# HP-Ge Crystal Growth at USD

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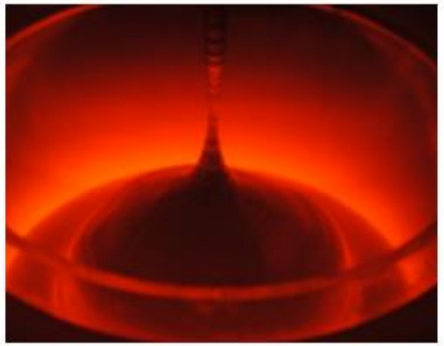
UNIVERSITY OF SOUTH DAKOTA

# Motivation

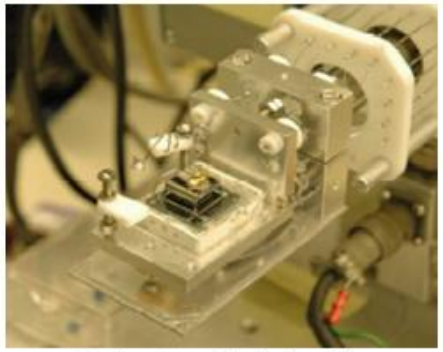
## Workflow

Zone refining



Crystal Growth

Crystal Characterization

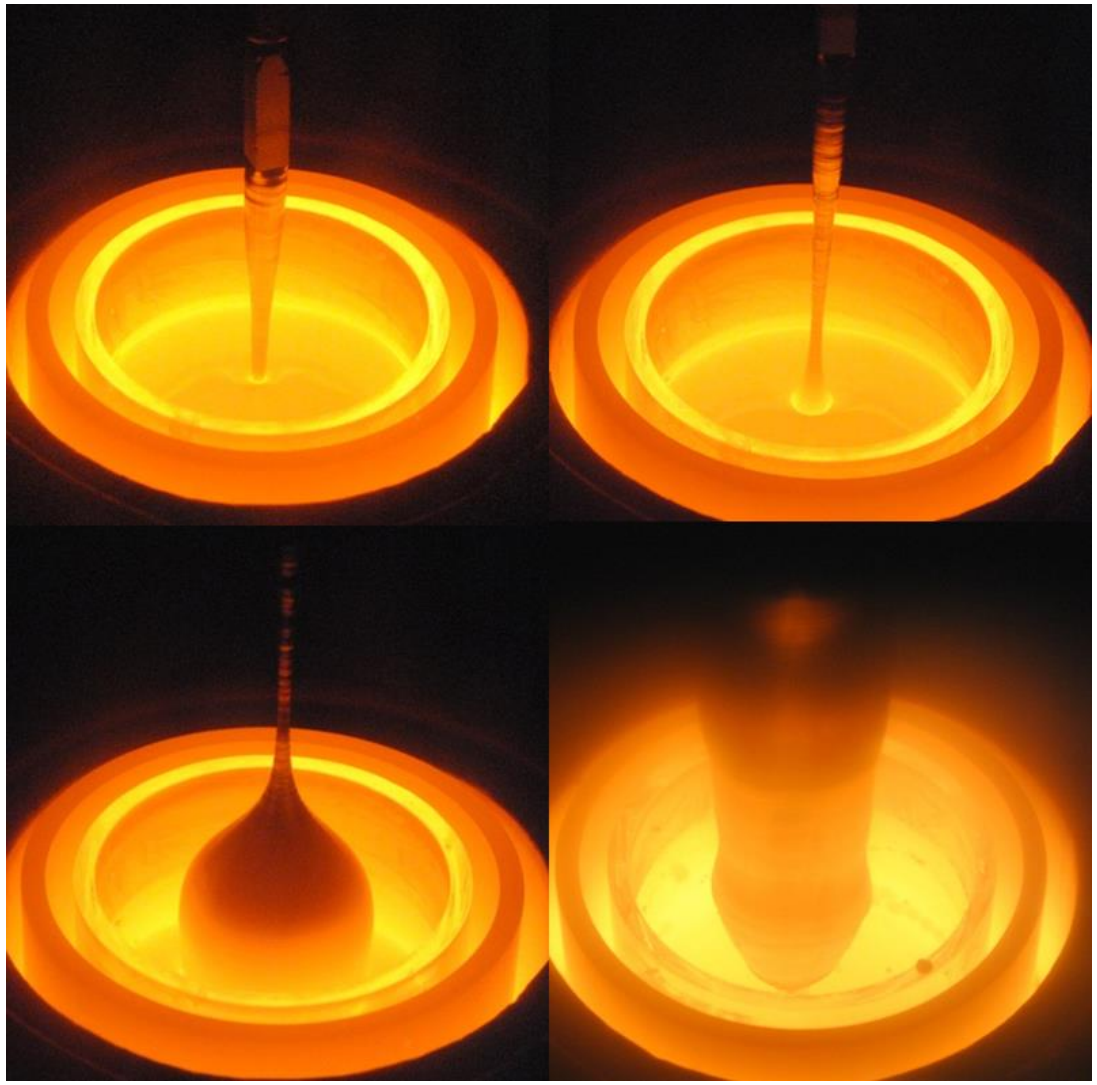


Detector Fabrication

## Requirements

- Mobility
  - Detector grade requires $> 35000\ cm^2/V \cdot s$.
- Impurity
  - Less than $3 \times 10^{10}/cm^3$.
- Dislocation density
  - $300 - 10000/cm^2$.
- Sizes
  - 5 to 10 cm in diameter.
  - 1 to 7 cm in length.

