Crystal Growth Procedure

This procedure is written assuming use of Cu -14.0 wt% Al- 4.2 wt% Ni. For other materials, changes may be required in the Heating Profile given in Section B.III. and the heat treatment procedure given in section C.III.

A. Preparation
   I. Material

Warning: Gloves should be worn when doing chemical polishing. DO NOT touch the material by hand after it has been cleaned.

1) Cut 3 cylinders from ingot with diameter 1.17 in (thickness of ingot ~ 1.7 in).
2) Use 2 and 2/3 cylinders as the starting material.
3) Mechanically polish cylinders to remove oxide. Use polishing strips found in room 24 (Buehler Handimet I Strip Grinder). Also, EDM cut .005" off both ends of each cylinder to remove deep oxide deposits.
4) Etch cylinders with 50% HNO3 for about 3 minutes (after etching, material should show yellowish color and grain boundaries can be observed).
5) Immediately submerge cylinders into distilled water and wash thoroughly.
6) Wash with ethyl alcohol to remove water, then dry with a hair drier.
7) Material should be ready to grow. Store in dry cabinet if growing is to begin later.

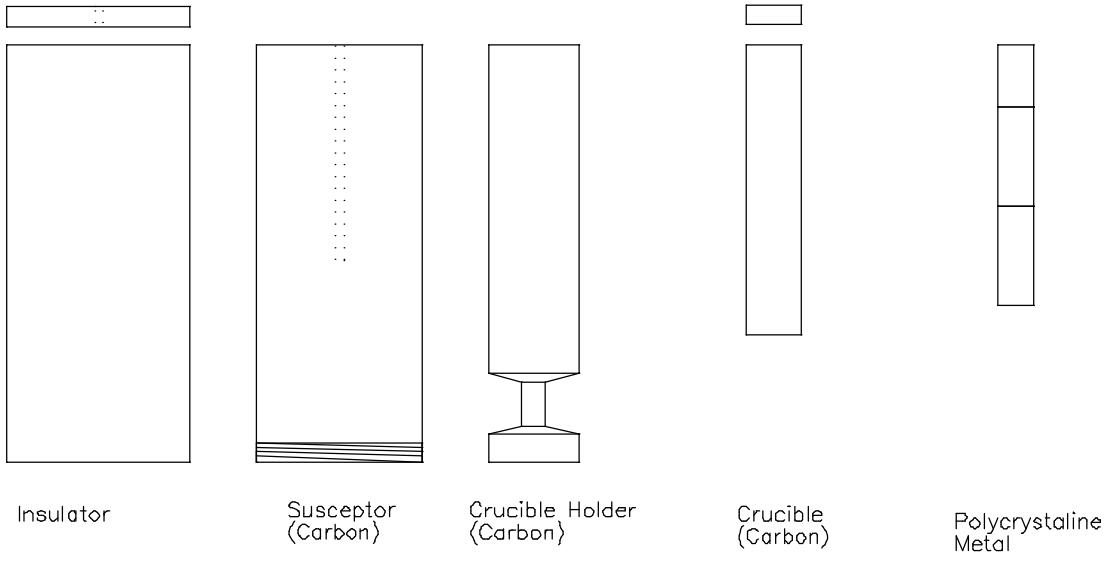


Figure 1

   II. Insulator, Susceptor, Crucible Holder, Crucible, and Thermocouples.

1) Remove the lower thermocouple. The lower thermocouple is at the bottom of the translation shaft in the cabinet under the chamber. There is a hole in the floor to allow this long thermocouple to be removed. The upper thermocouple should have been removed after the previous run and will not be in the chamber.
2) Remove the insulator, susceptor, crucible holder and crucible from the chamber (Figure 1).

3) Examine whether the inside of crucible (Figure 1) is clean. If not, use the special wood stick (Figure 2), wrap it with soft tissue and gently clean the inside. Keep crucible in the dry cabinet for 24 hrs. to remove moisture

