. The chair supports the stage where the detwinning takes place, and has as much empty space as possible. The empty space reduces the heat transfer between the sample stage and the external environment. The chair is welded to both the bottom and the sides of the tube. It is exactly opposite the pneumatic press, and the “seat” is higher than the feedthrough flanges.

The tube is four inches in inner diameter, one eighth inch thick, and is welded to a six inch flange. A plate welded to the bottom of the tube seals the environment. The tube has five additional holes cut in the sides, one for the pneumatic press (1.5” diameter), and four (0.75” diameter) for the other four feedthroughs. Four tubes of 0.75” outer diameter and 1.5” length are welded to 1.33” blanks, and a single tube of 1.5” diameter, and 2.5” length (lengths are measured at the top or bottom of the tube) is welded to a blank 2.75” flange, and all of these are welded to the main tube. The tube flanges and chair are all

stainless steel so that they are easy to weld, and structurally strong.

The holes in the chair are all tapped for #5/40 screws. This is not a common size, but is a good balance between size and strength.

The flanges came from Norcal and at the time of purchase cost \$63.00 for the 6" flange, \$12.60 for the 2.75" flange and \$10.80 for each of the 1.33" flanges for a total of \$118.80. The stainless steel for the chair and tubing was available at the university, so the price for those parts is not readily available.

6.1.1 Frontal View

Figure (8) is a diagram of the inside of the detwinning apparatus from the view of the pneumatic press detailed in 6.7. This figure is a diagram of the welded parts as described in detail in 9.1. Table (1) is the color guide of parts for all of the non electronic parts inside the detwinner, with the exception of the insulation.

6.1.2 Top down view

Figure (9) is a top-down diagram of the central support, the main housing tube, and all feedthrough tubes. This figure is a diagram of the welded parts as described in detail in 9.1.

6.1.3 Side view

Figure (10) is a side view of the central support, and main housing tube. This figure is a diagram of the welded parts as described in detail in 9.1.

6.2 Viewport

Figure (11) is a viewport available as item DN160CF from Lewvac, and is \$575 at the time of writing this manual. There is a limit on the space allowed between the surface of the stage and the top of the viewport. I chose this flange because the glass is flush with the bottom of the port, rather than flush with the top. This geometry allows the microscope to drop closer to the sample stage than if the glass were flush with the air side of the flange. If this piece ever has to be replaced the top of the glass can be no higher than 8mm above the copper gasket between the viewport and the main. A=135mm B=8mm C=12mm

6.3 Stage & Slide

The stage and slide drawn in Figure (12) are both machined Molybdenum blocks. Molybdenum the best choice for this part because of its incredibly high thermal conductivity, and its very low coefficient of thermal expansion. It does have a drawback of oxidizing at high temperatures in oxygen rich environments, and is a very brittle and fairly weak metal. There are some chromium compounds that might work almost as well, especially since chromium is an easier metal to machine, and does not oxidize. If the Mo breaks, or if oxidation is a problem chromium is a good substitute.

Table 1: Color Guide

Color	Part
Light Green	Slide
Orange	Adapter
Brown	Heat Shields
Black	Quartz
Dark Blue	Base Plate
Cyan	Flanges
Green	Tubing
Gray	Insulation

