

# Crystal Growth of Cesium Hafnium Chloride ( $\text{Cs}_2\text{HfCl}_6$ ) Scintillating Crystal



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## Introduction

Crystal growth is the process where crystalline substance forms and increases in size through the addition of atoms, ions, or molecules. In this research we used the Bridgman technique to grow Cesium Hafnium Chloride ( $\text{C}_2\text{HfCl}_6$ ).

This technique is ideal for growing large volume crystal. Using a vertical furnace with two heating zones for heating and cooling, the material solidifies into a crystal as it slowly transitions from hot to cold zone.

## Methods

### Vertical Bridgman-Stockbarger Method

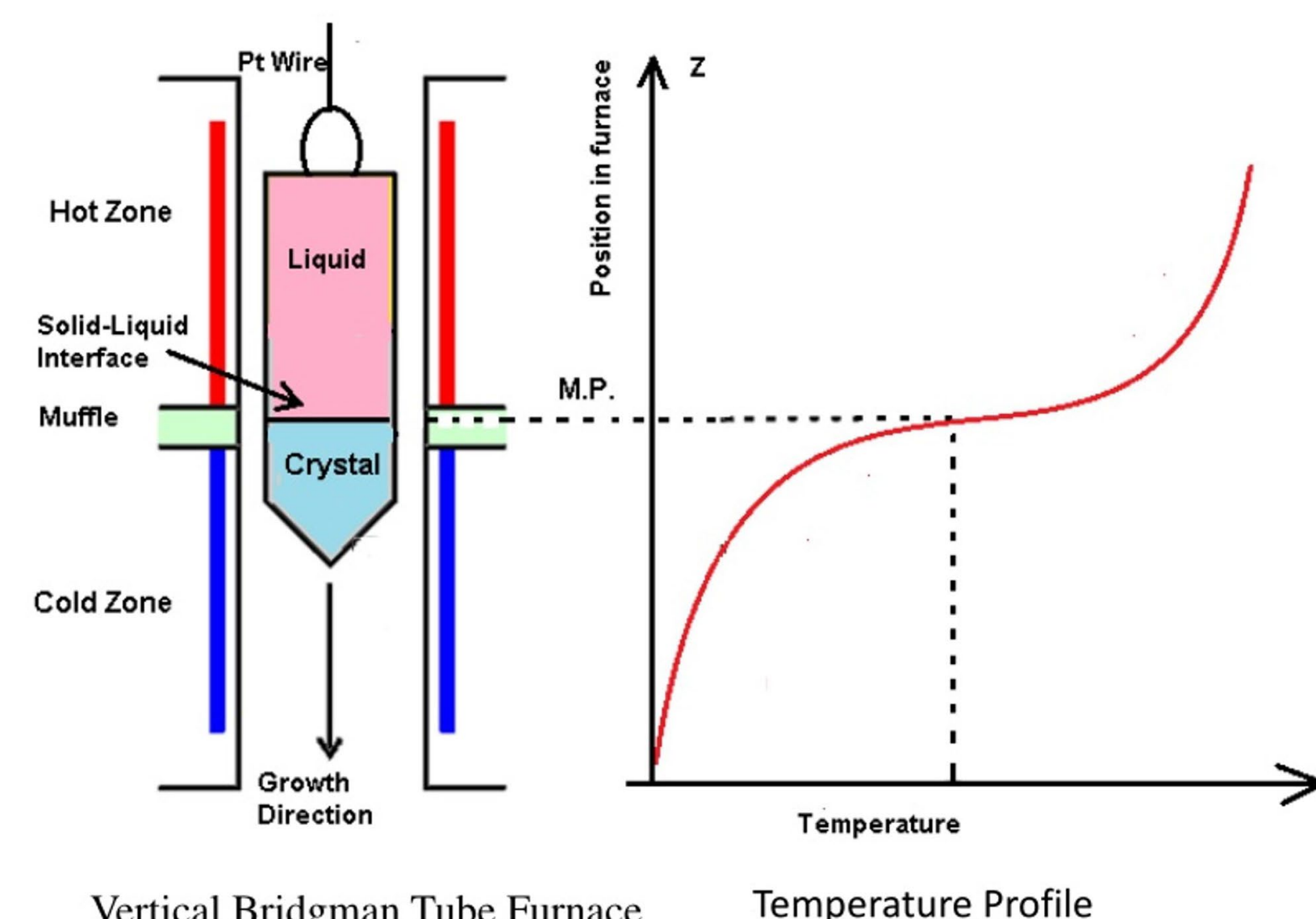


Figure 1: Graphical depiction of Vertical Bridgman-Stockbarger Method & Temperature Profile.<sup>1</sup>

CHC was fabricated from the starting material of Cesium Chloride and Hafnium Chloride in various processes. The vertical Bridgman-Stockbarger high temperature melt growth growing technique used is the vertical Bridgman method.

- Stage 1: Load materials  $\text{HfCl}_4$  into an ampoule for purification in the glove box for sublimation under vacuum. Load (amount) of  $\text{CsCl}$  into ampoule along with purified  $\text{HfCl}_4$  in glove box.