# Crystal Growth



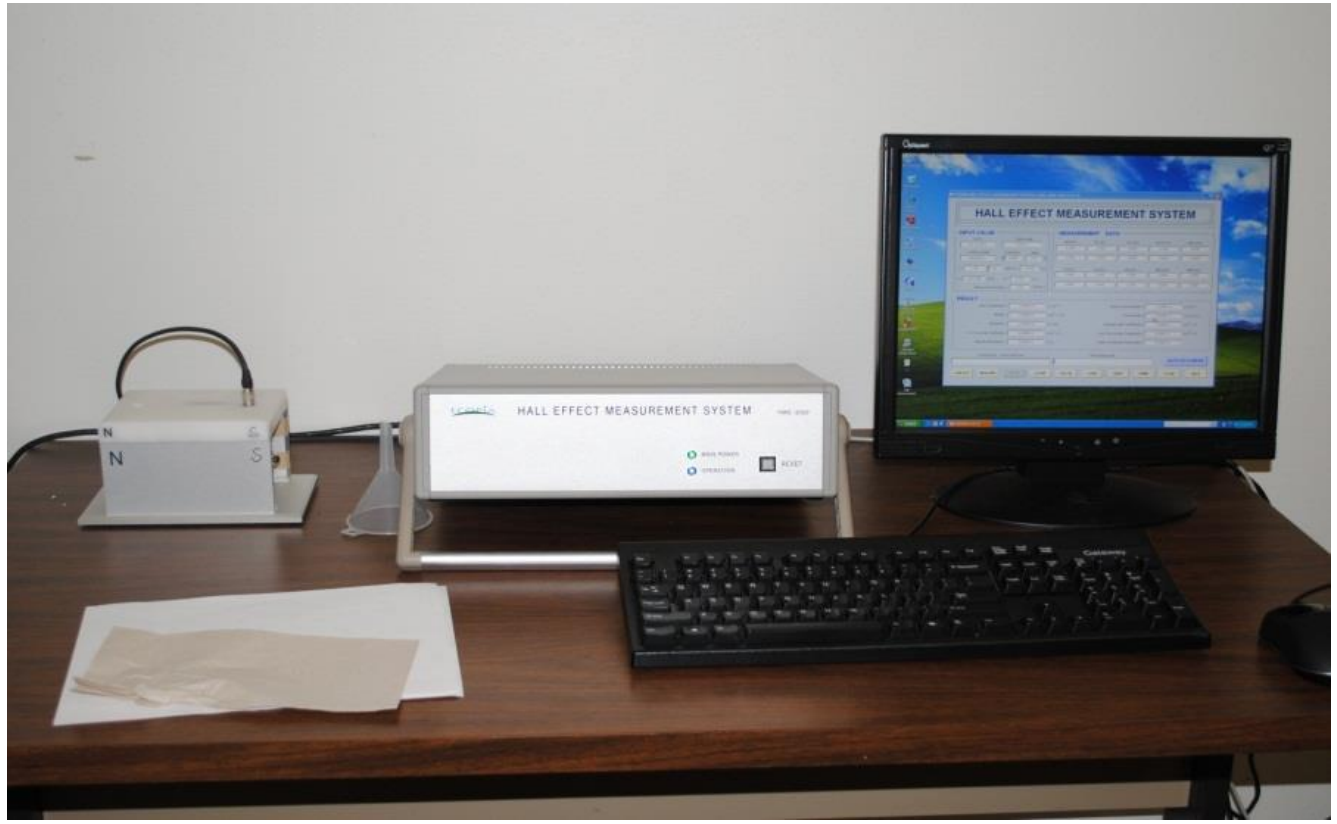
- Crystal Growth Process

- Crystal growth facility at USD

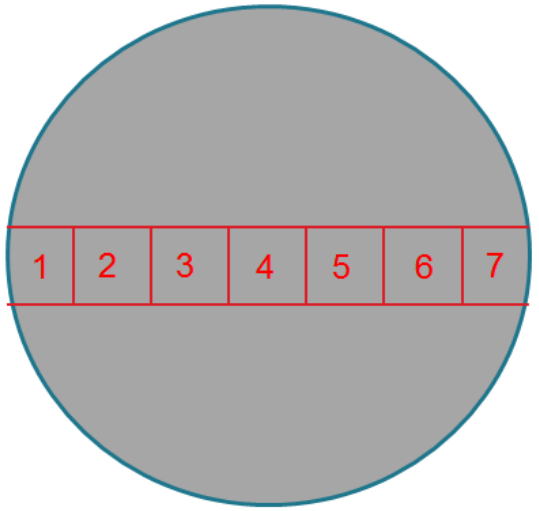
- A precisely cut seed crystal is dipped into the molten Ge and withdrawn slowly, while maintaining the temperature of the melt just above the melting point.

- The rate of crystal pulling, and the thermal field are adjusted to control the growth of the crystal.