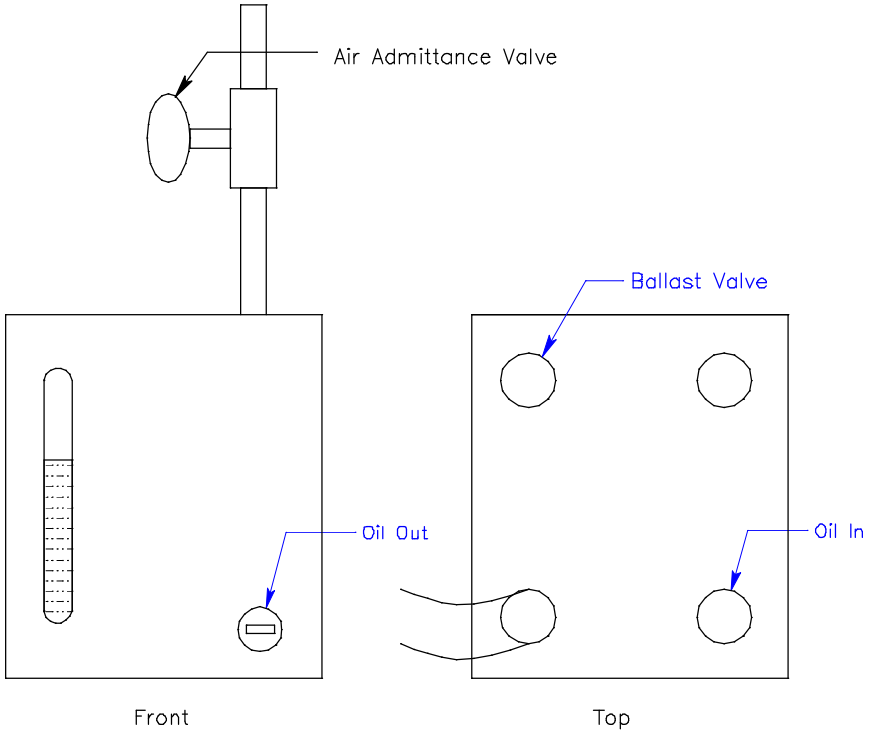
Figure 2

Special Wooden Stick

4) Wipe crucible holder (Figure 1) with methyl alcohol soaked tissue to remove excess oil and debris.
5) Wipe susceptor (Figure 1) with methyl alcohol soaked tissue to remove excess oil and debris.
6) The insulator and insulator cover (Figure 1) may be quite dark after several runs but it is not necessary to clean them. If there is difficulty in growing a crystal these may be changed.
7) The short upper thermocouple (type R), and the longer upper thermocouples should be cleaned with methyl alcohol. The O-ring on it should be greased with vacuum grease before use. Do not touch either thermocouple after cleaning.

III. Vacuum Pumps, Cooling Water, and Argon.

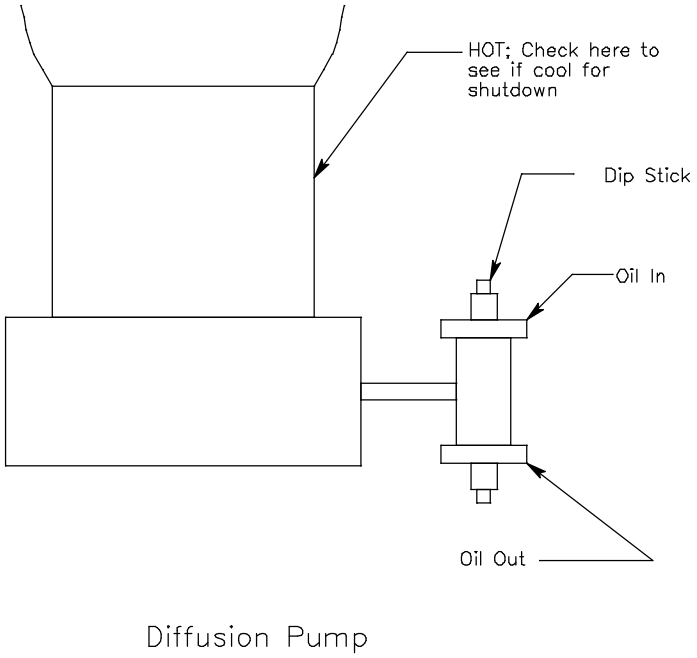
1) Check the fluid level of rotary pump (Figure 3) and visually examine the clarity of the fluid. If the fluid is dirty replace it. To drain the pump open the ballast and air admittance valves (Figure 3) to release any pressure within the pump. Drain through the oil out hole. Refill the pump using the oil in valve in the top of the pump (Figure 3). Refill till oil is just below the "Max." Line.



Rotary Pump

Figure 3

2) To check the oil in the diffusion pump the vacuum should be released by opening the air-admittance valve and the ballast valve then toggle the three position valve through all three settings (Figure 3), otherwise the fluid will be sucked up and the measurement will be incorrect. Check the fluid level of diffusion pump using the dipstick shown in (Figure 4). If fluid is dirty use the Oil Out (Figure 4) valve to drain, and the Oil In to refill with 100ml of diffusion pump oil. Close the air admittance and ballast valves when complete.



Diffusion Pump

Figure 4

3) Check the closed loop cooling water pressure by switching the two "light" style switches under the window. Be sure all valves in room 30B are open to allow water to the system. The water in the system should be changed yearly.
   If the cooling water needs to be changed:
   a) Order 30 gallons of distilled water from University Stores. The 6 gallon boxes work well because they are rigid enough to handle, and have a long spout for pouring.
   b) Drain the cooling water by attaching a garden hose to the fitting to the left of the pump switches near the window. The cap must be removed and the valve opened before draining.
   c) Turn on the pump will force the water out the hose and into the sink. When all the water has drained turn off the pump.
   d) Remove the hose and refill the water through the same pipe.
   e) Be sure to replace the cap and return the valve to its original position before continuing.
4) Turn on the cooling water to the chamber system using the yellow handled valve on the generator.
6) The pressure on the gauge near the floor next to the generator should read ~50psi. The upper pressure gauge should read above 60psi. If it drops below 60psi both of the Ametek water filters should be replaced. The filters are kept in the cabinet facing the pressure gauges.
7) Monitor the water-in temperature by placing a thermocouple on the copper pipe on the right side of the generator, floor level. This temperature must not exceed 30ºC or the generator will overheat.

8) If growing will not commence immediately, the water can be turned off.
9) Open the tank valve on the argon cylinder and be sure the pressure is >600psi. If it is not a new tank of argon should be ordered from University Stores. They will deliver it to the Mechanical Engineering loading dock. To swap tanks, remove the gauge assembly, apply new Teflon thread tape, and attach the gauge assembly to the new tank. Tanks should be secured with bungee cords at all times.

