# Optical Floating Zone (OFZ) Furnace Manual to Grow Single Crystals Within Quartz Ampules

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*Crystal Systems Corporation FZ-T-2000-H-NI-VPO-PC*

# Contents

# 1 Introduction

This manual provides instructions for operating the Optical Floating Zone (OFZ) Furnace in a specialized configuration: processing samples sealed within quartz ampules to grow single crystals. In this approach, the sample is heated and translated, but it does not "float" in the traditional sense. Instead, it remains fully contained within a quartz ampule suspended in the furnace.

This technique differs from the standard floating zone method, which suspends and translates a feed rod to create a traveling molten zone. In contrast, the ampule-based approach uses focused infrared light to generate and manipulate a molten zone inside the sealed ampule, enabling directional solidification and single crystal growth. This method is suitable only for samples with melting points below the deformation temperature of quartz.

The following sections outline the setup, interface navigation, safety protocols, and operational procedures required to successfully grow single crystals using this ampule-based translation method.

# 2 Safety Considerations

- Do not touch the ampule or surrounding components immediately after exposure to the halogen lamps. Quartz can retain extremely high temperatures and cause severe burns.
- Always wear gloves when handling the ampule or interacting with furnace components.
- Never leave the furnace unattended while in operation. At a minimum, ensure the furnace PC is networked to another computer so the internal camera feed can be monitored remotely.

# 3 Equipment Overview

This manual is based on the FZ-T-2000-H-NI-VPO-PC model manufactured by Crystal Systems Corporation. This is a high-performance Optical Floating Zone Furnace designed for advanced sample processing, including single crystal growth, under precisely controlled heating and translational conditions.

## 3.1 Furnace Specifications

- **Maximum Operating Temperature**: $2200\,^{\circ}\mathrm{C}$
- **Normal Operating Temperature**: $1800\,^{\circ}\mathrm{C}$
- **Lamp Max Power**: $4000\,\mathrm{W}$
- **Cooling System**: Air-cooled
- **Maximum Crystal Growth Length**: $120\,\mathrm{mm}$
- **Upper Shaft Moving Length**: $50\,\mathrm{mm}$

- **Mirror Stage Slow Moving (Growth Speed)**: $0.1\,\mathrm{mm/hr}$ – $100\,\mathrm{mm/hr}$
- **Mirror Stage Fast Moving (Positioning/set-up)**: $6\,\mathrm{mm/min}$ – $70\,\mathrm{mm/min}$
- **Upper Shaft Slow Moving (Growth Speed)**: $0.1\,\mathrm{mm/hr}$ – $100\,\mathrm{mm/hr}$
- **Upper Shaft Fast Moving (Positioning/set-up)**: $6\,\mathrm{mm/min}$ – $70\,\mathrm{mm/min}$
- **Shaft Rotation Speed**: $5\,\mathrm{rpm}$ – $60\,\mathrm{rpm}$

## 3.2 Internal Components

- **Halogen Lamps**: Four high-intensity lamps provide localized infrared heating, focused by ellipsoidal mirrors.
- **Mirror Assembly**: Four ellipsoidal mirrors concentrate light onto the ampule to generate a molten zone.
- **Upper Shaft**: Securely holds the ampule in place and can be moved vertically.
- **Mirror Stage**: The mirror stage can be moved vertically to adjust the position of the molten zone.
- **Cooling Fans**: Dedicated fans provide cooling for the ellipsoidal mirrors (4 fans), upper shaft holder (2 fans), and lower shaft holder (2 fans) to maintain safe operating temperatures.
- **Observation Unit**: The molten zone can be observed and monitored through the CCD camera and LCD monitor (color).

## 3.3 Control and Monitoring Systems

- **Control Board**: Software interface for adjusting lamp power and tracking process parameters (see Section Section 5.1 for details).
- **Hand Control Box**: Adjustable power output with real-time feedback (see Section Section 5.2 for details).
- **Power Switch Controls**: The furnace is equipped with four power switches: main power, lamp power, control power, and PC power (see Section Section 5.3 for details).
- **Camera System**: Internal furnace camera linked to the control board for monitoring the ampule and molten zone.

## 3.4 Auxiliary Equipment

- **Chamber**: A quartz tube that shields the quartz ampule from ambient airflow, helping to prevent swaying and maintain stable conditions during operation. A low-pressure type is used, as no internal gas sealing is required. See Section 4.1 and 9 in the official Operation Manual by Crystal Systems Corporation for more information on the quartz tube.