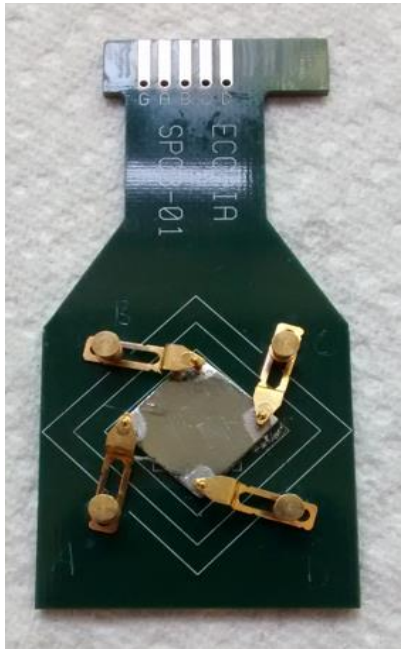
# Hall Effect Measurement




- Van der Pauw Hall effect measurement system
  - Resistivity $\rho = R_s \cdot \mathrm{d}$
  - Hall Voltage $V_H = -\frac{IBw}{nq\mathrm{d}w} = -\left(\frac{1}{nq}\right)\frac{IB}{d}$
  - Hall Mobility $\mu = \frac{1}{qn\rho} = \frac{|V_H d|}{IB\rho}$
  - Carrier Concentration: $n = -\frac{IB}{qtV_H}$



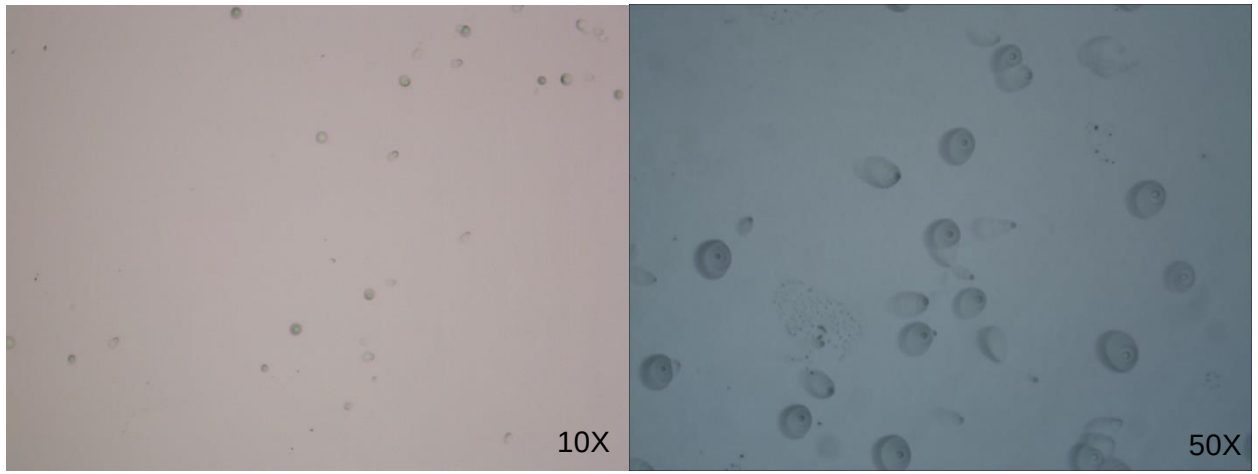

- Schematic diagram showing the location of the samples cut from the wafers.

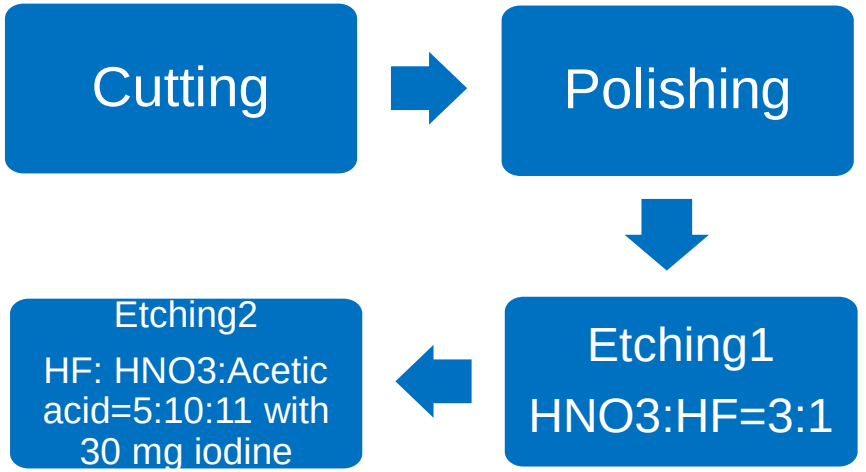


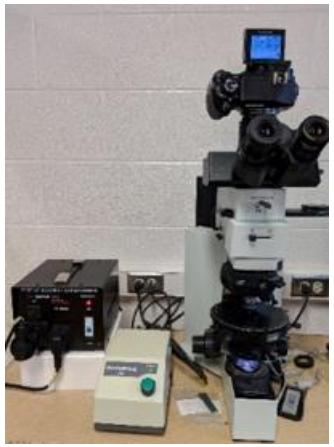
- The four corners of the sample are scratched by ohmic contacts. Depends on the conductivity type of the crystal, the contacts are different.[1]