IV Chamber

1) Clean the inside, door, and lid of chamber with methyl alcohol. A hand-held vacuum is helpful for the bottom recessed area.
2) If oil contamination has occurred in the chamber, remove the bottom plate of the chamber (inside the cabinet (Figure 5)) and clean with methyl alcohol. Disassemble internal fixtures and carefully expose the felt rings which seal the translation shaft. Re-soak rings with diffusion pump oil prior to reassembly. Apply a thin coating of vacuum grease to all O-rings before re-assembly..

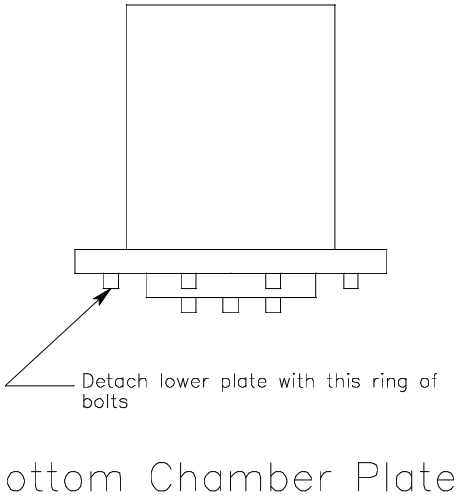


Figure 5

Warning: The next two steps should be done just prior the growth. Having a clean lubricated shaft is extremely important. If a high vacuum is not achieved or there is a suspicious leakage, the translation shaft is the first thing to check. Repeat steps 3 and 4 in this case.

3) To clean the translation shaft, turn on console power with green button at top. Then turn on the power to the translation controller (red). Use translate up and down (green and yellow) as needed to lower the translation shaft. Clean with methyl alcohol.
4) Apply a thin coating of diffusion pump fluid on the shaft using a small amount of fluid on lens paper. Rotation of the translation shaft (control panel) will aid in application.

V. Assembly

1) Raise the translation shaft so that the threads for the crucible holder are visible.
2) Screw in the crucible holder, then the susceptor. The translation shaft should be high enough so the top of the crucible holder is flush with the susceptor. There are adjustable stops on the side of the translation shaft to limit its travel. Use the DATUM button on the control console to set this as the zero position.
3) Place the metal polycrystal in the crucible and screw on the lid.

4) Using a partner push the lower thermocouple up into the chamber as far as possible. Then crucible should be gently lowered into the crucible holder. When the crucible makes contact with the thermocouple the partner should allow the thermocouple to be pushed down. When the crucible has reached the bottom of the holder tighten the lower thermocouple.
5) Check whether the crucible holder is concentric with the susceptor. If not, adjust the verticalness of the susceptor by adjusting the two screws at the bottom of the susceptor. The looseness of the susceptor will most adjustments without these screws.
6) Be sure that the thermocouple-hole in the susceptor is aligned with the thermocouple-hole in the lid of the chamber. Unscrew the susceptor until this is the case. An old thermocouple can be used to ensure alignment to prevent damage to the good (expensive) ones.
7) Lower the insulator between the coils and the susceptor.
8) Place the lid of the crucible holder on it.
9) Place the lid of the insulator on it and be sure the hole is aligned with the chamber lid hole and the susceptor hole.
10) Grease the o-ring on the cover of the chamber with vacuum grease lightly and uniformly.
11) Close the chamber cover and tighten the three screws alternatingly.
12) Mount the upper thermocouple (the o-ring of it should be greased). It should go through the chamber cover, the insulator cover and all the way into the susceptor.
13) Connect the signal output of the thermocouple.
14) Pull the thermocouple up for about 1-2 mm to avoid thermal stress build-up at the tip. Tighten the thermocouple.
15) Grease the chamber door with vacuum grease lightly and uniformly.
16) Close the chamber door by tightening the six big screws alternately.

B. Growth operation

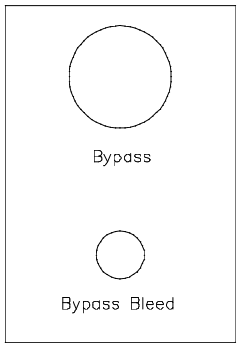
Warning: Resist the temptation to tighten the six chamber door screws while chamber is in vacuum. They will be extremely difficult to remove when the vacuum releases if you do.

I. Low power heating (baking)

The purpose of baking is to make sure the chamber is free of moisture. All parts inside the chamber can accumulate moisture over time. With 8% manual power, the temperature can get to 200ºC or so. Baking with this power for 8 hours is enough. Before turning on the generator, the chamber should be pump down to 10^{-5} mbar with both the rotary pump and the diffusion pump as follows:

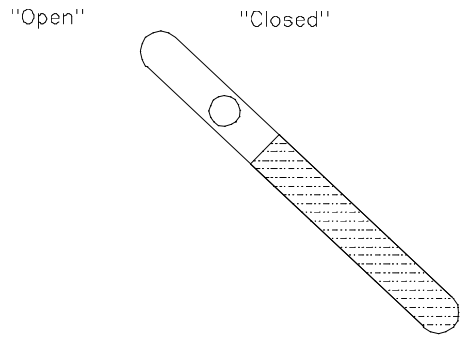
1) Check the positions of the following valves:

Valve	Position
Ballast Valve (Figure 3)	Closed
Air Admittance Valve (Figure 3)	Closed
Diffusion Pump Bypass Bleed (Figure 7)	Closed
Diffusion Pump Bypass Valve (Figure 7)	Open
Three Position Valve (Figure 9)	Neutral
Upper Butterfly Valve (Figure 10)	Closed
Gas in Valve (Figure 13)	Closed
Gas Out Valve (Figure 13)	Closed
Flowmeter Bypass (Figure 13)	Closed
Flowmeter Valve (Figure 13)	Closed
Chamber Valve (Figure 14)	Closed
Lower Butterfly Valve (Figure 6)	Closed