- **Quartz Ampule**: A sealed quartz container, typically evacuated or backfilled with inert gas, used to house the sample during processing.
- **Suspension Fixture**: Includes a feed holder (hook) used to suspend the ampule, typically with high-temperature wire such as nichrome or aluminum.
- **Filter**: Two kinds of filter glasses are available for the camera. One is #10 (dark) and the other is #8 (light).

# 4 Sample Preparation

The following steps outline the general procedure for preparing a sample inside the quartz ampule for use in the OFZ furnace.

1. **Create the sample material:**
   Measure high-purity elemental powders using an analytical balance (accuracy of 0.01%), based on the desired stoichiometry. Homogenize the powder mixture using a mortar and pestle or ball mill. Accurate stoichiometry and thorough mixing help minimize formation of secondary phases.
2. **Seal the material in a quartz ampule:**
   Transfer the powder into a clean, dry quartz ampule. Connect the ampule to a vacuum line and evacuate as required. Optionally, backfill with inert gas (e.g., argon) and evacuate again to improve gas purity. Seal the ampule using an oxygen-hydrogen torch while under vacuum or inert gas pressure.
3. **Melt and homogenize the sample:**
   Place the sealed ampule in a box furnace. Heat it to a temperature above the melting point of the sample. Hold for several hours to allow complete melting and homogenization. Cool the ampule slowly to room temperature to prevent thermal stress.
4. **(Optional) Perform XRD analysis:**
   If phase verification is needed, break the ampule open in a fume hood. Crush the solidified material into fine powder using a mortar and pestle. Perform X-ray diffraction (XRD) to confirm phase purity and identify any secondary phases.
5. **Re-seal the sample for crystal growth:**
   Transfer the verified powder or bulk material into a new quartz ampule. Ensure the ampule has:
   - A sharp conical tip at the bottom to aid directional solidification.
   - A hooked top for suspension in the OFZ furnace.
   - A length and diameter compatible with the furnace geometry.

   Evacuate and seal the ampule as done previously.

# 5 Interface Walkthrough

This section provides an overview of both the physical and software interface components used to control the OFZ furnace. Each subsection references a labeled photo, where numbers correspond to the described parts and functions.

## 5.1 Control Board

The image below shows the layout of the control board, with numbered callouts corresponding to the components described in the list that follows. The control board contains physical switches, knobs, and indicators used to operate and monitor the OFZ furnace.

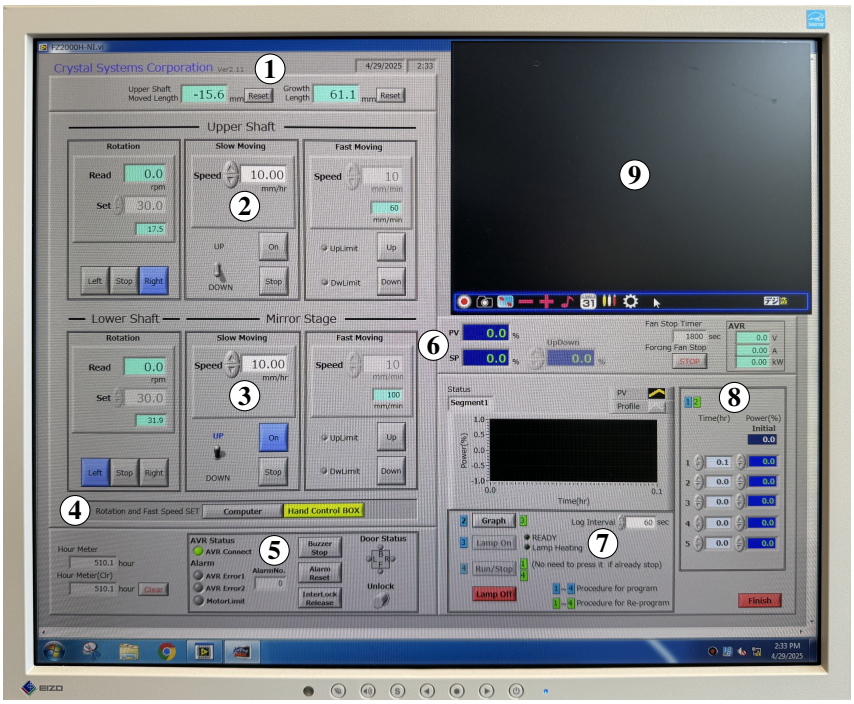


Figure 1: Control board (call-outs numbered in text).