[1]. *M.-S. Raut, et al., Journal of Instrumentation, Volume 15, October 2020*

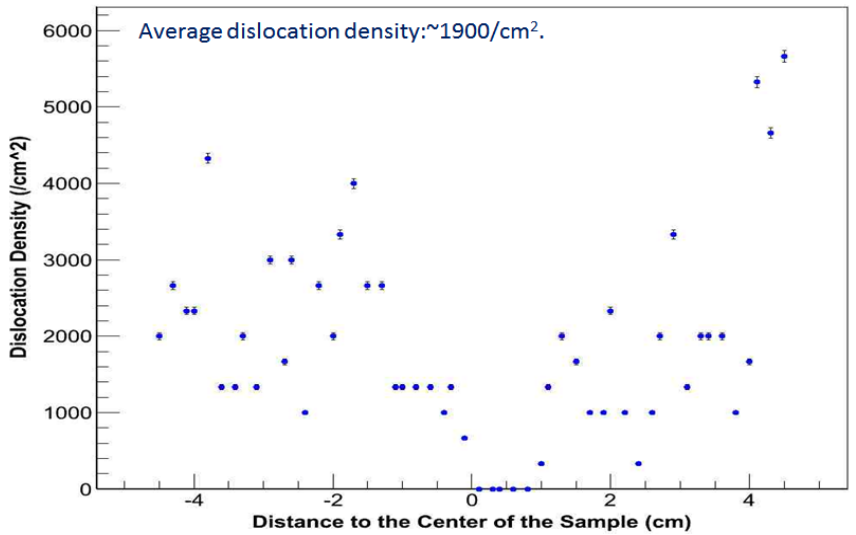
# Dislocation Measurement

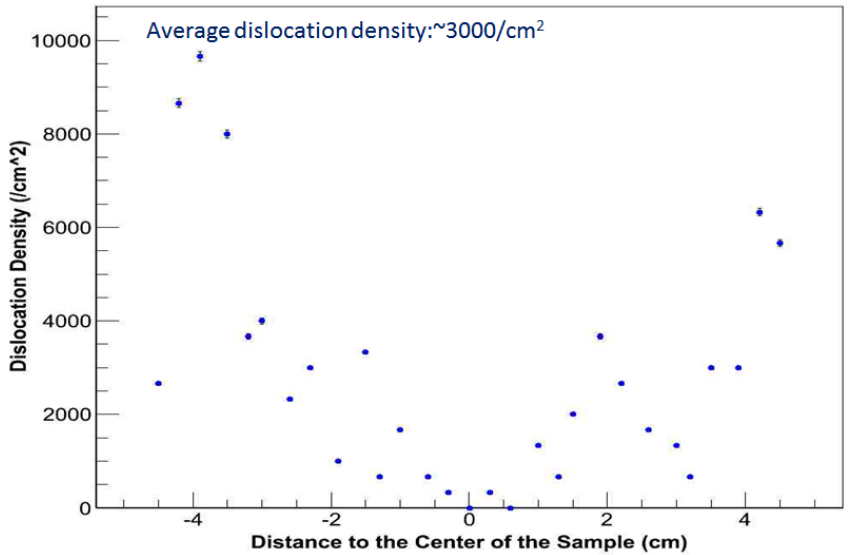


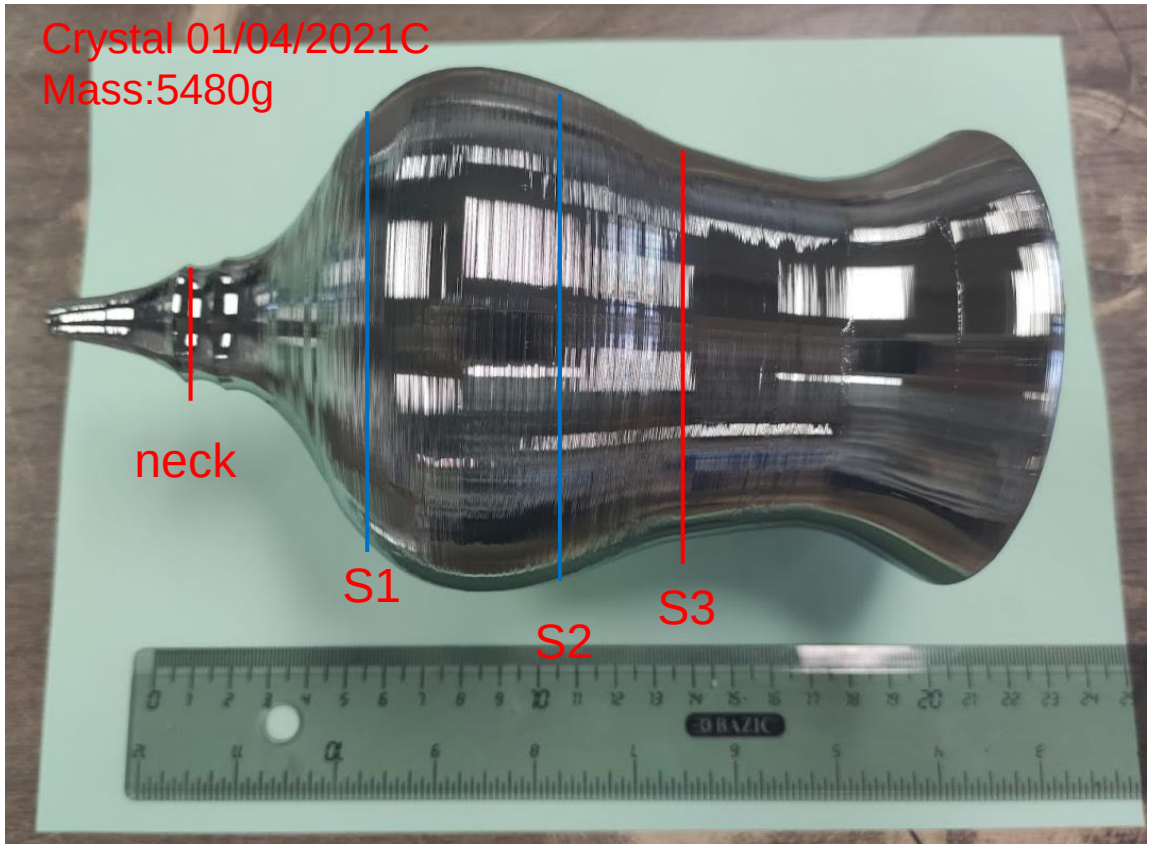
- Dislocations observation under microscope



