

Crystal Growth of Cesium Hafnium Chloride (Cs_2HfCl_6) Scintillating Crystal

Jalen Tribble* (presenter), Angel Reeder*, Aaron Johnson*, Samuel Uba**, Steven Demers**, Martine Duff**, Stephen Babalola*, Utpal Roy**
Alabama A&M University*, Savannah River National Laboratory**

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 (Cs_2HfCl_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

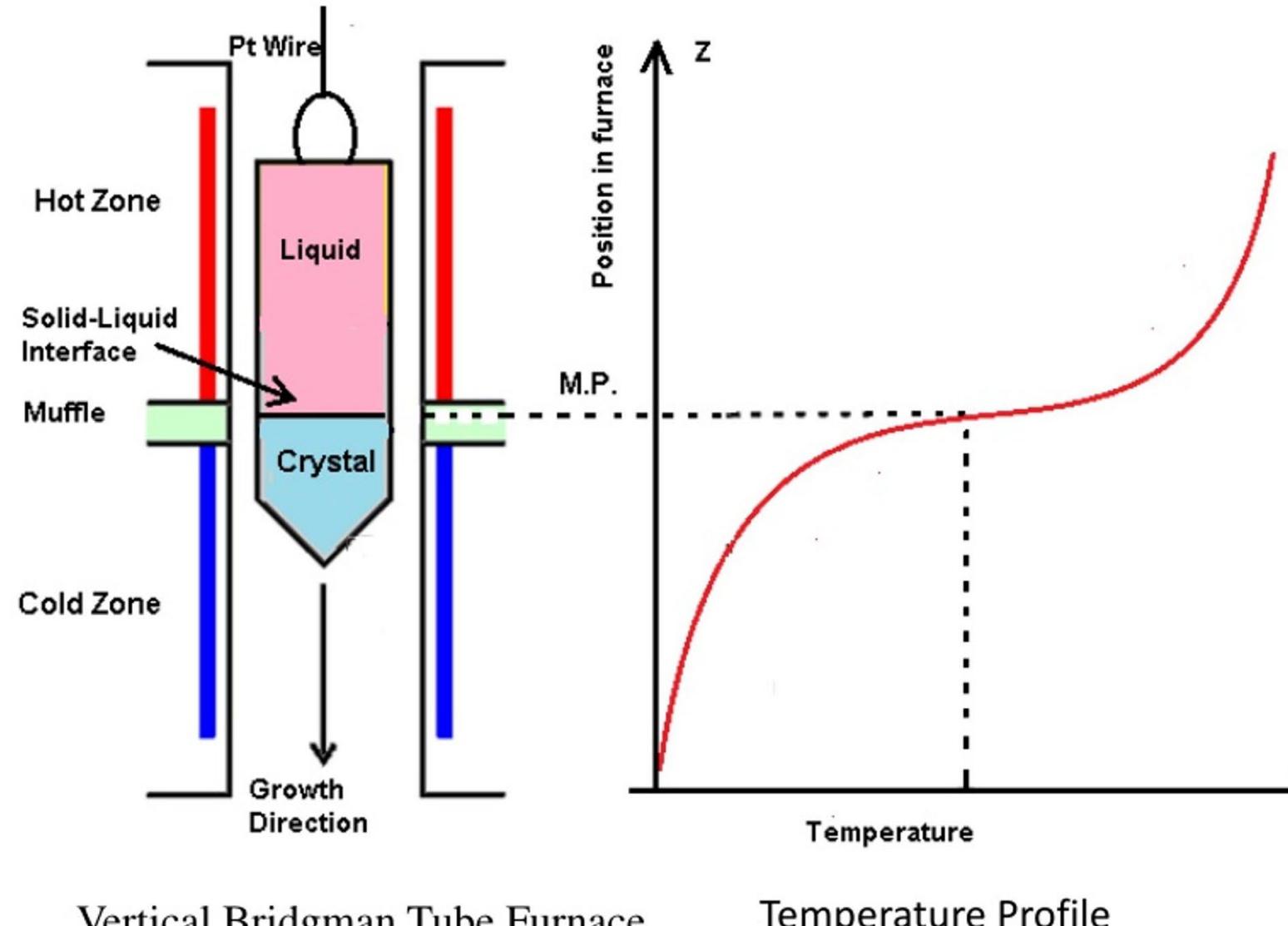


Figure 1: Graphical depiction of Vertical Bridgman-Stockbarger Method & Temperature Profile.¹

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 HfCl_4 into an ampoule for purification in the glove box for sublimation under vacuum. Load (amount) of CsCl into ampoule along with purified 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 (H_2O) < 0.01 ppm
- Oxygen Level (O_2) < 0.01
- Atmospheric Pressure < 2.3 mbar

HfCl_4 and 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 Cs_2HfCl_6 .

Growth Parameters

- Material Melting Point - 821°C – 822°C for Cesium Hafnium Chloride (Cs_2HfCl_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 HfCl_4 at 320°C. The purified HfCl_4 sublimes 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.



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

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 19th, 2019. <https://image4.slideserve.com/9086359/vertical-bridgman-stockbarger-method-1.jpg>¹

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.



Savannah River National Laboratory®