If the interface does not appear automatically after PC boot, manually launch both the *FullPlay-Rock* and *FZ2000H-NI* applications from the desktop. If the monitor remains black after startup, ensure the display is powered on.

① **Upper Shaft Movement & Growth Length** — Displays the distance the upper shaft translation (negative = up, positive = down) and the mirror stage translation (positive = up, negative = down). Growth length refers to mirror stage movement, as it is the primary mechanism used during actual crystal growth.

② **Upper Shaft Controls** — Controls for fast movement, slow movement, and rotation. Fast movement is used for furnace setup and positioning. Rotation is used during crystal growth. Slow movement of the upper shaft is typically not used for ampule-based growth.

③ **Mirror Stage & Lower Shaft** — Mirror stage has fast and slow movement controls. Use fast movement for positioning and slow movement during growth. Ensure the mirror

stage is initially low enough to allow upward travel during the run. Lower shaft rotation is unused in ampule-based growth.

④ **Rotation and Fast Speed SET** — Selects whether rotation and fast movement are controlled by the Computer (i.e., the control board) or the hand control box. If Hand Control Box is selected, the physical controls on the box override the corresponding functions on the control board, which will appear greyed out. If Computer is selected, control is returned to the control board and the hand control box becomes inactive. In Figure 1, Hand Control Box is currently selected.

⑤ **Status Display** — Displays any alarms, errors, or system messages. Red indicators mean the furnace is not ready. The front door can also be unlocked here by clicking the *Unlock* button. See Section 7 for troubleshooting.

⑥ **PV & SP** — PV (lamp output value) shows current lamp power output. SP (lamp output set value) shows the target lamp power. These values should generally match during stable operation.

⑦ **Graph & Program Control** — Includes buttons to graph the ramp schedule, toggle the lamp on/off, and start/stop the run. The Run/Stop button allows temporary pauses and mid-run adjustments.

⑧ **Power Ramp Schedule** — Defines the programmed lamp power settings. The "Initial Power" sets the baseline value, and all subsequent values are offsets added to this baseline. See Appendix B for example configurations.

⑨ **Live Camera Feed** — Displays a real-time view of the ampule and molten zone. Additional controls for recording and camera settings are available at the bottom of the window.

## 5.2 Hand Control Box

The image below shows the hand control box layout, with numbered callouts corresponding to the controls described below.

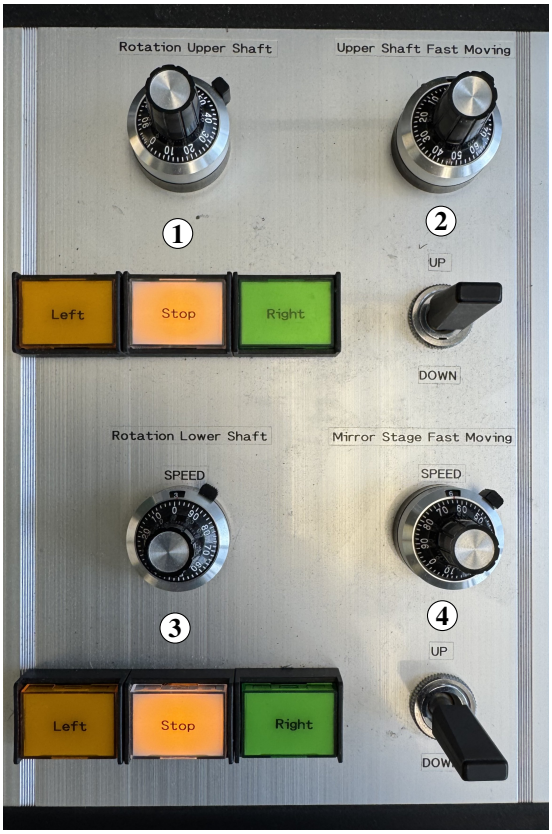


Figure 2: Hand control box (call-outs numbered in text).

The hand control box provides a physical interface for fast movement and rotation. It is primarily used during setup, especially when the furnace front panel is open. It only works when the "Hand Control Box" option is selected on the control board (see *Rotation and Fast Speed SET* above).