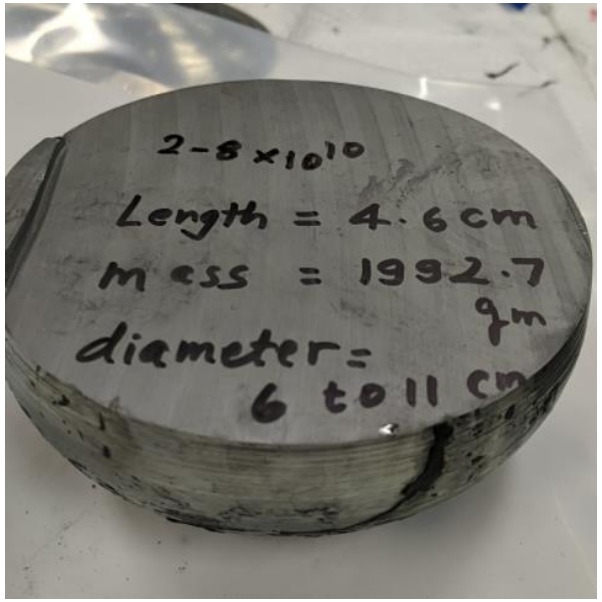

- Full view of the dislocation distribution on the whole slice

# Crystal Growth at USD




| Position | Concentration $(/cm^3)$ | Mobility $(cm^2/Vs)$ | Resistivity $(\Omega cm)$ |
|---|---|---|---|
| Neck | 2.06E12 | 4.15E4 | 7.31E1 |
| S1 | 3.61~8.86E10 | 5.85E3~2.61E4 | 6.63E3~1.21E4 |
| S2 | 2.57~8.93E10 | 2.95~3.58E4 | 1.95~8.24E3 |
| S3 | -3.94~-5.33E11 | 6.71E3~1.20E4 | 9.76E2~2.36E3 |
| Tail | - | - | - |

- High-purity germanium crystals with diameters up to 12 cm were grown by the Czochralski method. Red lines shows the position of the cutting slices for characterization of the HP-Ge crystal.




Detector grade crystal:

- Mass: 1922g
- Thickness: 4.6cm
- Diameter:11cm

# Crystal Growth at USD

- To enhance the chance to have a detector-grade region from the grown crystal, we have been trying to increase the mass of input materials. We have successfully increased the mass of the crystals from 3.5kg to 4kg for one of our crystal growers (grower A).
- Despite the thermal field for grower A is quite different from that in the other grower (grower C can grow crystal up to 6.2kg with diameter ~11cm), the diameters and shapes are controlled well, demonstrating our ability to grow HP-Ge crystals with large diameters.

- Crystal grew on 10/07/2021 from grower A with mass of 3957g.

- Crystal grew on 10/14/2021 from grower C with mass of 5283g.

# Crystal Growth at USD

- In addition, we are also investigating the effect of the chamber pressure on the thermal field and gas flow rate during crystal growth.



## Pros:

•Stable gas flow rate.

- The gas flow rate shows less fluctuation. Occasionally it will increase, barely decrease.
- Less chance to have poly, even with visible amount of graphite on melt surface.

•Less outside air backflow.

- Much more visible during crystal growth for grower C.
- The glass cylinder are cleaner than before.

## Cons:

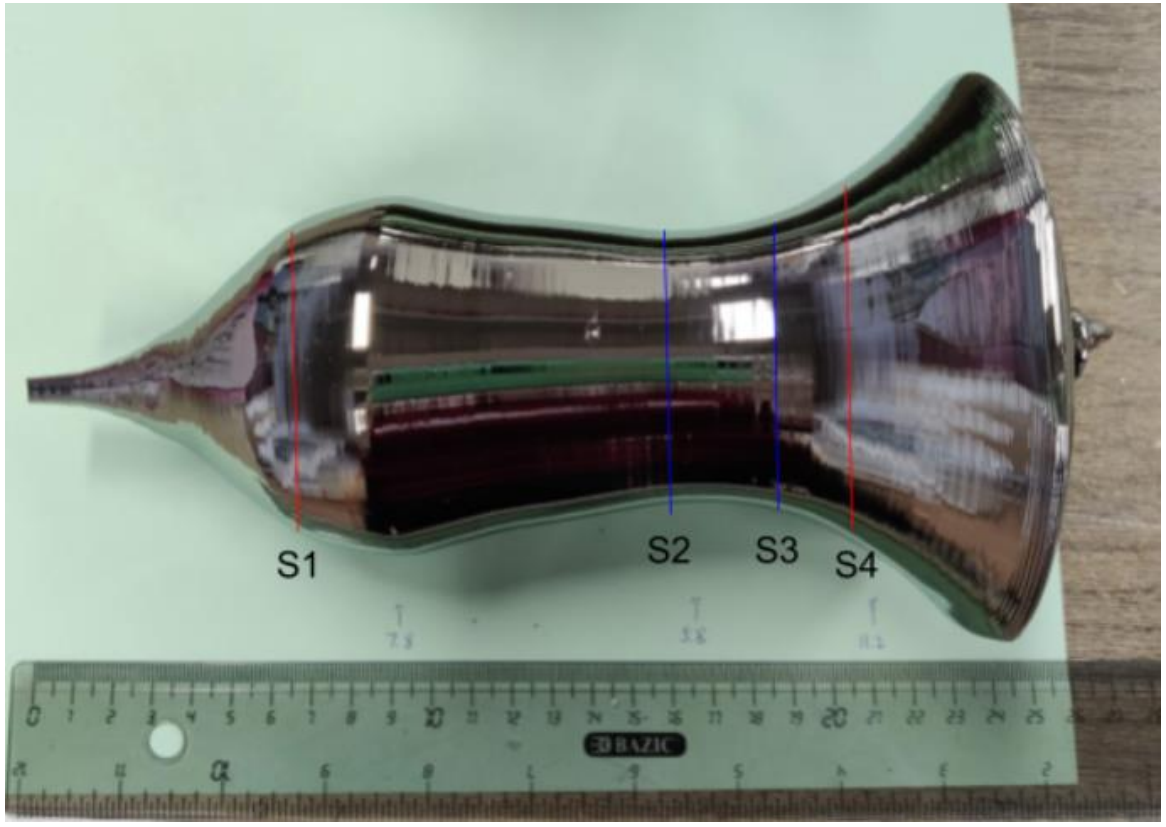
•Thermal field has been changed.