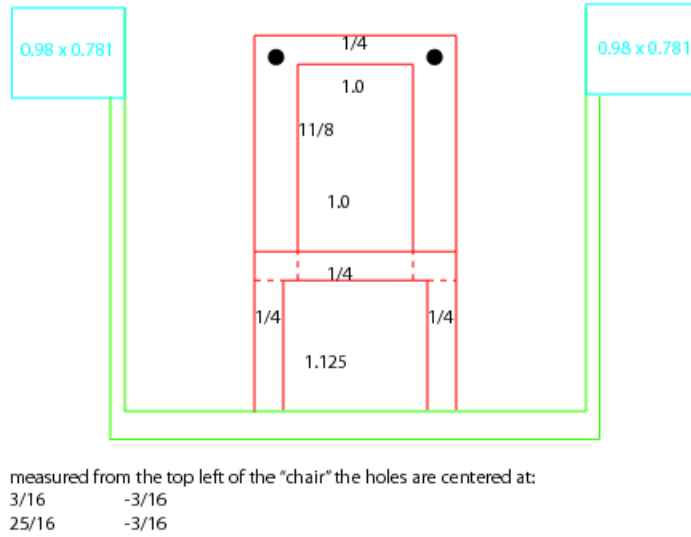


Figure 8: Direct view of the central support and main housing tube

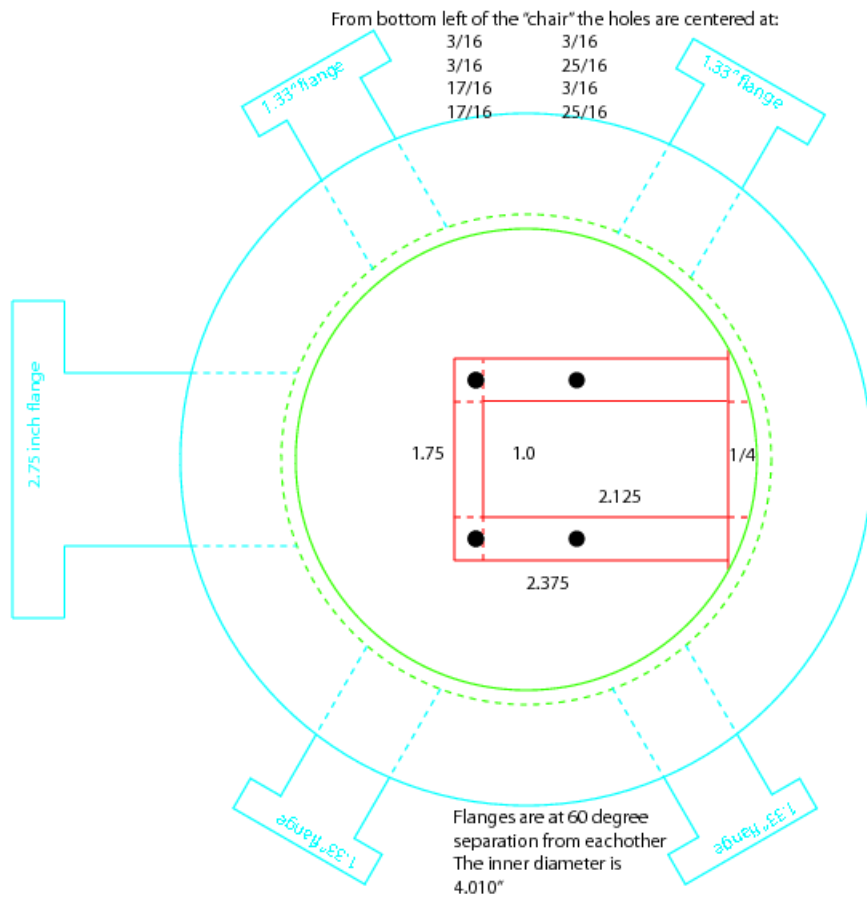


Figure 9: Top down view of the central support and all tubes

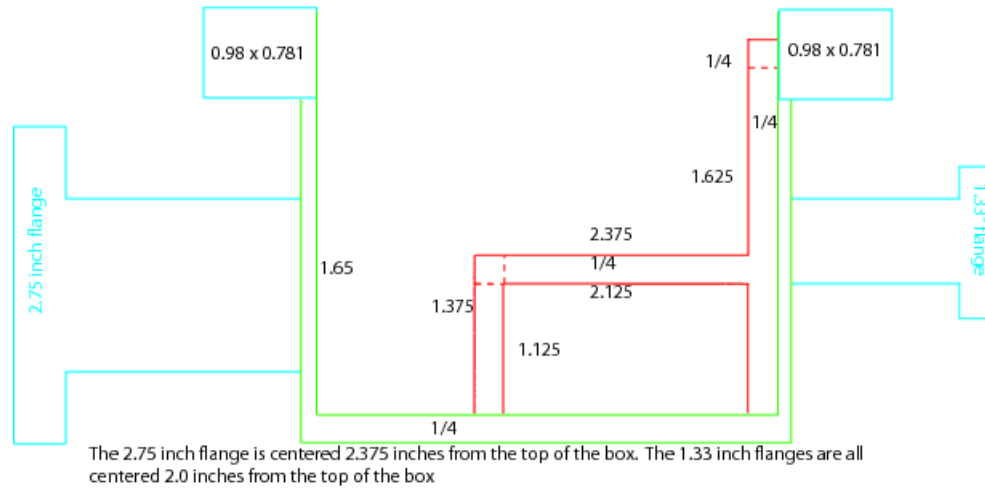


Figure 10: Side view of the central support and housing tube

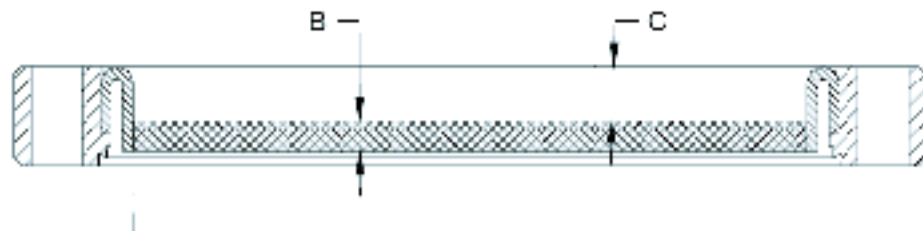
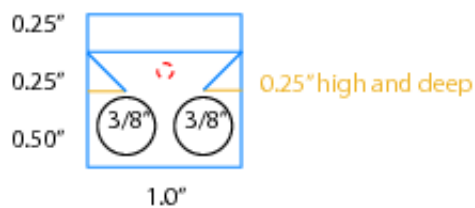
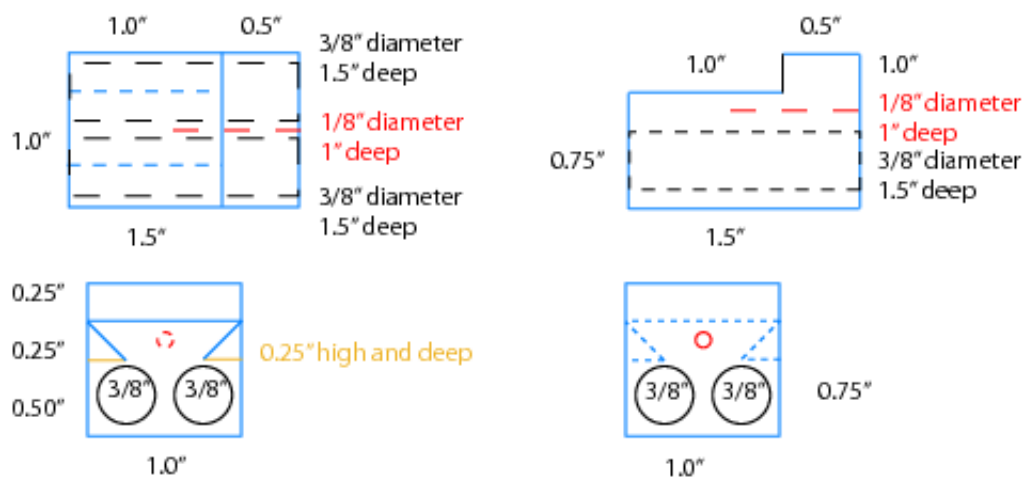


Figure 11: Viewport

Sample Stage: Cut From
One piece of Mo 1.5 x 1.0 x 1.0



Slide: Cut from
One piece of Mo 1 x 1.75 x 0.5

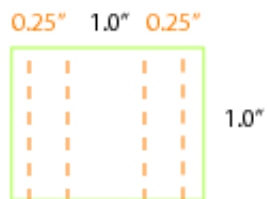
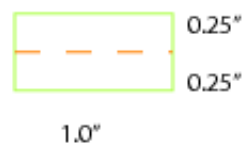


Figure 12: Sample Stage and Pressing Slide

The Mo is from National Electronics alloys and costs \$300 as the figures are drawn, but an additional blank for a slide costs only \$5 more.

6.4 Press Adapter & Heat shields

The pneumatic press cannot mount in a way where the press is in direct contact with the slide due to geometry constraints. The adapter drawn in Figure (13) attaches to the press, and extends vertically to transfer the force of the press to the slide. The adapter is stainless steel because it must have considerable structural integrity to handle the torque given at the highest possible pressures for the pneumatic press (120psi).

The heat shields in Figure (13) reflect much of the radiated heat back at the sample stage to reduce the amount of heat lost to radiation, and keep the sample at a more uniform temperature. The shields are 0.02" thick, and made of Mo since they must maintain very high temperatures without sagging or emitting impurities. The holes in the Mo plates allow viewing of the sample through a microscope. Samples are not all the same size, so it is ideal to have several pairs of viewing plates with different sized holes cut out. With an extraordinary amount of care in shaping the insulation the detwinner might house three levels of heat shields, but two is sufficient. The lower heat shield is trimmed slightly so it fits in a groove notched into the insulation. In order to hold three heat shields the insulator must have an additional notch. However, if the heat shields touch, their functionality drops dramatically.

The heat shields come from National Electronic alloys for \$150 for a set of 10. This allows a total of five pairs of heat shields with different size holes.

6.5 Quartz Insulation

Quartz and Alumina are the best hard solid insulators above 600 C. Many insulators work at high temperatures, however most are too soft for accurate machining. In order to properly detwinn crystals the pressure on a crystal must be uniform. This means the sample stage must be flat, and to reduce heat loss the sample stage should have as little thermal contact with the any metals attached to the tube as possible. These two requirements direct the construction of a box where the sample stage is mounted to a hard insulator which is in turn mounted to the box. Many of the soft insulators that are readily available are not hard enough to provide a consistantly flat surface. Many of the hard insulators are much better at lower temperatures, but lose thermal resistivity or structural integrity at high temperatures. Many of the ceramic insulators are not machinable, and are unusable for this application. Each quartz plate is less than 0.25 inches thick due to price considerations from the supplier. Thicker plates are available, but the price increases dramatically. The thin quartz has a tendency to crack, so the thicker quartz is probably worth the price difference.

The quartz pictured in Figures 14-18 comes from G.M Associates, and the price varies from order to order. The parts in the detwinner cost approximately \$580.