① **Upper Shaft Rotation** — Controls the rotation speed and direction of the upper shaft. Use the dial to set speed, and the buttons below to start/stop in either direction.

② **Upper Shaft Fast Moving** — Controls vertical positioning of the upper shaft. Use the dial to set speed. The up/down switch moves the shaft (up = lower the shaft, down = raise the shaft).

③ **Lower Shaft Rotation** — Controls lower shaft rotation. Similar to upper shaft: adjust speed with the dial and direction with the buttons. Lower shaft rotation is unused in ampule-based growth.

④ **Mirror Stage Fast Moving** — Controls vertical positioning of the mirror stage. Use the dial to set speed. The up/down switch moves the mirror stage (up = raise the mirror, down = lower the mirror).

## 5.3 Power Switch Controls

The image below shows the power switch control panel, with numbered callouts corresponding to the controls described below.


Figure 3: Power switch control panel (call-outs numbered in text).

These switches control power to all major systems in the furnace.

① **Main Power Switch** — Supplies power to the entire furnace system.

② **Lamp Power Switch** — Controls power to the halogen lamps.

③ **Control Power Switch** — Powers the control electronics and user interface.

④ **PC Power Switch** — Powers the dedicated PC (control board).

**Startup Procedure**: Activate the switches in the order ①, ②, ③, ④ to turn on the furnace.
**Shutdown Procedure**: Deactivate the switches in reverse order ④, ③, ②, ① to shut down the furnace.

# 6 Operation procedure

The following steps outline the general procedure for growing a single crystal using the OFZ furnace with a sealed quartz ampule.

1. **Turn on the OFZ furnace:**
   Turn on the power switches in the following order: [1] main power, [2] lamp power, [3] control power, [4] PC power. See Section 5.3 for the switch layout.

2. **Prepare the quartz ampule:**
   Ensure the homogeneous sample is inside the ampule and either evacuated or backfilled with inert gas, as required. The ampule should have a sharp conical tip at the bottom to aid in single crystal growth, and a hooked top for suspension. See Section 4 for an in-depth sample preperation procedure.

3. **Load the ampule into the furnace:**
   Suspend the ampule from the upper shaft by securing it with high-temperature wire (nichrome or titanium recommended). Ensure the ampule hangs vertically and is centered.

4. **Install the quartz tube:**
   Place the outer quartz tube around the ampule to minimize airflow disturbances. See Section 4.1 and 9 in the official Operation Manual by Crystal Systems Corporation for more information on installing and using the quartz tube.

5. **Align the molten zone:**
   During all operations, the molten zone should begin just below the tip of the ampule and travel upward. Use the control software to position the camera view just beneath the ampule tip. Adjust the mirror stage and upper shaft so there is sufficient upward travel available for the growth run.

6. **Determine the appropriate power setting:**
   Begin at a power level corresponding to a temperature below the sample's melting point. Align the molten zone and briefly operate the furnace. Do not use any translation when completing these operations. Gradually increase the power by 1 – 3% increments until melting is observed (e.g., the sample shifts or flows downward). For reference, $Bi_2Se_3$ (melting point: $710\,^{\circ}\mathrm{C}$) begins to melt at approximately 34% lamp power. See Appendix B for PC configuration settings.

7. **Consolidate the sample at the bottom:**
   The material must be packed at the bottom of the ampule to enable directional crystal growth. Use the aligned molten zone and operate the furnace just above the melting power threshold until all sample settles. Repeat as necessary.

8. **Initiate single crystal growth:**
   Align the molten zone, start the lamp operation, and begin translation. Set the growth speed and rotation rate appropriate to the material ($0.1\,\mathrm{mm/hr}$ – $55\,\mathrm{mm/hr}$ growth speed and $5\,\mathrm{rpm}$ – $60\,\mathrm{rpm}$ rotation). Reset growth length on control board.

9. **Shut down the OFZ furnace:**
   Turn off the power switches in the following order: [4] PC power, [3] control power, [2] lamp power, [1] main power. See Section 5.3 for the switch layout.

# 7 Troubleshooting

This section outlines a few common errors that may occur during furnace operation. For most issues, refer to the Status Display on the control board to determine the error type.

- ***MotorLimit* indicator is red on the Status Display**:
  This error occurs when the upper shaft or mirror stage moves beyond its allowed travel range. When triggered, all motion controls (upper shaft, lower shaft, and mirror stage) will be disabled. To resolve this, press the *Alarm Reset* button on the control board.
- ***Door Status* indicator is red on the Status Display**:
  This means that one or more furnace doors are open, preventing the halogen lamps from being activated. To resolve this, open and close the affected door to relock it.