- Need more data to control the diameter.

•The digital scale gets disfunction when pressure > 6 psi.

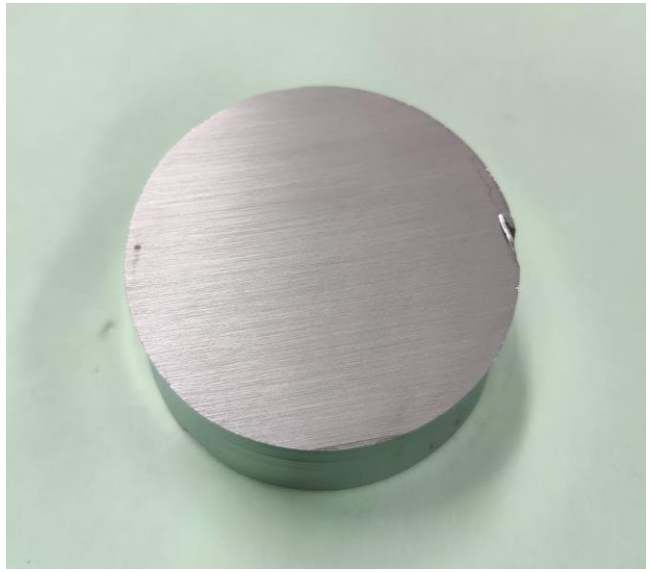
- Still looking for the best chamber pressure for crystal growth.

# Crystal Growth at USD



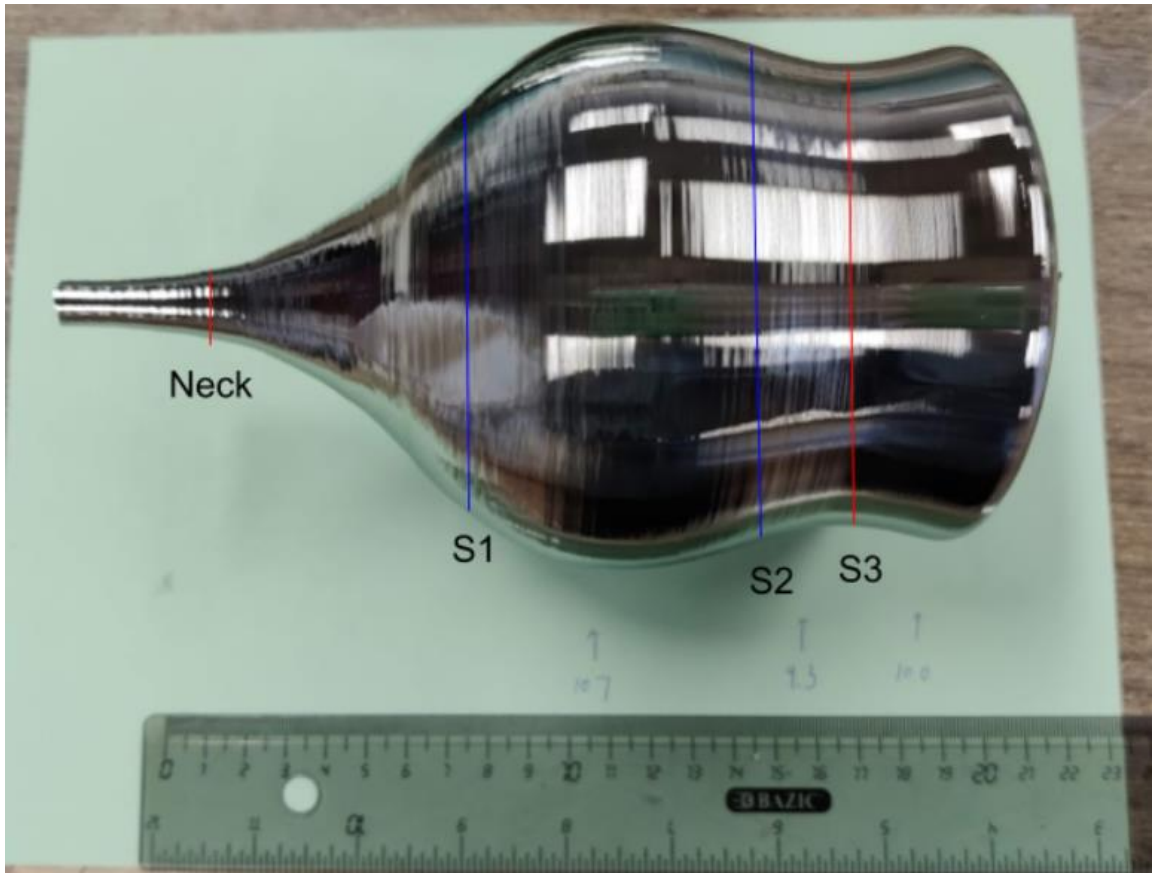

| Position | Concentration ($/cm^3$) | Mobility ($cm^2/Vs$) | Resistivity ($\Omega cm$) |
|---|---|---|---|
| Neck | 7.96E11 | 1.09E4 | 7.19E2 |
| S1 | 2.45~3.18E11 | 1.71~5.49E4 | 3.58E2~1.49E3 |
| S2 | 5.79~9.17E10 | 2.55~3.13E4 | 2.17~4.23E3 |
| S3 | -2.94~-5.53E10 | 2.43~3.74E4 | 4.64~5.69E3 |
| S4 | -5.51~-6.31E11 | 1.47~3.36E4 | 3.12~6.74E2 |
| Tail | -1.25E13 | 2.91E4 | 1.72E1 |