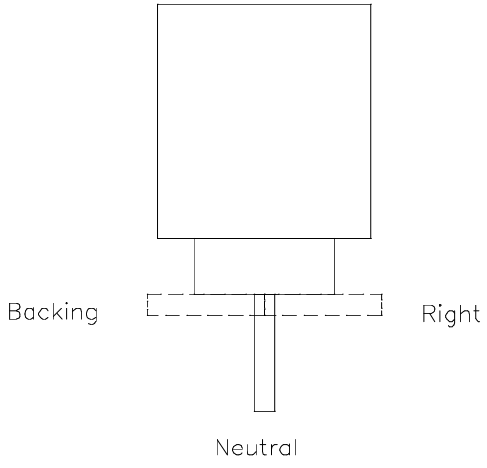
Diffusion Pump Bypass

Figure 7



Lower Butterfly Valve
Closed Position

Figure 8



Three Position Valve

Located above the Rotary Pump

Backing (Left) = Roughing the Diffusion Pump
Neutral = Roughing the Chamber through the Bypass
Right = Roughing the Cold Trap

Figure 9

2) Put the exhaust hoses out the window. It will get awfully smoky if this isn't done.
3) Turn on the rotary pump using the white console button.
4) Turn on the Pirani gage #1 by pulling the power knob out so that the green shows. Select #1 to monitor the pressure at the pump. Since the pump is working only on the chamber at this point this is also the pressure of the chamber. The other sensor (#2) measures the pressure in the cold trap.
5) Pump down the chamber to about 0.1 mbar.
6) Close the diffusion pump bypass valve.
7) Switch the three-position valve (Figure 9) to the right to pump down the cold trap to 0.1 mbar. Use Pirani gage #2 to monitor the pressure.

8) Switch the three-position valve to the left (backing) and pump down the diffusion pump to 0.1 mbar (Pirani gauge #1).
9) When the pressure in the chamber, cold trap, and diffusion pump are about equal to 0.1 mbar (Pirani Gauge #2), first switch the three-position valve to the right, then open the valve between the cold trap and the chamber (upper butterfly valve Figure 10). With this valve open the rotary pump works on the chamber through the cold trap.
10) Get liquid nitrogen from Smith Hall outside the chemical stockroom. The big storage ball can hold enough liquid nitrogen for more than 8 hrs. Fill the cold trap (Figure 10) with liquid nitrogen using a funnel. It is full when the liquid nitrogen is spewing out the top. A full cold trap can last about 4 hours. Refill every 2 to 3 hours. Liquid Nitrogen should continue to be added during the entire baking process.

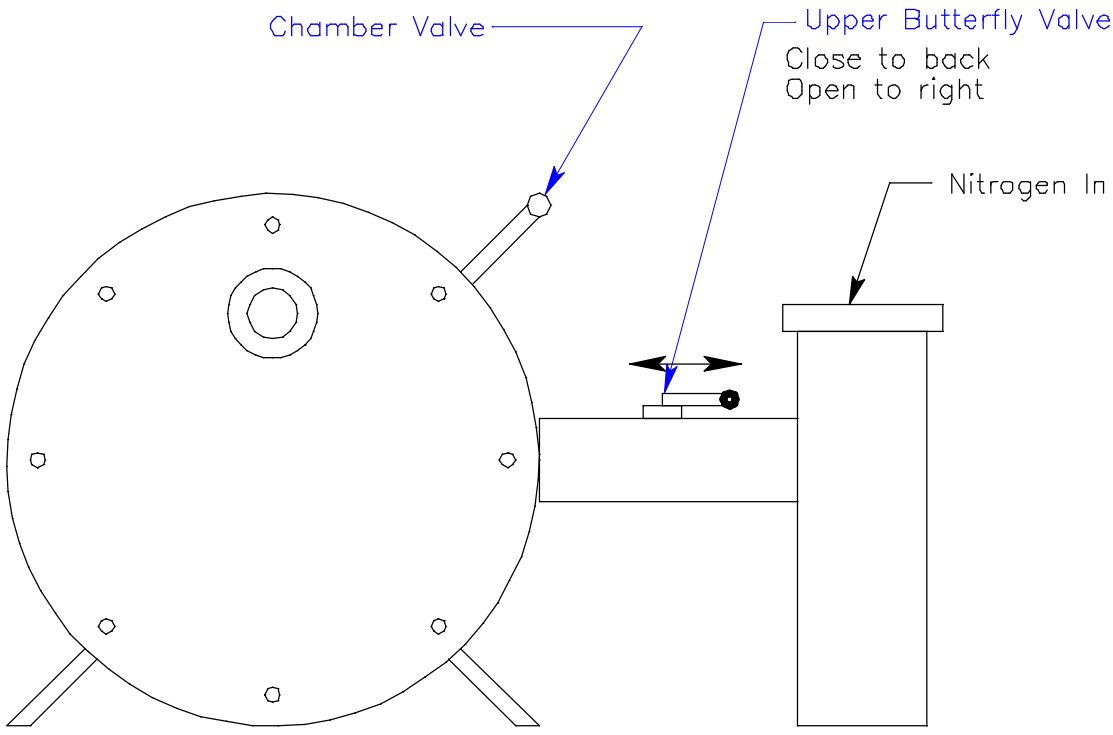


Figure 10

11) Turn on the cooling water to the chamber system. This is a yellow valve about waist high on the right side of the generator. Make sure that all three water lights on the control panel are lit. If not, open the water valves just behind the diffusion pump in the cabinet (Figure 11). There are valves for the "Chamber", "Crucible Shaft", and "Diffusion Stack" there. All three should be open at this point.
12) Switch the three-position valve to the left (backing) and ensure that the diffusion pump is at 0.1 mbar (Pirani gauge #1).