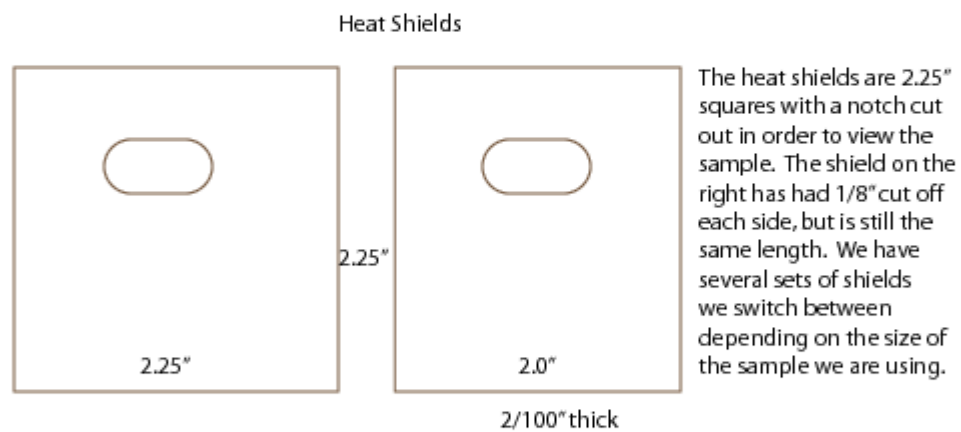
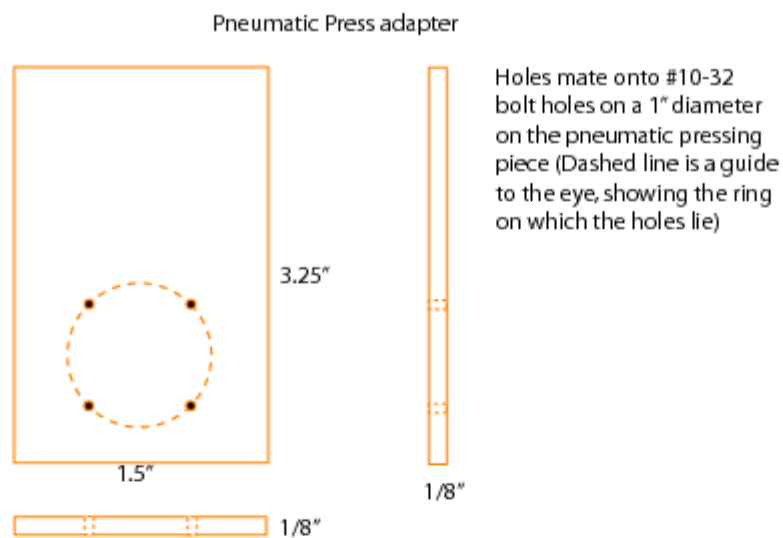


Figure 13: Press Adapter and Heat Shields

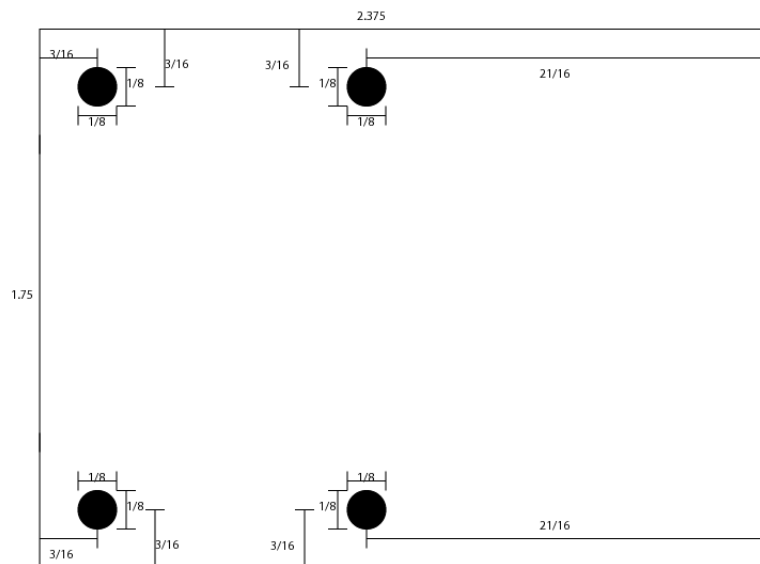


Figure 14: Base Plates (2plates, $\frac{1}{4}$ " thick each)

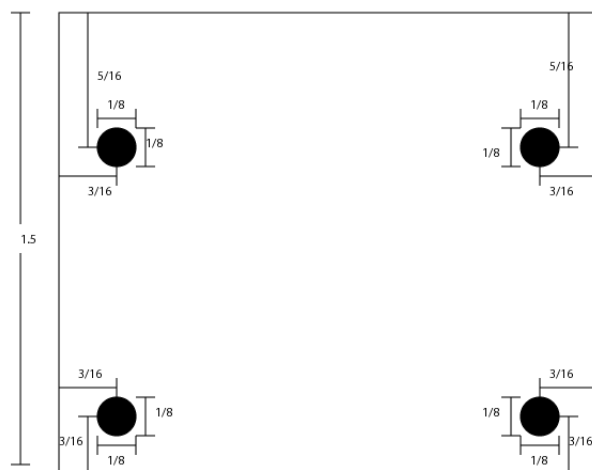


Figure 15: Base Plates (2 plates, $\frac{3}{16}$ " thick each)

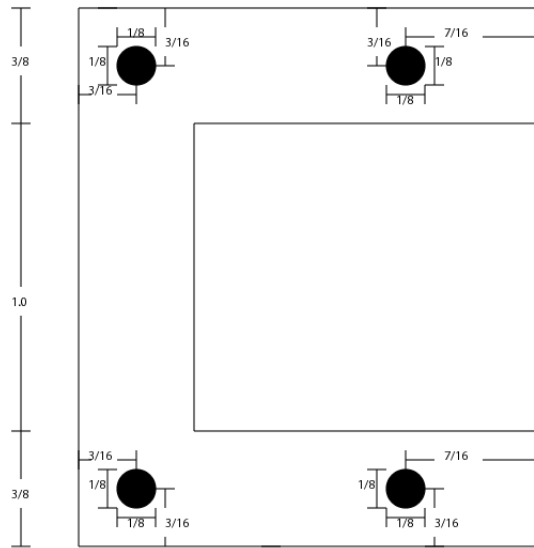


Figure 16: Base Mount (2 plates, 3/16" thick each)

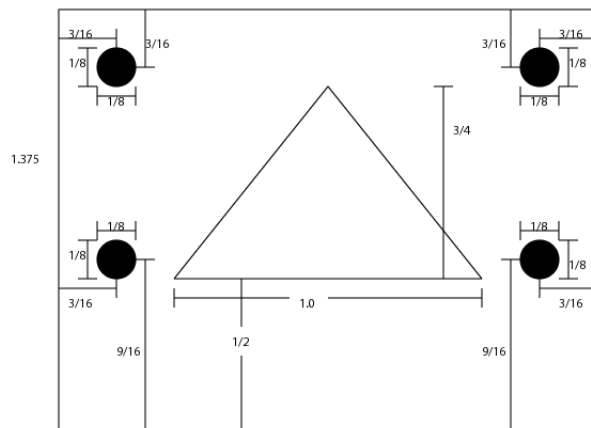


Figure 17: Rear Plates (2 plates, 1/4" thick each)

