| **6/15/2023** |  | | | |
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| Objectives | Follow methods from 2017 Quantitative 3D paper, lowering temp to -39 C at a rate of 0.5 C per minute. This paper stuck to a range of -39 C to -29 C, with an accelerating voltage of of 17kV and probe current of 70 uA. Set pressure to 50Pa, corresponding to -32C equilibrium. Pump down to -31 and then reduce to -39 C at 0.5 C per minute (manually). Increase temperature to -33 C or above once crystals located and imaged for calibration | | | |
| Accomplishments/  Reflections | The snapshot mode is much faster, gain 3 produces nice image. Is this good enough?  Use snapshot for calibration when crystal is growing fast. Use normal for imaging roughness, temperature will have increased so growth won’t matter as much.  Crystals are best “quality” when they first start growing. Achieving this at lower temperatures is difficult though, since we are lowering incrementally. | | | |
| Cold stage size | | 51 mm | | |
| Cold stage height | | +8 mm | | |
| Distance of detector from stage  (5-10mm) | | 5 mm | | |
| Probe current (70-90) | | 70 | | |
| Accelerating voltage (Vacc) (12-17kV) | | 17 kV | | |
|  | | | | |
| Time | Action/observation | Temperature | Pressure (25-150 Pa, 40 most common) | Working Distance  (3 factors: focus, mag, stage height)  Error message if not from 9-11 mm |
|  | Cleaned copper cold stage, residue from Kayden’s work with potassium sulfate may have been left over. Procedure provided by Amy Repogle. |  |  |  |
| 11:15 | Set temperature to -31  Hovered around -31.8 for several minutes. Temperature reading off from temperature input by .8. | -31.8 | 50 |  |
|  | Set temp to -32  Lower temperature 1 degree every 2 minutes manually (can’t set decimals) | -32.9 |  |  |
| 11:24 | Found crystal and stopped lowering temperature. Case 1.0 far right | -32.9 | 50 | 9.7 |
| 11:29 | Case 2.0 is a little to the left of 1.0, a smaller crystal |  |  |  |
| 11:40 | Found more crystals but none had distinctive facets, edges kind of rounded. Lowered temperature, still incrementally. |  |  |  |
| 11:45 | No more new crystal growth so starting fresh. Raised temp to -15 |  |  |  |
| 11:48 | No more ice crystals, Set temp down to -32 then step incrementally | -32.7 |  |  |
| 11:50 | Set to -33 (2 minutes later) | -33.8 |  |  |
| 11:53 | Reset to -32 (accidentally pressed enter at -3 instead od -34) |  |  |  |
| 11:55 | Found crystal! | -33.8 |  |  |
|  | Found another, stopped temperature. Waited a couple minutes for crystals to stop growing so rapidly. | -34.8 |  |  |
| 12:00 | Analyzed first few crystals, but did not have distinctive facets. Only visible facets were too close to the crystal edges. | -34.8 |  |  |
| 12:06 | Set temp to -35. Want to see if lower temperatures produce more distinctive edges. |  |  |  |
| 12:08 | Set to -36 |  |  |  |
| 12:09 | Crystals spotted. Found one with 3 facets | -36.8 |  |  |
| 12:12 | Raise temp to -35 to slow growth | -35.7 |  |  |
| 12:14 | Imaging case 3.0 | -35.7 | 50 | 10.2 |
| 12:21 | Played around with 3D capture settings.  **\*Note:** Snapshots greatly alter the brightness/contrast settings after they are taken. |  |  |  |
| 12:40 | Set temp to -20 to reset |  |  |  |
| 12:43 | Set temp to -31. Wait 1 minute. Lower incrementally 1 degree a minute down to -39 before imaging |  |  |  |
| 12:53 | Set to -39, don’t go below this | -39.4 |  |  |
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