For other issues, please refer to the full *Optical Floating Zone Furnace* guide provided by Crystal Systems Corporation—specifically Section 3.3 of the PC Manual and Section 3.12 of the Operation Manual.

# 8 Best Practices & Operator Notes

The following notes outline best practices for ampule-based OFZ furnace usage, with a particular focus on improving single crystal quality.

- **Checks before turning on the furnace**:
  - Ensure the ampule hangs straight down. If tilted, rotation will disrupt the temperature gradient required for crystal growth. Rotating the ampule during this check makes misalignment more visible.
  - Verify the halogen lamp shutters are fully closed and locked before turning on the lamps. This is not visible from the outside—use the small window in the furnace door to confirm.
  - Move the mirror stage along the full length of the sample to ensure no translation limits are hit during operation.
- **Halogen lamp precautions**:
  - Ramping the halogen lamps up and down is essential for lamp longevity. Always allow at least 0.1 – 0.2 hours (6–12 minutes) for both ramp-up and ramp-down. Rapid thermal changes can stress the quartz envelope and lead to premature failure or cracking.
  - Minimize time when lamps are powered but not focused on a sample. Without a thermal target, lamps can reflect energy back onto each other, reducing lifespan.
  - Keep lamp lenses clean as dust or particles can cause localized hotspots on the bulb, increasing the risk of failure.

- **Other notes**:
  - After reaching the sample's approximate melting power, a small molten zone should appear within 1–3 minutes.
  - Do not use any translation when trying to determine the lamp power required for a specific sample.
  - When identifying the correct lamp power, record a video and scrub through it to spot subtle signs of melting (e.g., formation of a gap or slight downward flow).
  - Log each run's exact power ramp schedule, translation speed, and rotation speed. This makes future optimization and reproducibility much easier.

# Appendix

## A Furnace Photographs and Parts Location

Figures 4 through 7 show key furnace components and attachments for reference.

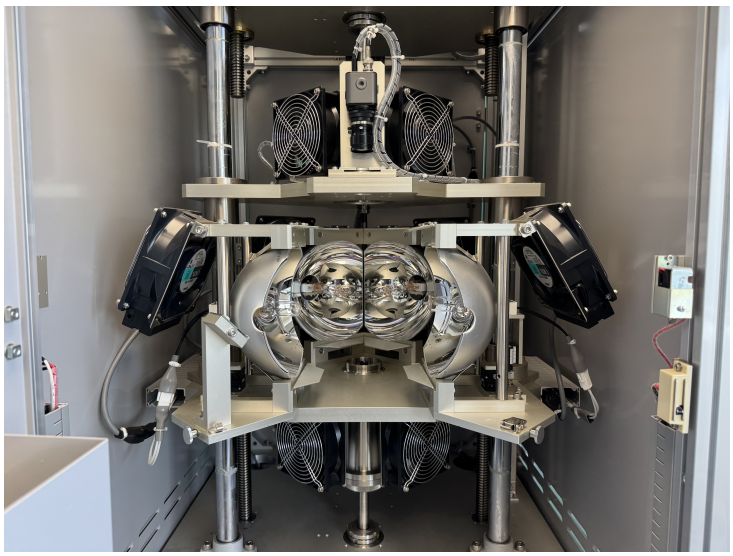

Figure 4: Mirrors in the open position.

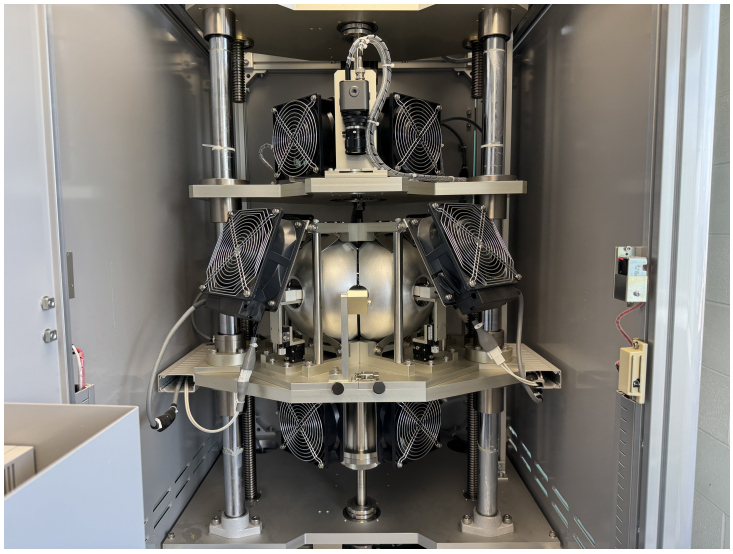

Figure 5: Mirrors in the closed position.