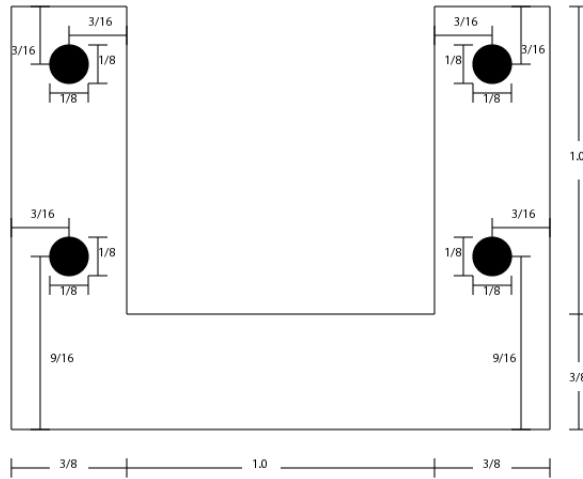


Figure 18: Rear Mounts (2 plates, $3/16$ " thick each)



Figure 19: Spacers (3 plates, $3/16$ " thick each)

6.6 Insulation

The insulation in the detwinner is very soft as produced, but hardens significantly when heated for several days. The cuts on the insulation are not exact, since the material is fairly soft even when hardened, and exact cuts are impossible. However, precision is not needed when cutting the insulation, since the insulation serves no structural purpose except holding the heat shields in place. The insulation has additional notches cut underneath to allow wires to pass, and on top to allow gas flow. The picture does not include these because the notches are unexact, and the dimensions are unknown. The insulation was donated. The sketches for the insulation are Figures (20), and (21).

6.7 Pneumatic Press

The press pictured in Figure (22) is a high load lifter with a load return, and along with the heaters forms the heart of the detwinner. The load return option means the lifter does not have the ability to return to its compressed state without an external force applied to the shaft, or pulling a vacuum on the gas inlet valve. According to the engineers at HuntVac it is fine to pull a vacuum in the pressure chamber. Without the vacuum the press applies a force of about 5lbs on any crystal due to the spring like construction of the casing around the shaft. The disadvantage of using this part is that the detwinner applies pressure to the crystal constantly, and while the pressure is reducable, it is not removable. Also, this press is not ideal for crystals requiring 5 lbs or less of force.

The high load lifter is a vacuum component for lifting and lowering heavy components inside a vacuum. This series can apply forces up to 110lbs with an air supply of 120 psi. The press can very likely withstand pressures significantly above the rated limits, but I do not recommend testing that without first contacting HuntVac. There are very few devices capable of delivering more than 20 lbs of axial force inside a vacuum, and of those this is the only one that costs less than \$1000. This is the best choice on the market partly because of price, and partly because the simplicity of the design. Most detwinning devices have a linear motion transfer device that presses on the crystal, and a piezoelectric crystal between the linear motion feedthrough and the slide for measuring the pressure. A pneumatic press has fewer internal parts than a linear motion feedthrough mechanism, does not need the piezoelectric crystal, and is able of delivering a far greater pressures when needed. Additionally, engineers designed this device specifically for applying large forces, as opposed to the linear motion feedthroughs that apply forces only as a byproduct of their intended functionality of linear motion. Together this implies the pneumatic setup should be less prone to breaking than a typical linear motion feedthrough mechanism.

The only disadvantages of the pneumatic setup are that the air pressure measurement for the pneumatic press is not directly equivalent to pressure on the crystal (there are corrections due to the area of the press and the area of the crystal, a different calculation is needed for each crystal), and it also has

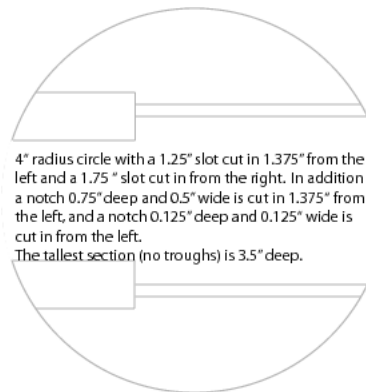


Figure 20: Insulation: Top Down View

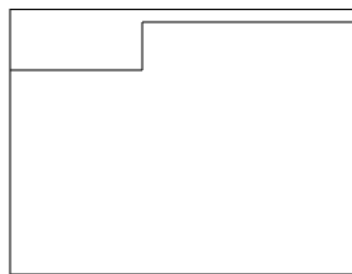


Figure 21: Insulation: Side View

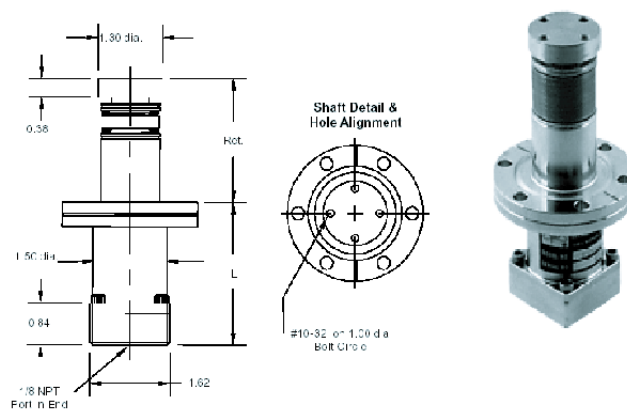


Figure 22: Pneumatic Press

a larger uncertainty than the electronic crystals. Though, when detwinning the crystals the exact pressure is not critical, all that is needed is a balance where the pressure is large enough to detwin, and small enough not to break the crystal. The advantages of a pneumatic press outway the drawbacks.

The press is available as part number PFHL-275-1-LR from Huntvac for \$680. Part PFHL-275-1 is a better part for this application, and only costs \$765 (an \$85 increase).

6.8 Power Feedthrough (for heaters)

The heater draw in Figure (23) feedthrough is an 8-pin power feedthrough. For sealing reasons the leads in this feedthrough are kovar (en.wikipedia.org/wiki/Kovar), which is a very soft material. Feedthroughs with ceramic leads are available with higher power limits, however the detwinning apparatus requires less than 1.5 amps for each of the leads. So, the price considerations outweighs the benefits of the more rigid leads in the ceramic feedthroughs. Other connectors with fewer than eight pins exist, however they come at a premium price. The extra pins are cut short inside the box since they are not only not needed, but also increase the chance of one of the heaters being entirely shorted out if one of the long pins contacts two of the active pins.

The 1.33" diameter model costs \$140 and is part number 643001 from MDC vacuum. When installing for the first time I suggest cutting the extra pins as short as possible, and the cutting needed pins fairly short as well. Trimming kovar does not damage the material.

CAUTION: The leads inside the casing are very soft, and pushing the pins onto the leads frequently bends the leads. If the leads bend too far they will short out against each other, or against the side of the detwinning box. This will be a 120 VAC short, and can cause injury and damage the device. Also, if only one of the pins shorts connects with the box then the box will charge to 120 VAC and become dangerous touch. Be careful when assembling this part.