- Crystal on 03-31-2022A with a mass of 4050g. The detector grade region is at 55-60% location along the axial direction. The Hall effect measurements show the total impurity level of this crystal is slightly higher than we expected and further purification is needed.



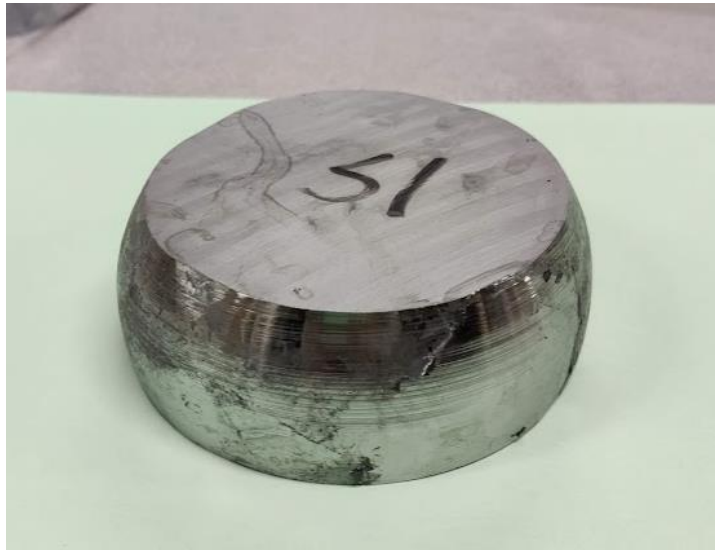
- Mass: 364g
- Diameter: 5.9cm
- Thickness:2.45cm

# Crystal Growth at USD



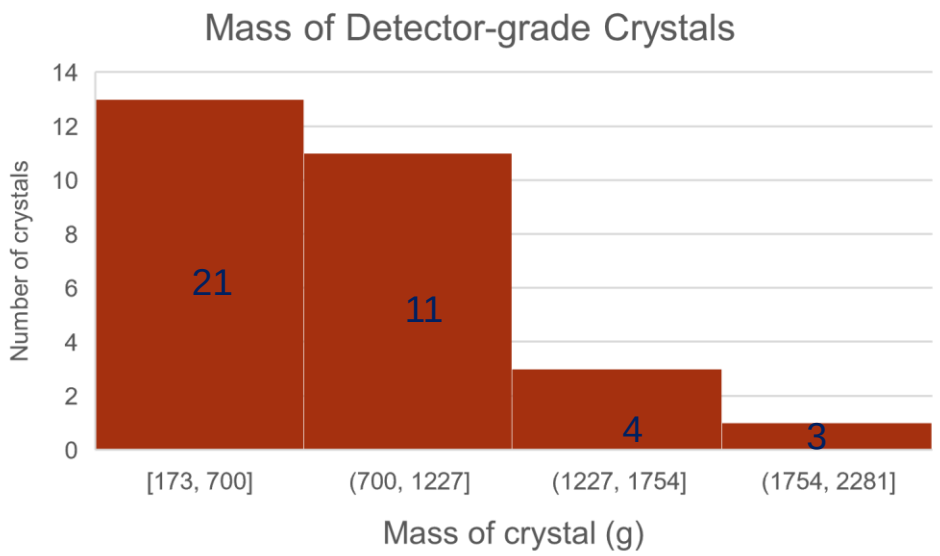

| Position | Concentration $(/cm^3)$ | Mobility $(cm^2/Vs)$ | Resistivity $(\Omega cm)$ |
|---|---|---|---|
| Neck | - | - | - |
| S1 | 1.34~5.22E10 | 2.38~4.49E4 | 3.11E3~1.57E4 |
| S2 | -5.21~-7.27E10 | 2.43~2.98E4 | 3.53~4.02E3 |
| S3 | -1.60~-2.42E11 | 2.34~3.22E4 | 1.21E3 |
| Tail | -8.69E12 | 1.63E4 | 4.41E1 |

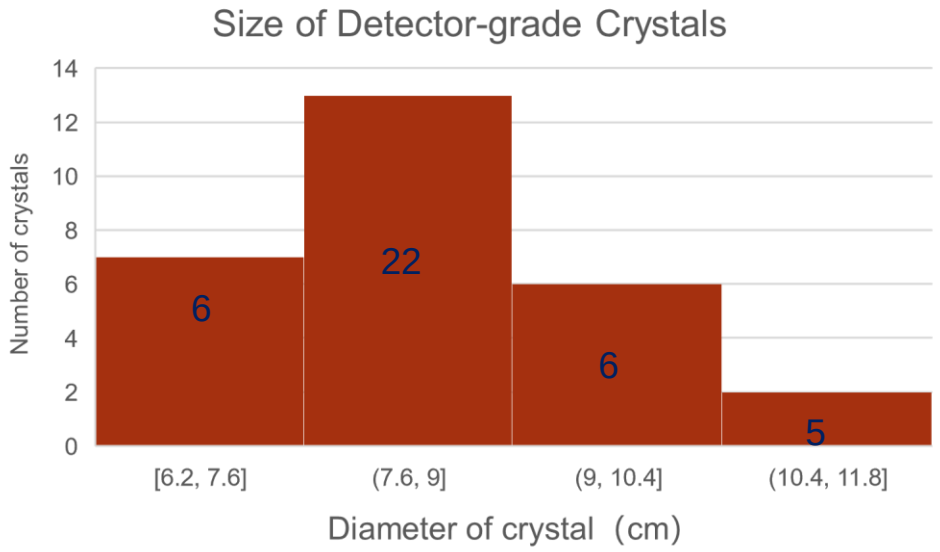
- Crystal on 04-07-2022C with a mass of 5398g. The detector grade region is at 20-55% location along the axial direction.