- Stage 2: Evacuate impurities in ampoule under vacuum and seal ampoule for growth.
- Stage 3: Measure the temperature within the growth furnace to determine temperature profile.
- Stage 4: Place into the furnace with each zone set at specific temperatures.

A single crystal was produced and tested as a scintillator to detect gamma particles.

## Crystal Growth Process

### MBraun Glove Box Condition:



Figure 2: Two station MBraun glove box.

- Moisture Level ( $\text{H}_2\text{O}$ ) < 0.01 ppm
  - Oxygen Level ( $\text{O}_2$ ) < 0.01
  - Atmospheric Pressure < 2.3 mbar
- $\text{HfCl}_4$  and  $\text{CsCl}$  are loaded into ampoule due to the material being hydroscopic.

### Temperature Zones of Vertical Furnace:



Figure 4: Bridgman furnace.

Zone 1: 877°C

Zone 2: 0°C

Zone 3: 757°C

The zones are specific to melt the material and to cool them for crystallizing to produce  $\text{Cs}_2\text{HfCl}_6$ .

### Growth Parameters

- Material Melting Point - 821°C – 822°C for Cesium Hafnium Chloride ( $\text{Cs}_2\text{HfCl}_6$ )
- Growth Rate – 2.5 cm/day
- Cooling Rate – 100°C/day