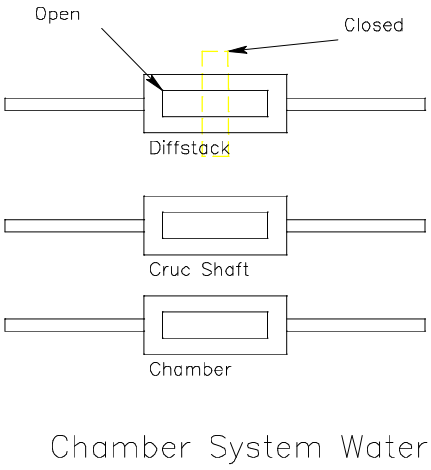


Figure 11

13) Turn on diffusion heater using the "Diffusion Heater" button on the control console. It takes 20 minutes to warm up to working temperature.

Warning: The three-position valve is always in the backing (left) position when the diffusion pump is on.

14) After the diffusion pump has warmed, Pirani gauge #1 should still read 0.1 mbar (less than .5 mbar is an absolute requirement). Pirani Gauge #2 should also read 0.1 mbar (less than .3 mbar is an absolute requirement). If the pressure is too high on Pirani Gauge #2, switch the three-position valve back to the right to pump down the chamber and cold trap again. Return the three-position valve to the backing (left) position as soon as possible to ensure that a vacuum is maintained in the diffusion pump.
15) **Slowly** open the lower butterfly valve (Figure 12) to initiate operation of the diffusion pump.

Warning: Only use the Penning gauge when the pressure in the chamber is below .1 mbar!

16) Turn on the Penning gage by pulling out the power button to reveal the green section. The pressure should drop down to 10^{-5} mbar in a few minutes. Some fluctuation may indicate out gassing and is O.K. If fluctuation is very severe and does not stop in a few minutes, there may be a leakage. The translation shaft is the first thing to be checked. With fast mode button depressed on control panel, the shaft up and down to stop fluctuations. If this affects, but does not stop the leaking, then the shaft needs to be cleaned (see step "IV-4)"). If the pressure does not drop below 10^{-2} mbar in a few minutes, then it is likely that the diffusion pump was stalled when the lower butterfly valve was opened. Ensure that the diffusion pump is warm (don't burn your fingers), close the lower butterfly valve, allow the diffusion pump to stabilize, and return to step 14.
17) Turn on the closed-loop cooling by switching on the two switches under the window. Be sure all the valves in room 30B are open to allow cooling water to the system. Monitor the water-in temperature by placing a thermocouple on the copper pipe on the right side of the generator, floor level. This temperature must not exceed 30ºC.
18) Switch the generator on to the "test" mode using the key on the generator panel. Wait for three minutes, then press the "off/reset" button next to the trip set light on the generator panel.
19) Set the controller to give the generator 8% power. Press the following keys to set the generator.

Display	Key	Comments
	2	Cycle to Function Program
Function Program?	**5**	"No" , change option
Function Auto?	**5**	"No" , change option
Function Manual?	**6**	"Yes" , accept option
Manual PWR ?%	**4** ▲ or **5** ▼	Raise and lower as needed
Manual PWR 8.0%	**6**	Run at set percentage

20) When the chamber pressure is less than 10^{-5} mbar and is steady, turn on the generator. This is done by turning the generator key to the "on" position and pressing the "generator on" button on the console. In one hour, the temperature should reach 100ºC. Watch the pressure closely, because leakage can still develop.

Warning: Never turn on the power when the pressure is between 0.1 and 10 mbar, otherwise arcing can occur and the coils can be damaged.

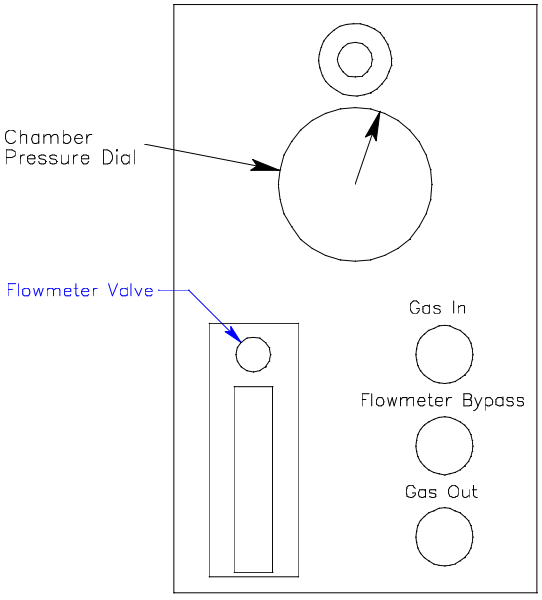
21) After baking for 8 hours, shut off the diffusion pump as follows (see also the Operating Handbook):
   a) Turn off the generator (control panel button).
   b) Isolate the diffusion pump from the chamber by closing the upper butterfly valve which isolates the chamber from the cold trap. Then close the lower butterfly valve which isolates the diffusion pump from the cold trap.
   c) Shut off the diffusion pump using the "Diffusion Heater" button on control panel.
   d) Turn off the Pirani and Penning Gauges.
   e) Let the rotary pump work for another hour with the three position valve to the left (backing mode). The lower region of the pump can be touched by hand (Figure 4) for 3 sec when the diffusion pump is sufficiently cool. Be very careful not to burn fingers. The chamber should be filled with argon as described in the purging stage below. HOWEVER, the argon should not be pumped out until the diffusion pump is cool!