- Mass: 1929g
- Diameter: 11.1cm(bottom) 9.6cm(top)
- Thickness: 4.0cm

# Detector-Grade Crystals Summary

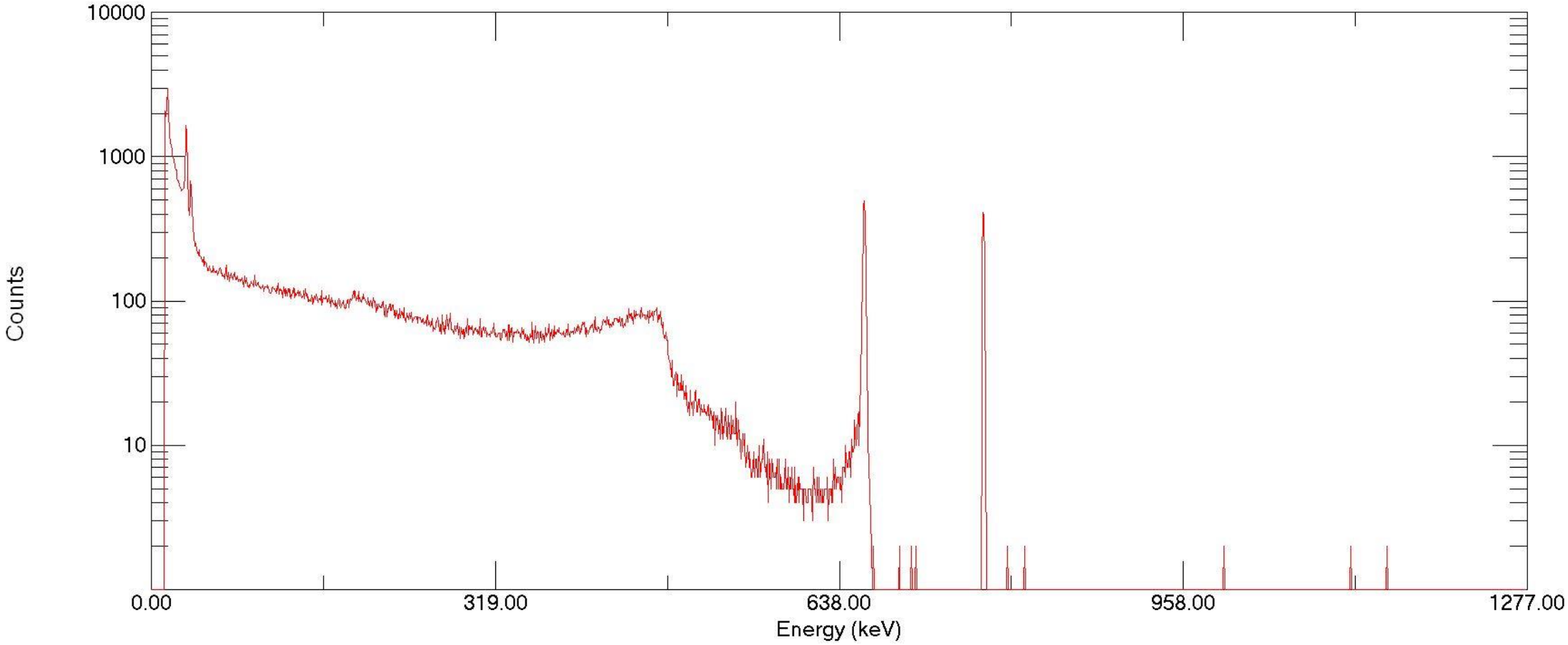



- 31.9kg detector grade crystals at USD:
  - 39 detector grade crystals. 3 detector grade crystals (2928g) this year so far.
  - Impurity level: $3.77 \times 10^{9} \sim 7 \times 10^{10}/cm^{3}$.
  - Average diameter: 8.5 cm.
  - Average thickness: 2.5 cm.

- Mass: 2247g
- Diameter: 10cm
- Thickness: 5.4cm
- Impurity Level: $2.06 \times 10^{10}(top) \sim 2.68 \times 10^{10}/cm^{3}\,(bottom)$

# Energy Spectrum of Cs-137




- Energy spectrum from a Cs-137 source measured with the detector USD-R03. The source was positioned facing the detector top. The bias voltage of -2500 V was applied to the bottom electrical contact on the detector while the signals were measured from the top.

# Conclusions

- At USD, we have successfully built a production chain that can purify the Ge raw materials, grow detector-grade HP-Ge crystals, and fabricate them into Ge detectors.
- A well-controlled segregation method and thermal field are used to control the distribution of impurities during crystal growth to meet the requirements for detector-grade Ge crystals.
- We are investigating some essential parameters to improve the quality of the crystal; we will continue to investigate and develop the influential parameters for the entire HP-Ge development chain.

# Thanks!