6.9 Thermocouple Feedthrough

The thermocouple feedthrough diagramed in Figure (24) is a standard part from Kurt J Lesker Company, with the exception that the leads are shorter in order to fit into the tubes as designed. The tube for the thermocouple can extend as far as necessary from the main environment, so the shorter leads are mainly for aesthetic and convenience purposes. In future models use a longer tube and longer leads for the thermocouple feedthrough, since Kurt J Lesker Company took almost three months to build and ship the part, and ordering shorter leads almost doubled the price. The power feedthrough needs the shorter leads (the leads they provide bend easily, and can short out the heaters) but the thermocouple is attached by screws, and so it does not have the same problem. When replacing this part, it might be possible to modify the standard part delivered by Kurt J. Lesker to shorten the leads. If the thermocouple is clamped to the leads along

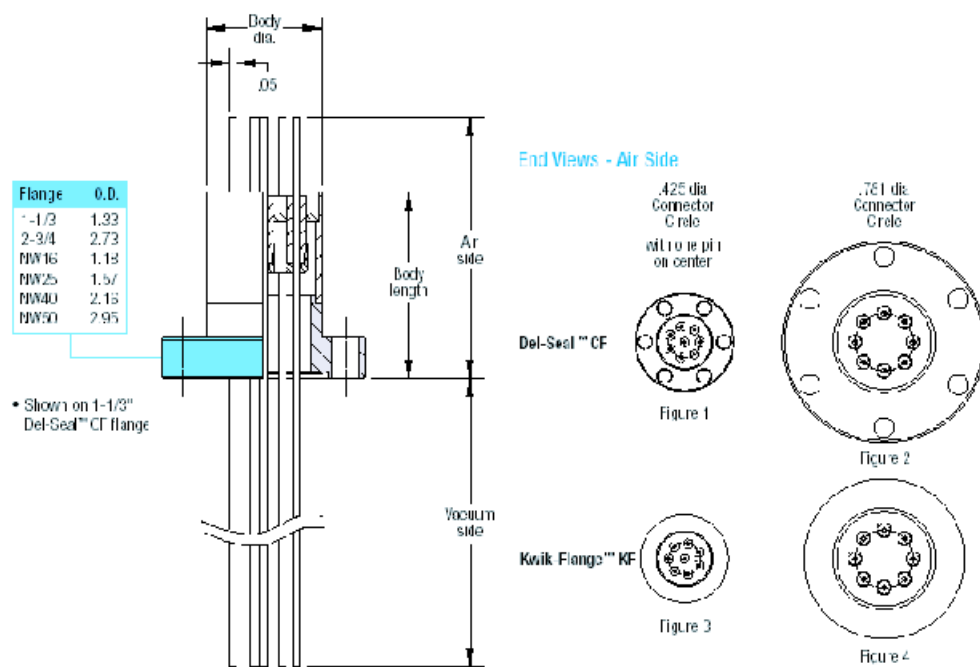


Figure 23: Power Feedthrough

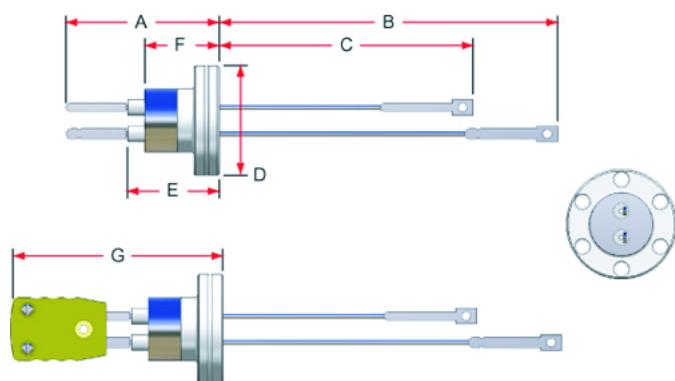


Figure 24: Thermocouple Feedthrough

the stud rather than attached by screws at the end, the standard part should be usable rather than the special order part.

This is part number TFT1ky00002 from Kurt J Lesker Company with the leads shortened to 1" and 7/8" and costs \$205 rather than the listed \$109.25.

A=1.81" B=4" C=3" D=1.33" E=0.58" F=0.38" G=2.42"

6.10 Gas feedthrough and shut off valve

These gas valves pictured in Figure (25) are some of the very few valves that operate at UHV. The valves have a directional arrow stamped on them, but the flow is slow enough that the direction of the gas flow does not make a large difference. Having two valves gives some flexibility of the environment. Either gas can flow in one side and out the other, or one valve can be closed, and a vacuum drawn through the other, or pull a vacuum then allow a gas flow through the detwinner, and then shut off the flow and close both the valves in order to have a stagnant environment. Once assembled the closed box will hold a rough vacuum (0.01 atm) for at least 3 days.

The feedthroughs are part number 133-STV-1/4-SWAG from NorCal and cost \$205 each.

6.11 Assembled Box

The parts assembled as described in Section 9 form the detwinner. The sample sits on the sample stage and is pressed against the back of the stage by the slide which derives its force from the pneumatic press. Figures (26) and (27) depict the parts as assembled in the final apparatus. Figures 28, 29, and 30 are several pictures of different angles of the partially assembled and fully assembled apparatus.