Warning: Improper procedure can result in a contaminated chamber, because the vapor of the hot diffusion fluid can be sucked into the chamber. Therefore, enough backing by the rotary pump is extremely important. If the oil is hot the backing pump must be on or the chamber will be contaminated.

II. Purging

Now the chamber is absolutely clean and free of moisture. To further make sure the gas line is also free of oxygen, we need to purge the gas line and the chamber at least once.

1) Open the Argon tank and the inlet valves to the chamber. Open these 4 first:
   a) Argon Tank valve. The pressure gauge should read above 600 psi.
   b) Toggle valve (on gas line) should supply argon to the chamber and not the heat treat furnace.
   C) Gas In valve inside cabinet under chamber (Figure 13).
   d) Chamber valve to the upper right of the chamber door (Figure 14).



Chamber Gas Control

Figure 13

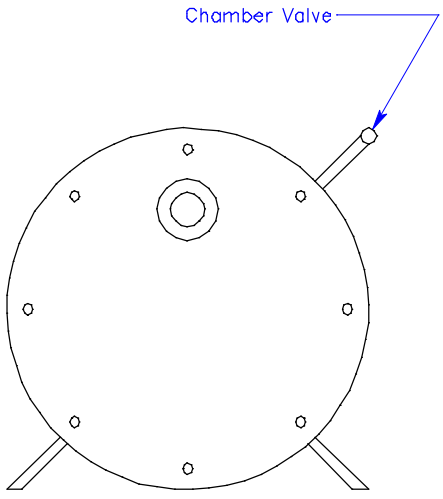


Figure 14

2) Slowly open the silver flow meter valve (Figure 13). Start the flow rate at 0.5 l/min. and gradually increase to 2 l/min. as the chamber is filling up.
3) Close the argon tank valve, Gas In valve, and chamber valve when the pressure reaches 1 ATM. The chamber pressure dial (Figure 13) reads gauge pressure and will read 0 when chamber is at one atmosphere. If you overshoot the pressure the Gas Out valve (Figure 13) can be used to release the pressure

Warning: Do not proceed to the next step unless the diffusion pump is cool!!

4) Switch the three position valve to the neutral position.
5) Open the diffusion pump bypass valve (Figure 7).
6) Turn on the Pirani gage #1 to monitor the pressure. The pressure should easily drop to below 0.1 mbar.
7) Close the diffusion pump bypass valve.
8) Follow steps 1 and 2 and fill chamber with argon again.
9) Follow the shut down procedure for the rotary pump:
    a) Open the ballast valve on top of the rotary pump.
    b) Run the pump for 15 more minutes.
    c) Turn off the rotary pump using the control console.
10) When the pressure is > +3 psi. (chamber pressure dial will read 3), adjust the flow rate and open the gas-out valve a little so as to maintain a steady state pressure of + 3 psi. and a flow rate of 0.5 l/min.

Notice: This is a good place to stop the procedure overnight. Since the Chamber is at a positive pressure and relatively little argon is being used there is no danger in leaving the grower till the next morning. If the diffusion pump and generator are completely cooled, ensure the diffusion pump is off and turn off the generator using the console button. Both the closed loop and chamber supply water can then be turned off overnight.

III. Melting and Growth

The melting temperature, growth temperature, and translation speed are crucial in the growth of a single crystal. The two temperatures should be adjusted according to the water temperature of the open-loop cooling system, which can change by 20ºC from summer to winter. Associated with the two temperatures, there are two dwelling times. The idea is that there should be enough time for the material to melt and enough time to finish the translation. The translation speed will determine the amount of time the crystal takes to grow. The speed presently used is 10 mm/hour. There is evidence in literature that the translation speed can vary between 6 and 60 mm/hour for Copper alloys but rate also depends on the impurity level of your alloy. The growth process could be considerably shorter if higher speeds are found to be acceptable for this apparatus.

The following settings have found to be satisfactory for Cu -14.0 wt% Al- 4.2 wt% Ni alloys:

1) Growth speed: 10 mm/hour.
2) Growing time: 24 hours for the above speed (total translation is ~ 240 mm).
3) Melting temperature: 1350ºC in summer and 1370ºC in winter.
4) Growing temperature: 1260ºC in summer and 1290ºC in winter.
5) Melting time: 2~2.5 hours.
6) P (proportional band) = 20%, I (integral time) = 500 sec, D (differential time) = 90 sec. These are the three temperature control parameters for an argon environment when using the EUROTHERM CONTROLLER/PROGRAMMER.

A suggested process is to start with a higher rate (e.g. 480°C/hour), then reduce to a lower rate (e.g. 60°C/hour). During heating, the rotation can be set at 2 r/min. After having dwelled at the melting temperature, the rotation should be stopped. The temperature rate from the melting temperature to the growing temperature can be set at -60°/hour. After the translation is finished, the temperature can be reduced to 500°C at a rate 60°C/hour (or to 1000°C at 60°C/hour, then to 500°C at 120°C/hour). Then the generator can be shut down. The following is a typical heating program used with successful results in both the summer and winter.