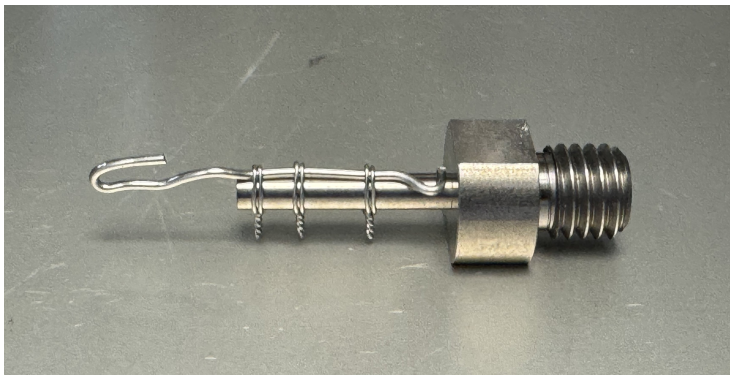

Figure 6: Top hook used for ampule suspension.

Figure 7: Ampule attachment to the hook with nichrome wire.

# B Example Power Ramp Configurations

The following figures show example PC configurations.

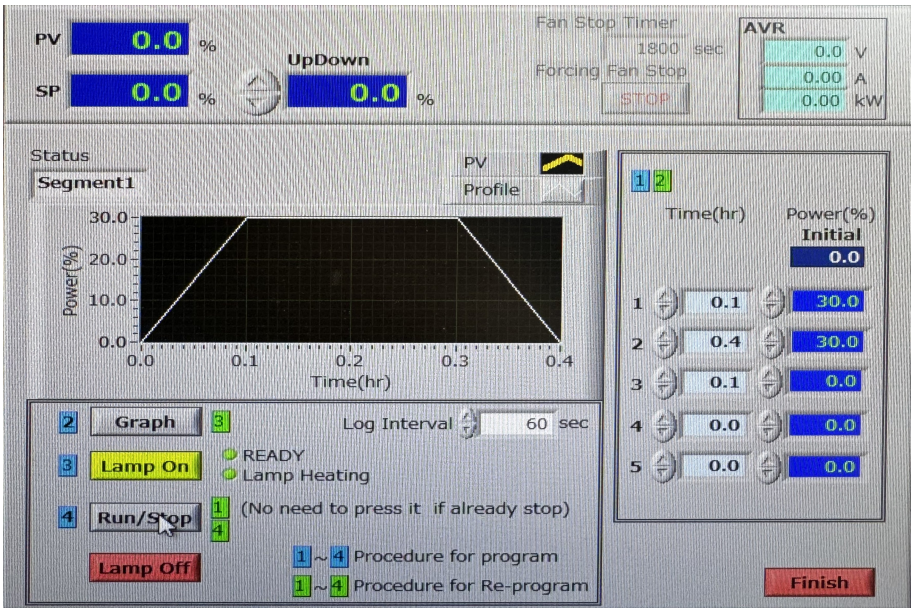


Figure 8: Example PC configuration for determining correct lamp power for a sample.

# References

[1] Crystal Systems Corporation. Application of the optical floating zone apparatus for researching material sciences, n.d.

[2] Crystal Systems Corporation. Fz-t-2000-h space saving type optical floating zone furnace: Product information brochure, n.d.

[3] S. Koohpayeh, D. Fort, and J. Abell. The optical floating zone technique: A review of experimental procedures with special reference to oxides, 09 2008.

[4] Daisuke Nakamura, Takeshi Goto, Nobuyuki Shibata, and Masahiro Hirano. The optical floating zone technique: A review of experimental procedures with special reference to oxides, 2006.

[5] Ushio Inc. Lamp safety and technical resources, n.d.

[6] Zernike Institute for Advanced Materials. Optical foating zone system, 2024. Last modified: 15 May 2024.