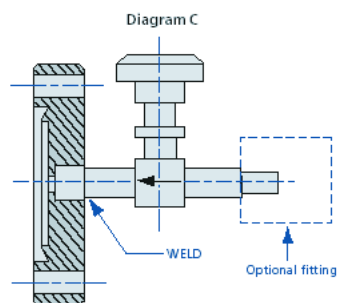


Figure 25: Gas Feedthrough

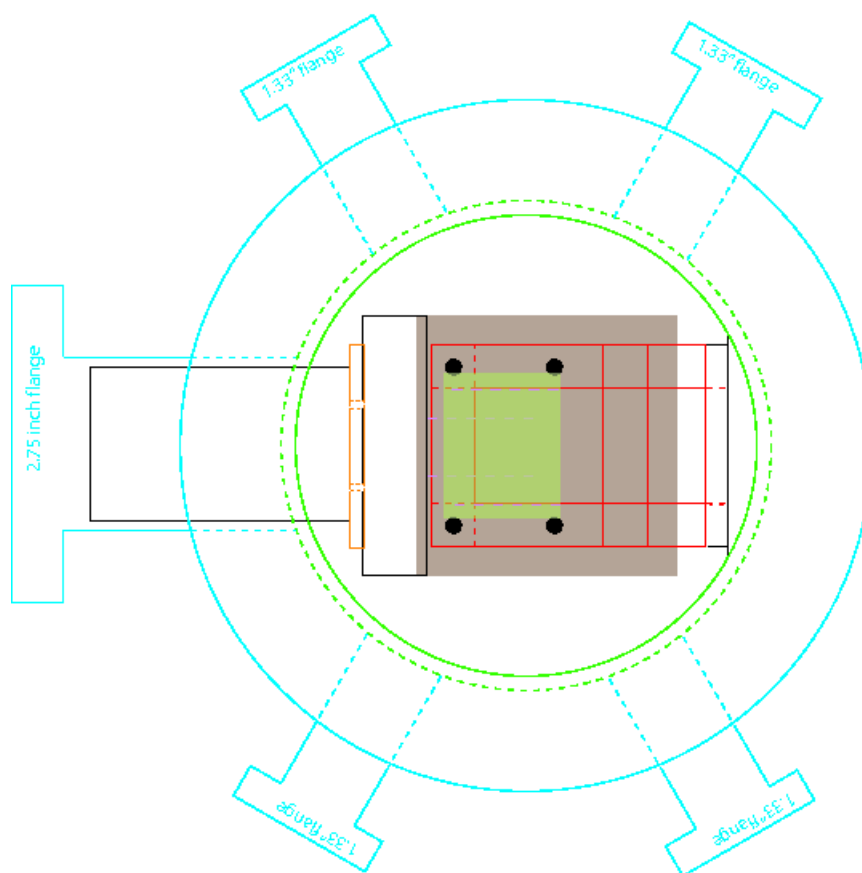


Figure 26: Assembled Apparatus: Top down

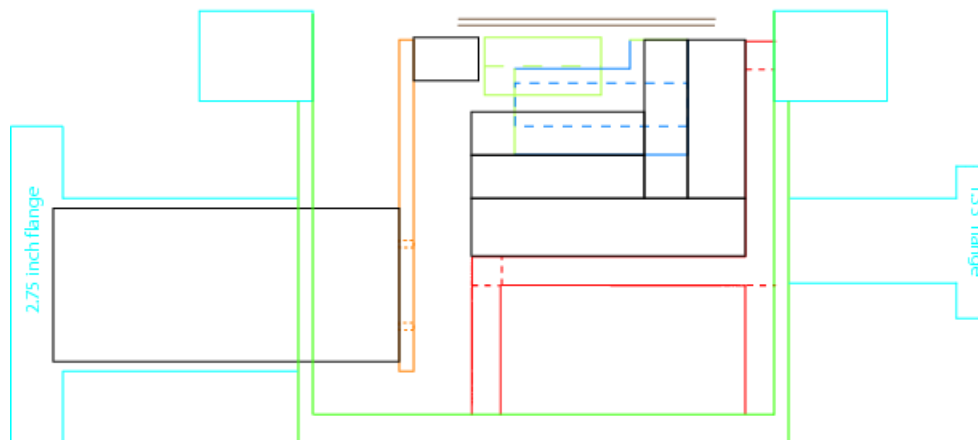


Figure 27: Assembled Apparatus: Side

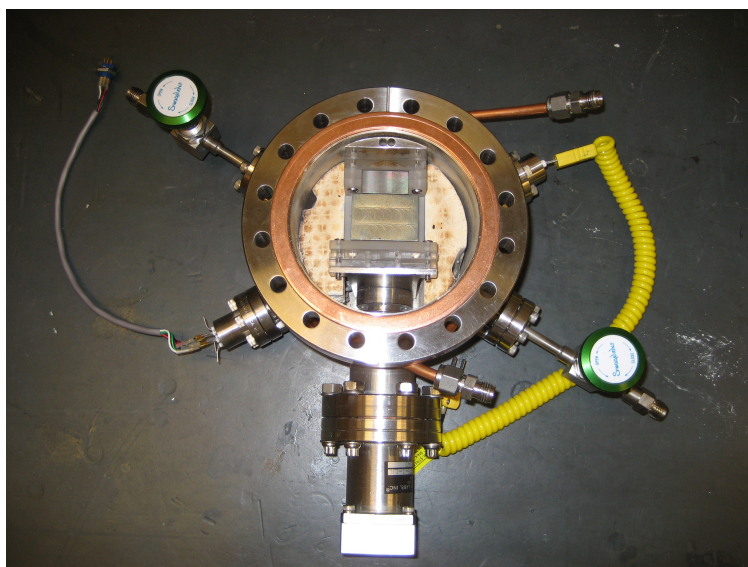


Figure 28: Assembled Picture: Top Down



Figure 29: Stage Assembly

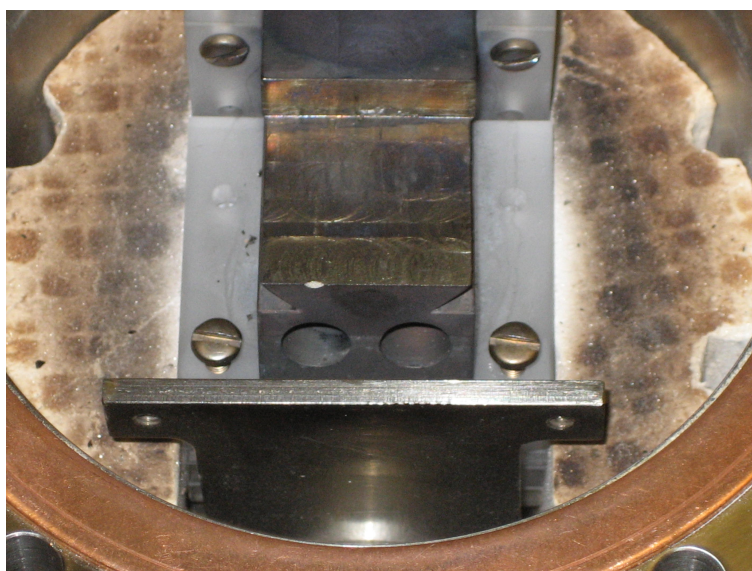


Figure 30: Sample Stage

6.12 Water Cooling

Originally the cooling tubes were wrapped around the outside of the detwinner. However, the largest heat problems come at the surface of the viewport and themicroscope objective. So, the original tubing was replaced by a ring that sits on top of the viewport and wraps around the objective. This cools the objective and the viewport where the cooling is most needed. This setup is good for temperatures up to about 400 Celsius. Above that temperature more tubing should be wrapped around the outside of the detwinner for safety reasons. The tubing came from spare parts, and so the price is unavailable.

7 Control Box Component Descriptions & Schematics

This section describes the componens of the temperature control box pictured in figures (31), and (32). The temperature control reads in the temperature from a thermocouple and turns on or off two heaters based on a fuzzy logic circuit inside the heater relay control.

7.1 Heaters

The heaters are 100 ohm heaters that run at 120 VAC, and 150 watts. The heaters have a non-stick coating on them in order to ease the process of inserting them into, and removing them from the sample mount. These were ordered from McMaster and are no longer available, but part number 3618K352 should be a decent replacement, at \$19.04 each. The most important factors is making sure the heaters are the right diameter, length, and power rating. Ideally the heaters will fit very snugly into the holes, however the processing technique used on cartridge heaters is not exact, and although the heaters have the same nominal diameter, in practice that varies somewhat. In our detwinner the stage was bored for two particular cartridges to fit snugly, so it is possible that even if the correct nominal diameter cartridge is ordered it might not fit correctly into the mount.

7.2 Thermocouple

The thermocouple is a K type thermocouple. All standard lengths are far too long for the internal workings of this machine. Any purchased thermocouple must be cut shorter in order to work properly. A good source for thermocouples is omega.com. Our thermocouple is BARE-20-k-12, and is available for \$6. The K type thermocouple is ideal for oxidizing or inert environments, while in vacuum or reducing environments a type E thermocouple should work better.