Crystal growth is a combination of art and science. We are still learning about all of the factors that affect the growth process on this Bridgman crystal grower. Melt temperature, growth temperature, and crucible rotation during the growth process may all significantly affect your abilty to grow a single crystal. These factors are currently under investigation.

Reference: Brice, “Crystal Growth Processes”, lab copy available in 130C Akerman

Step	Start Temp	End Temp	Rate (deg/hr)	Time (decimal hrs)	Elapsed Time (decimal hrs)
1	Room T	500	480	0.99	0.99
2	500	900	360	1.11	2.10
3	900	1200	240	1.25	3.35
4	1200	1320	120	1.00	4.35
5	1320	1350	60	0.50	4.85
6	1350	1350	Dwell	2.00	6.85
7	1350	1260	-60	1.50	8.35
8	1260	1260	Dwell	27.5	35.85
9	1260	900	-60	6.00	41.85
10	900	420	-120	4.00	45.85

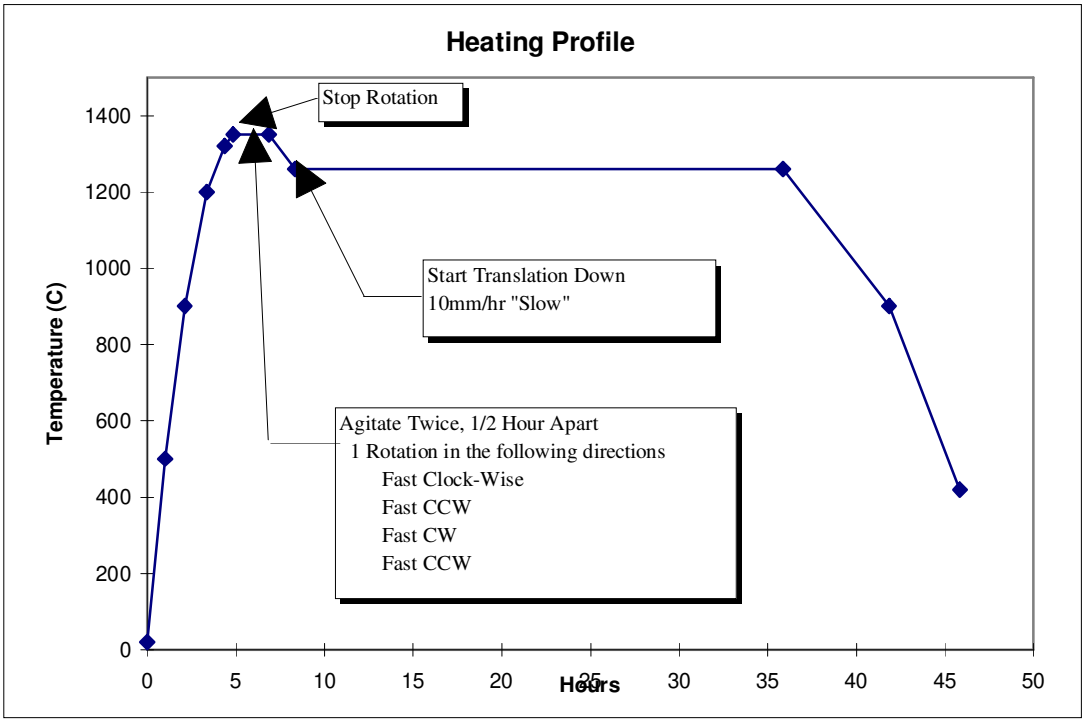


Figure 15

If gone overnight make sure that you turn both water systems back on and resart the generator.

1) Start the program using the Eurotherm Controller. Usually the program will be saved, but it may be necessary to reprogram it. General programming commands are given below. However, there are many contingencies which will cause deviation from this script. Yes, you will have to get out the manual. Note that the #6 key only works sporadically.

Starting a Pre-entered Program:

Display	Key	Comments
	2	
Function Program?	**6**	"Yes" , accept option
Choose Program?	**4** ▲ or **5** ▼	Choose desired program
	6	Start the program

Pausing a Running Program

	2	Hold the program
	5	Stop the program
	6	Start the program or reset

Writing a Program

Display	Key	Comments
Enter Prog	Hold **6** press **1**	
	6	"Yes" , accept option
64% Free Prog 3.?	**4** ▲ or **5** ▼	Choose program to edit
Servo Yes?	**6**	
Ramp Yes?	**6**	Run at set percentage
Ramp/Min	**5**	"No" cycle option
Ramp/Hour	**6**	"Yes" Accept option
To Level 1600ºC	**4** ▲ or **5** ▼	Raise and lower as needed
	6	"Yes" Accept option
P=4 S=2 End	**5**	"No" program the next segment. The first number is program number and second is program segment.
		Program segments as dwell, step or ramp as needed. Select end when done
Loop 1	**6**	"Yes" Loop one time.
Shutdown Yes?	**6**	"Yes" Shutdown after program ends.
Enter Prog	**1**	Gets you out of alter program mode and lets you choose to run a program.

