Powder Preparation

The first step is to prepare a powder of your sample - the steps below are NdLSCO specific, but this will vary wildly sample to sample. Need between 20-40g of sample to properly do a floating zone growth.

1. Pre-fire the Lanthanides (La_2O_3 and Nd_2O_3) overnight at 1000°C
2. Measure the desired sample masses
3. Grind together (roughly 1 hour per 10g)
4. Fire at 1000C for about 1 week
5. Powder X-ray to confirm composition

Rod Preparation

Once the powder is prepared, it is time to shape it into a rod and create the Feed and Seed rods.

Feed Rod

The feed rod is the primarily element which will melt and create the crystal

1. Find black balloon, fill NdLSCO into it with Funnel patting down with wooden Qtip, once filled, tie it off (loosely, so air can still travel) close to powder
2. Place a cotton swab at the top to catch powder
3. Vacuum it using the blue box by the sink, simply attach it and turn it on. Once vacuumed, tighten the knot and turn the vacuum off
4. Use the two plastic pieces by the vacuum or hands to roll it like bread into a uniform rod
5. Place into the ceramic cylinder (on press), make sure it is fully inside and tape off the top - placing the rest of the balloon inside
6. Place it into the actual press (remove the flat metal top for pellets). Top up with water as needed to make sure it is not touched by the press component, but is fully covered
7. Slowly pump it up until 60 and leave it for 5 minutes
8. Remove pressure, remove top (may need to place whole press component on ground), carefully remove rod and place it inside the ceramic carry-boat (looks like a little home for the rod)
9. Gently cut open the balloon with sharp scissors. Make sure to not hit the rod with any high force etc as it can crack
10. Once cut open, remove balloon completely and fire rod in the boat at desired temperature (1150C for 20hrs)
11. Re-fire if needed - know it is done when rubbing finger across the rod leads to barley any residue coming off
12. Take metal wires, and attach as a mount to the rod. Do your best to centre the rotation of the rod when attaching the mount. Don't need a lot of room above the top loop

Seed Rod

The seed rod is the bottom element, which will serve as a basis for the feed rod. It should be a similar material, but does not have to be exact doping etc. (Ex. in initial X=0.07 growth, a seed rod of X=0.125 was used).

1. Create or find the seed rod, similar steps as above but only needs about 3-5cm of length
2. Find the seating element for the seed rod, located above drawer 3
3. Wire it up tightly, make sure that the rotation is at the centre so it barley precesses when rotating - If needed there are spacers for both height and width in that same drawer

Floating Zone Preparation

Basic Steps

Here are the steps for NON-SOLVENT floating zone. Steps to make this a solvent floating zone are listed below.

1. Turn main switch on
2. Remove door lock to open it, and pull apart the mirrors
3. Can set the rotation speed and mirrors how you want them using knobs. Hang the feed rod and seed rods. Make sure that when rotating they are roughly 50% overlapped at all times. Re-adjust as necessary
4. Move the mirrors all the way down, and reset the growth length . Then move them little bit up (5mm) and reset growth length again, this is the new starting position
5. Loosen bottom black knob (all the way at the bottom) then move the grey lock over to the right and open it more with a wrench
6. Move the seed rod shaft all the way down once the bottom black knob piece is open, it should be out of the way
7. Open the black piece at the top, lock it in do it doesn't rotate and open it fully. Move the feed rod up until it is also outside of the mirrors contraption
8. Place the CLEANED quartz tube into the floating zone and lock it in place with the grey rings. If you need to clean it, use a rod with paper towels on it.
9. Lock the upper grey lock and move the upper shaft to lowest possible position with the motors, reset position, and move it up 10mm. Then unlock the grey and move it to half way in the quartz tube, and lock it into place again.
10. Manually align the bottom rod with the top rod, until they are barley touching and somewhat centred in the mirrors. Then lock it into place as well.
11. Once position is set, turn on lamp, TV, lamp cooling air, SCR switch and door fan, reset upper shaft and grow length

12. Turn on cooling water (beside fire extinguisher, closer valve to FZ) turn on oxygen (2nd canister to the left of X-ray room door)
13. Flow meter is middle meter on FZ tube to check if it's flowing
14. Click page on the black/green screen to set manual mode, then increase SP to desired set point
15. Hand icon changes it from manual mode to auto mode and it will automatically ramp to the set point
16. Once at roughly 40 (or melting), start rotating. Rotation speed of 19RPM and 21RPM as a starting point isn't bad
17. If shaft doesn't melt, can increase set-point until it finally melts. Move the rods closer together until melting starts
18. Once melting starts, can turn on the mirror movement to start the crystal growth
19. Monitor the speed of rotation and growth speed, as well as temperature, and change as required throughout the growth.

Solvent Zone Preparation

This simply involves adding an extra solvent to help the floating zone process. Simply create a thin pellet of about 0.3g of solvent material (in this case, CuO) and place it on the seed rod.

1. Gather 0.3g of solvent material (CuO in this case)
2. If required, grind it to be uniform
3. Press it into a circular pellet
4. place it onto the seed rod when doing initial setup

The below image shows roughly what the feed and seed rods should look like when seated in the floating zone. And gives you an idea of what the wiring should look like.

Image Courtesy of Monika Azmanska, 2024