Figure 31: Temperature control: Outside

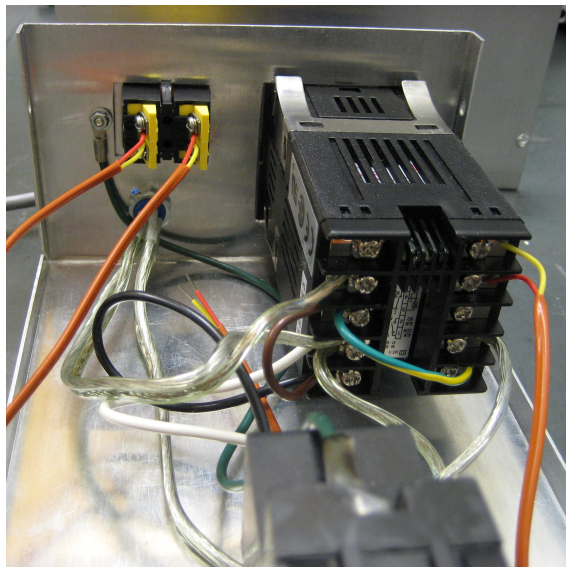


Figure 32: Temperature Control: Inside

7.3 Pressure control

The original pressure control valve was a high accuracy pressure control device. It was a Type 100G Precesion Air Pressure Regulator available from ControlAir Inc designed to regulate pressures from 2 to 120 psi. However, the control leaked gas, and had to be replaced. Now we have the pressure line attached directly to the pressure regulator on the gas supply tank. This works almost as well. The problem with this setup is that there is no relief valve if the pressure needs to be reduced quickly. If the gas supply is a compressed air line rather than nitrogen or argon, the air ought to be filtered to prevent dust from degrading the functionality of the press.

7.4 Interconnect Cables

The cable for the heaters is custom built by the electronics shop at the University of Tennessee. The cable is a 4 pin directional cable. The cable is directional in order to reduce the chance of crossing the wires. The plugs on the bare wires attached to the electric feedthrough are “Bird Pins.” The bird pins are very delicate, and likely to break unless attached with a specialized clamp. Although better methods might exist, this was the best method at my disposal.

7.5 Control Box

7.5.1 Power Cable

The power cable is a standard AWG cable similar to any used on a standard PC desktop.

7.5.2 Thermocouple Connector

The thermocouple cable is part 3873K272 from McMaster and costs \$26.32. The thermocouple panel information is unavailable, but a good replacement is part MPJ-K-F from Omega for \$2.25. The clipped portion of the thermocouple can function as the connection inside the box, but sheathed wires are much better.

7.5.3 Power entry module

Any power entry module will work. Schurter part number 4301.1014.08 is a good model because of the fuse box available and the rocker switch included. This model is available from newark.com under the part number 50F2873 for \$5.50. The power entry module is not essential, as long as the power line has a proper fuse on it the components will be protected. However, the power entry module is very aesthetically pleasing, and simplifies the process of wiring the box. Additionally the power entry module is very cheap compared to the rest of the components.

The temperature control box requires a 5 amp fuse. Never use a “slow blow” fuse in the power entry module. Slow Blow fuses allow a large current for a

short amount of time. This feature is added in order to allow time for motors to warm up. However, circuit boards have no motors included, and a large current, even for a very short time, will blow the board. Replacing a fuse is much easier than repairing the circuit board.

7.5.4 Casing

The casing is a standard box available from any electronics manufacturer. It is not important for the box to be any particular material. For safety reasons the case must be grounded if it is metal. Our box was provided by the University of Tennessee Physics Department Electronics Shop, and is a standard off the shelf box.

7.5.5 Wiring

The wiring guide in the manual for the regulator is incorrect for our the device we ordered. The regulator manual is written for a low current, single output box, while ours is a high current dual output model. The proper wiring diagram is shown in Figure (33).

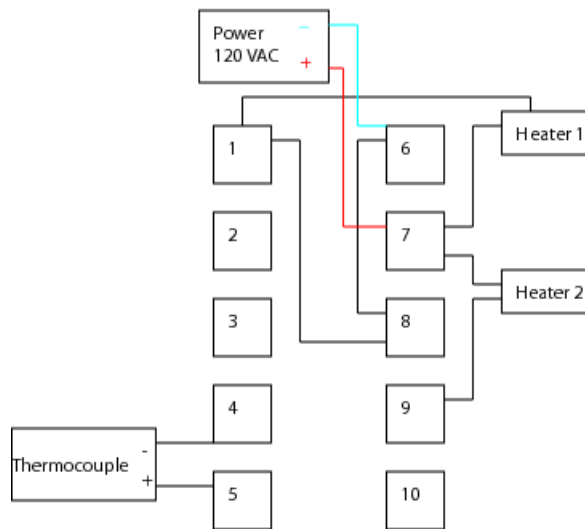


Figure 33: Corrected Wiring Diagram

7.5.6 Temperature Control Regulator

The temperature control regulator we ordered is part number TEC-9090 11013 from Tempco, and costs \$180. The controller comes with its own manual (See Appendix A), so here I will overview its integration into the rest of the system. The temperature control regulator is rated for 5 amps, however all internal working parts are rated for 10 amps. The box should be technically able to run at 10 amps, however there is a very high chance of blowing the heaters if the regulator runs any higher than about 5 amps.

The regulator we used is not ideal. I suggest contacting Omega Engineering and finding a controller with either a USB or ethernet connection, and two 1-amp output channels. There are higher current output models, but they would, of course, blow the heaters.

7.6 Total Cost

The total cost for all parts as I built this machine is approximately \$3400. However, I do suggest a press that is about \$100 more than I have listed, and a temperature regulator about \$100 more than I have listed. Additionally, any annealing products would have to be purchased separately. We have built a separate gas flow control mechanism, and purchased gasses and a vacuum. Depending on the quality of the gas flow controllers, purity of the gasses, and quality of the vacuum, these costs can vary wildly, our annealing apparatus costs approximately \$2500 plus gasses. Also, Molybdenum is an incredibly difficult material to work with, and cutting the dovetail in the two pieces wore a new carbide tip out, so that might have to be purchased separately. We have a cooling plate mounted to the bottom of our detwiner, but it is as effective to place the device on top of an insulator, unless you are planning on running at very high temperatures (above 600C) at which point you start risking breaking the seals inside the feedthroughs. In either case, it is more important to have the tubing wrapped around the flanges than under the box. Finally, all the tubing we used was from material we have laying around, and so if you have to purchase your own tubing that will cost extra as well.