### Mellen NACCI 6”:

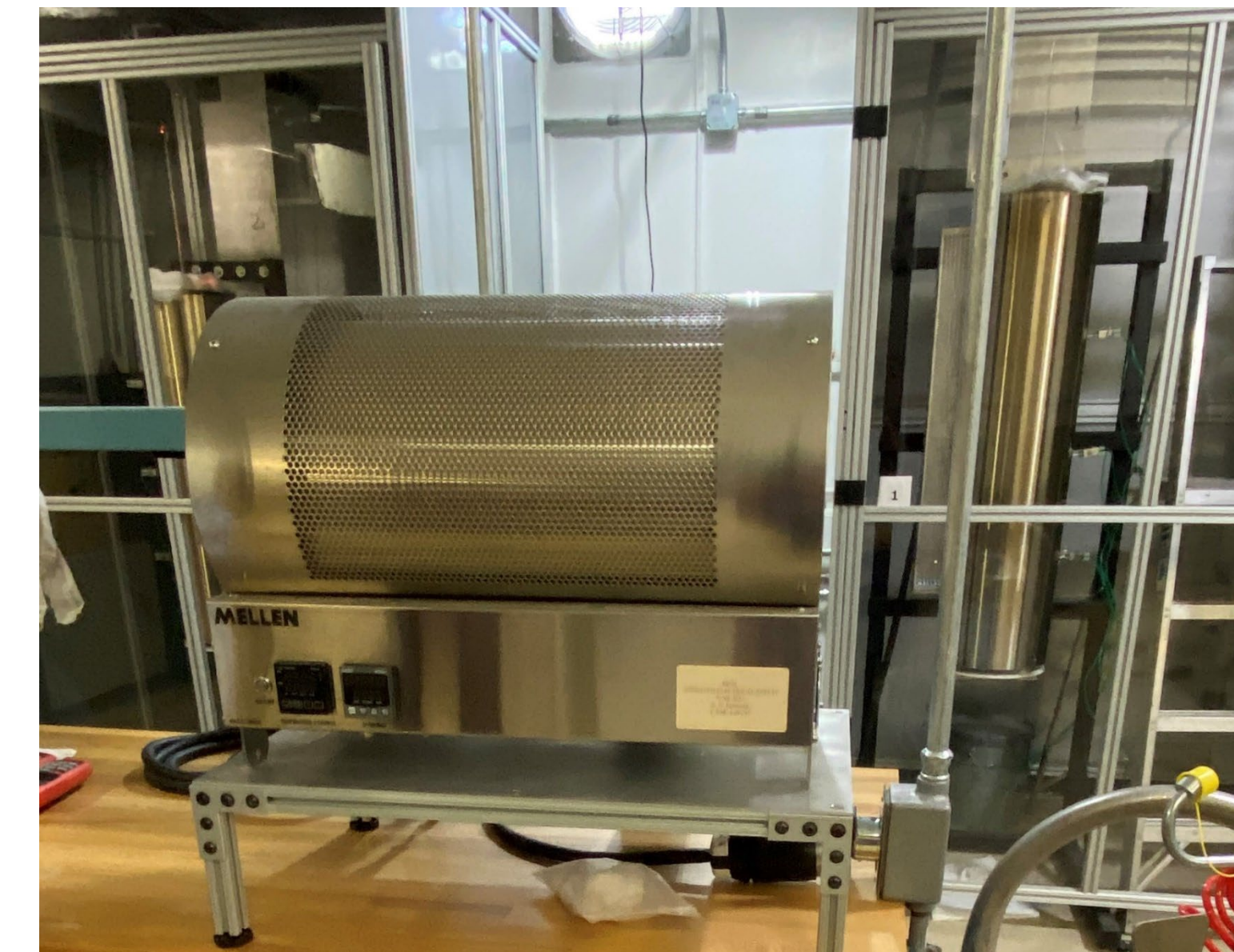


Figure 3: Tube furnace.

Sublimation is performed by heating part of the ampoule containing  $\text{HfCl}_4$  at 320°C. The purified  $\text{HfCl}_4$  sublimates at the cooler end of the ampoule, leaving impurities behind.