2) Start the chart recorder.
   a) Open the recorder door and pull out the recorder. Use the power switch on the side to turn it on.
   b) Press list to print the channel settings. They should read:
      Ch1 Type R 400-1400 °C (upper thermocouple - controller)
      Ch2 Type R 200-1200 °C (lower thermocouple)
      Ch3 6V 0-3V (generator power)
      Chart Speed 10mm/hr
   c) If recorder is correctly programmed press "RCD" on the side panel and press "Display" until the recorder is in the auto cycling mode.
   d) If reprogramming is needed consult the manual. Some useful pages are:
      5-12 Time
      5-13 Date
      5-14 Chart Feed Speed
      5-19 Channels Use RNA Code 10 for Temp Channels
      Use RNA Code 02 for Voltage Channels
3) Turn on the closed loop and chamber supply water if they were turned off overnight.
4) Turn on the generator using the console button.
5) Start rotation of the crucible at 2 rev/min - .2 on dial, depress x10 button, and depress SLOW CW button.. Turn off rotation after program segment 5 (Figure 15).
6) Agitate the crucible twice during the high temperature dwell as indicated in Figure 15. Be sure to give the crucible time to stop rotating in one direction before starting the opposite rotation. Crucible holders have been broken by overly aggressive rotation.

7) Press the Slow Down button on the console translation controller at the end of the seventh program segment as shown in Figure 15.

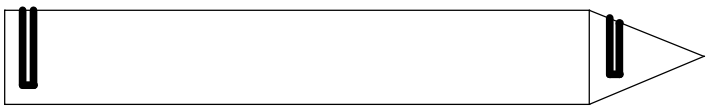
C. After Growth

I. Removing

1) Turn off the generator.
2) Let the crystal cool to room temperature (it takes 2 to 3 hours).
3) Close the argon tank valve, the Gas In valve, and the chamber valve. Turn off both water cooling systems.
4) Release chamber pressure to 1 ATM by opening the "Gas Out" valve (Figure 10).
5) Open the air admittance valve then toggle the three position valve to all three positions to ensure that the diffusion pump and cold trap are at atmospheric pressure.
6) Unplug and pull out the upper thermocouple.
7) Open the top and front chamber doors.
8) Raise and take out the crucible.
9) Turn off strip chart recorder.
10) Turn System Power off.
11) Removethe exhaust hoses from the windows and close windows.

II. Examining

1) Visually examine the crystal. It should have a golden appearance. The reflection should be shinning but may not be uniform. Extensive graphite stains may be on the surface.
2) Clean and wash off the graphite stains with scouring pad and water. Now the reflection should look dull but uniform.
3) Under the fume hood, etch the crystal with 50% HNO_3 in a plastic beaker for about 3 minutes. Slightly shake and rotate the beaker. Then quickly remove the crystal to a plastic beaker of water. Wash the crystal thoroughly with water. The crystal now should have a very uniform and very fine texture (size of different reflection should be less than 1~2 mm).
4) Place the crystal in the cutting holder. With EDM machine, cut one slice (~ .09" thick) from each end with a step cut as shown in Figure 16.



Single Crystal Boule

Figure 16

5) Remove the oxide on the surface of each disc by mechanical polishing.

III. Heat Treatment (for CuAlNi)

1) Clean the discs with methyl alcohol and put in the furnace.
2) With argon flowing at 0.5 l/min. and 7 psi., set the temperature in the furnace at 1000ºC.
3) After heat treating for an hour, quickly remove the disc into ice water (ice can be obtained from room 375, Mechanical Engineering).

Note: If the disc is thin, then it may warp as dipped into the ice water. In this case, the disc can be sandwiched in a pair of graphite plates before put in the furnace.

4) The disc should be mostly in the austenite phase (copper-like color).
5) To make sure it is a single crystal, first polish the disc mechanically to remove the oxide. Then etch it with 50% HNO3 (wash after etching). For a single crystal, the disc should have a smooth appearance and no grain boundary should be seen.
6) To further check the quality of the crystal, lower the temperature (using ice water) of the disc. If large wedge-like microstructure appears (contrast to many tiny microstructures), then it is a good crystal. The final crystallinity is checked by X-ray.

IV. Orientation

See "Crystal Orientation Procedure" for instructions.

Important manuals or handbooks:

CRYSTALOX BRIDGMAN STOCKBARGER GROWTH SYSTEM HANDBOOK,
CULLITY, ELEMENTS OF XRAY DIFFRACTION,
EUROTHERM CONTROLLER/ PROGRAMMER TYPE 822,
EDWARDS ROTARY VACUUM PUMPS (E2M8),
EDWARDS MK2 CRYO-COOLED DIFFSTAKS,
YOKOGAWA CHART RECORDING INSTRUCTION MANUAL

Supplies

Argon - University Stores
Distilled Water - University Stores
Dow Corning 704 Diffusion Pump Fluid - University Stores (from Fisher)
Fisher Brand 19 Mechanical Pump Fluid - University Stores (from Fisher)
Liquid Nitrogen - Chemistry Stock Room
Dow Corning High Vacuum Grease
Alcohol - Chemistry Stockroom
Crystal Grower Consumables - Crystalox
Plotter Paper and Pens - Yokogawa, 2Dart Rd, Shenandoah Industrial Park, Newnan, GA 30265, 404-253-7000; part numbers: plotter paper, charts, 0-100 linear, B9565AW; red pen, B9565AP; green pen, B9565AQ; blue pen, B9565AR; purple plotter pen, B9565AS
High Temperature Thermocouples -

W.C.C. Grebner
Revised Jan. 1996, Feb. 1996, April 1996

S. Jeseritz & W.C.C. Grebner
Revised Dec. 1995

C. Chu
Original Feb. 1994