8 Control Box assembly

8.1 Heater wire preparation

1. Cut a cable housing 4 separate wires into a 5" length, an a 18" length, and a 4" length
2. Trim 0.5" of the casing, and 0.25" of the wire sheathing off of one end of the 5" lengths, and both ends of the 18" length
3. Cut 2" of the casing and wire sheathing off one of the 5" lengths, and attach the other end to a female 4 pin connector

4. Attach a female 4 pin connector to one end of the 18" length, and a male connection to the other end
5. Cut 1" of the casing, and 0.25" of the wire sheathing off the other end of the other 5" wire, and attach a male 4 pin connection to the other end
6. Attach the end cut to 1" to the heater

8.2 Box assembly

1. Cut out a 1.75" x 1.75" hole on the front side for the temperature regulator
2. Cut out a 0.32" x 0.66" hole on the front side for the thermocouple connector
3. Cut out a 1 cm diameter hole on the front side for the heater power cable
4. Cut out a 1" x 2.25" hole on the back side for the power entry module
5. Attach three wires to the appropriate ports on the power entry module (be sure to use the 120 VAC terminals on the power entry module)
6. Snap the power entry module into place
7. Screw the ground cable to the box
8. Attach the thermocouple connection into place, either snapping it into place, or screwing it to the box
9. Trim the casing around a 4 wire cable, and the casing around each of the wires about 0.25"
10. Solder the wires to the 4 pin connector
11. Attach the connector to the box, with the wires feeding into the box
12. Run a wire to short pin 6 to pin 7
13. Run another wire connecting pin 7 to pin 1
14. Insert part way into the box (If it is snapped into place now most screw drivers will not fit in the box to tighten the screws on the terminals. So, feed it 1 inch or so into the box in order to have more room to manipulate the wires.)
15. Attach the Cr thermocouple lead to pin 5 and the Al lead to pin 4
16. Connect the leads of one heater to pins 2 and 7
17. Connect the leads of the other heater to pins 9 and 7
18. Connect the thermocouple cable to the thermocouple connector

19. Plug in the box and turn on switch. The LCD screen should read an error since the thermocouple cable is not connected to a thermocouple.
20. Change the set temperature to 0 so when it is plugged in later there are no sparks. (If the temperature is set above room temperature the cables from the box will be hot at 120V, reducing the set temperature reduces this risk.)

9 Detwinner Assembly

9.1 Build empty box

1. Cut a piece of 4" inner diameter (ID), 4.25" outer diameter (OD) stainless tube pipe to a length of 3.5"
2. Cut out 4 holes of diameter 1.33" circling the tube (these do not have to be placed exactly)
3. Cut out a 2.75" diameter hole centered 1.125" from the bottom of the tube
4. Cut out a 0.25" thick 4.25" diameter stainless steel disk for the bottom of the detwinner (Suggestion: Cut the disk so the radius is 2mm larger than needed, this will give more room for adjustments in step 10)
5. Weld 2" of 1.33" tubing to the 4 blank 1.33" flanges
6. Weld 2" of 2.75" tubing to the blank 2.75" flange
7. Weld the 5 flanges to the 4" tube
8. Assemble the chair as specified in the drawings
9. Weld the chair to the bottom of the detwinner
10. Place bottom of detwinner into the tube, shift until chair fits perfectly against the wall exactly 180 degrees opposite the hole cut for the pneumatic press
11. Weld the bottom in place
12. Weld the back of the chair to the tube for support

9.2 Insert Electronics

1. Feed the cables for the heaters and the thermocouple through the rear mount quartz plate, through the hole at the back of the chair and out the respective ports
2. Trim power and thermocouple cables until approximately 1-2" remains out of the port

3. Trim leads for the heater feedthrough as described in ??
4. Attach “bird pin” connectors to heater power leads
5. Feed heater power leads through a 1.33” copper gasket
6. Attach “bird pin” connectors to the power feedthroug
7. Bolt the heater feedthrough into place
8. Feed thermocouple leads through a 1.33” copper gasket
9. Attach thermocouple leads to thermocouple feedthrough
10. Bolt thermocouple feedthrough into place
11. Check that the thermocouple leads do not touch eachother or the walls of the chamber (if using bare wire leads)

9.3 Attach quartz insulation, sample mount, and slide

1. Roughly place base plate level one in place
2. Place rear plate and rear mount in place
3. Attach the 4 rear plates to the chair using #5/40 screws trimmed to 0.9” of thread
4. Place base plate level two in place
5. Place the sample mount against the base plate level two, and into the rear mount back against the rear plate while sliding the heaters and thermocouple into the drilled holes
6. Attach the quartz base plates and base mount to the chair using #5-40 screws trimmed to 1.75”.
7. Check that the sample mount is held firmly in place
8. Using a dentist mirror check that the thermocouple wires are still not touching eachother or metal under the chair or inside the tube leading to the thermocouple feedthrough
9. Slide the slide into place

9.4 Insert the Pneumatic press

1. Place a copper gasket around the pneumatic insert
2. Insert the pneumatic press into the tube constructed for it
3. Bolt the press into place
4. Attach the adapter to the press (This process is difficult, I had to use a clamp to hold the adapter in the retracted position while using tweezers to start the screws. Once the screws are in most of the way you might be able to find an allen wrench that will fit into the gap between the adapter and the chair. If not, remove the screws holding the base plates and mounts in place, remove the base mounts, and turn the base mounts a quarter turn to give more room for movement. Once the screws are tightened turn the plates back to their original position, and screw them back in place.)
5. Attach the three spacers into place using #5-40 screws trimmed to 3/4" of thread.

9.5 Final Box assembly

1. Drop insulation in place
2. Place heat shields (with appropriate holes cut out) over the sample stage and slide
3. Put copper gaskets in place and bolt the gas feedthroughs into place
4. Attach the thermocouple cable to the thermocouple feedthrough and the control box
5. Connect the cable attached to the power feedthrough to the power connection cable, and plug that into the control box

10 Troubleshooting

10.1 Erratic or incorrect temperature measurement

The thermocouple works by producing a voltage differential in two wires of different materials soldered together. K type thermocouples are formed from Nickle Chromel and Nickle Aluminum soldered together. The negative wire is the Nickle Alumel and the positive wire is the Nickle Chromel. If the wires touch other than at the solder junction, or if they both touch metal elsewhere, the voltage differential can be disturbed, and the results become unreliable. The solution is to open the box, and try to separate the wires so that they no longer touch each other, or the stainless steel walls. If the wires are crossed at the feedthrough the temperature will be noticeably wrong, but in a stable manner. In this case just open the box and change which lead is connected to the feedthrough pins.

10.2 Constantly blowing heaters and or fuses

The control box is sensitive to the current that comes in from the wall. I am not exactly sure why, but for a long time the box would blow heaters and or fuses. The only way I found to work around this problem was to plug the control box into a surge protector. After being plugged into a surge protector the device has run for weeks without any of the electronic parts needing replaced.

11 Possible improvements

11.1 Cable Casing

The cable attached to the power feedthrough could be improved drastically. The wires on the feedthrough ought to be trimmed significantly. Heat shrink ought to be applied around each of the connections. The cable should be held in place somewhere other than the just at the connection in order to reduce the risk of the feedthrough being damaged if the cable is accidentally jerked.

11.2 Temperature Control

The temperature controller I have picked out is decent, but not fantastic. A better controller would be the CN74000 Series controller available from www.omega.com with a RS-485 serial port for \$180. The computer connection allows a much greater control over the warming and cooling process through a program like labview.

11.3 Pressure Control

There are computerized pressure control devices available, such as the OEM-EP Minature Pressure controller produced by Pneutronics and available through www.parker.com If one of these were combined with a computerized temperature control the entire process could be observed and controlled remotely, with the exception of loading and unloading the crystals.

11.4 Viewport

The viewport suggested is made using crown glass. Quartz does not have the same depolarizing problems, and is a possible replacement part. However, a quartz viewport costs approximately \$2000, as opposed to the \$600 for the part in this assembly.

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Vita

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