### Edwards T-Station:

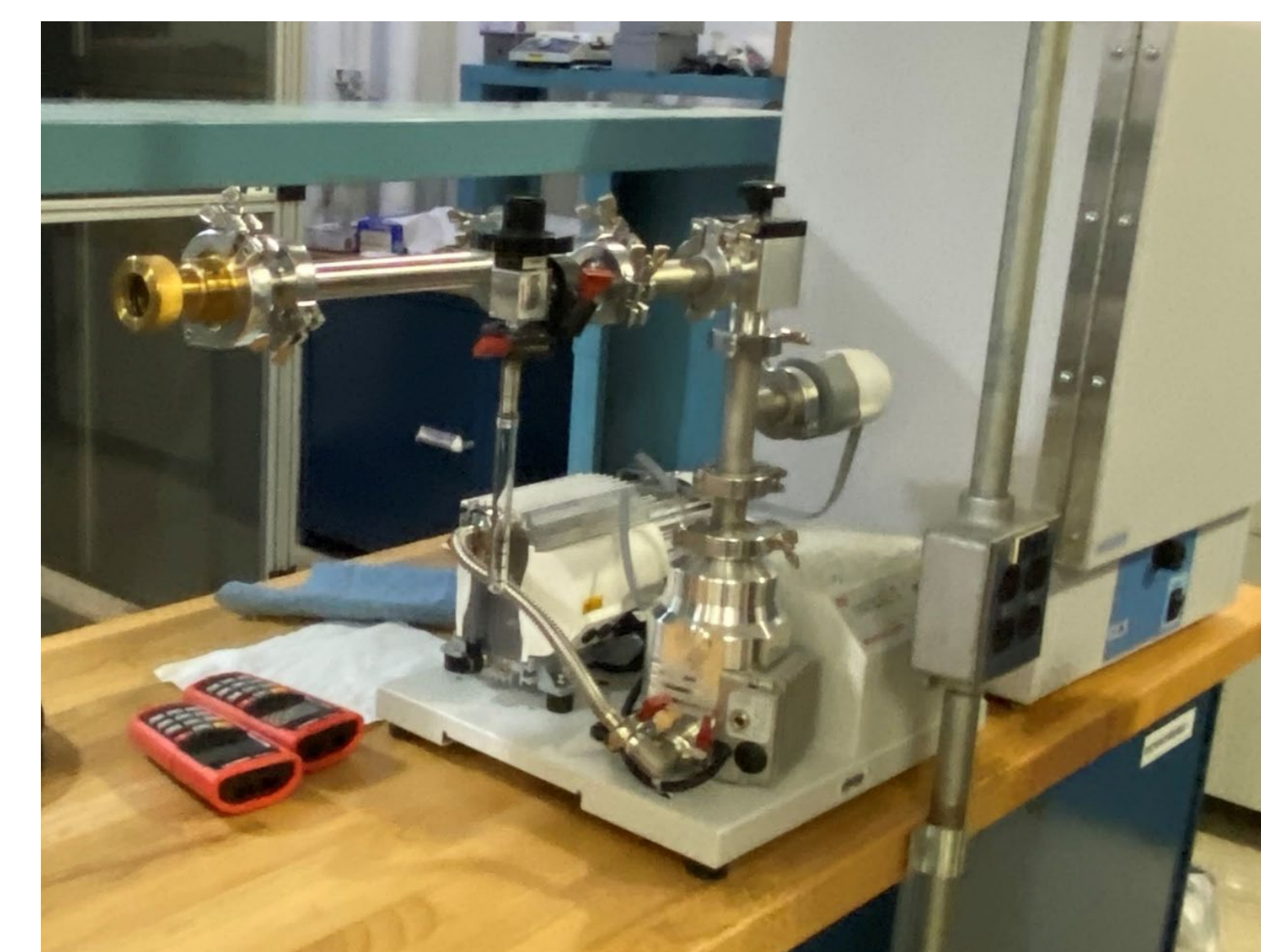


Figure 5: Stationary vacuum.

The materials were evacuated under vacuum to remove moisture at  $10^{-6}$  torr.

## Results



Figure 6: Example of CHC grown at Alabama A&M.

A 1 cm diameter ingot cut out from grown CHC crystal. Polishing the crystal with mineral oil creates the milky substances around it.



Figure 7: The polished crystal of CHC grown at Alabama A&M.

Fabricated CHC crystal detector from a single crystal ingot. After proper polishing, the crystal is visibly transparent.

## References

Float Zone & Bridgman Crystal Growth Techniques. 1. Abu Syed Md. Jannatul Islam Lecturer, Dept. of EEE, KUET, BD. Department of Electrical and Electronic Engineering Khulna University of Engineering & Technology Khulna-9203. Limitations of CZ Method. 2. December 19<sup>th</sup>, 2019. <https://image4.slideserve.com/9086359/vertical-bridgman-stockbarger-method-l.jpg><sup>1</sup>

SRNL-STI-2024-00309

## Acknowledgements

Funding provided from NNSA-MSIPP for the proposal entitled: *MSIPP Partnership for Radiation Studies (PaRS) Consortium*.

Funded by the NNSA MSIPP Pars Award No. DE-FOA-0002494, DOE/BSRA SRNL, LLC.; 10/01/2022 – 09/30/2027.



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