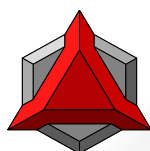


HKZ

High-Pressure High-Temperature Optical Floating Zone Furnace



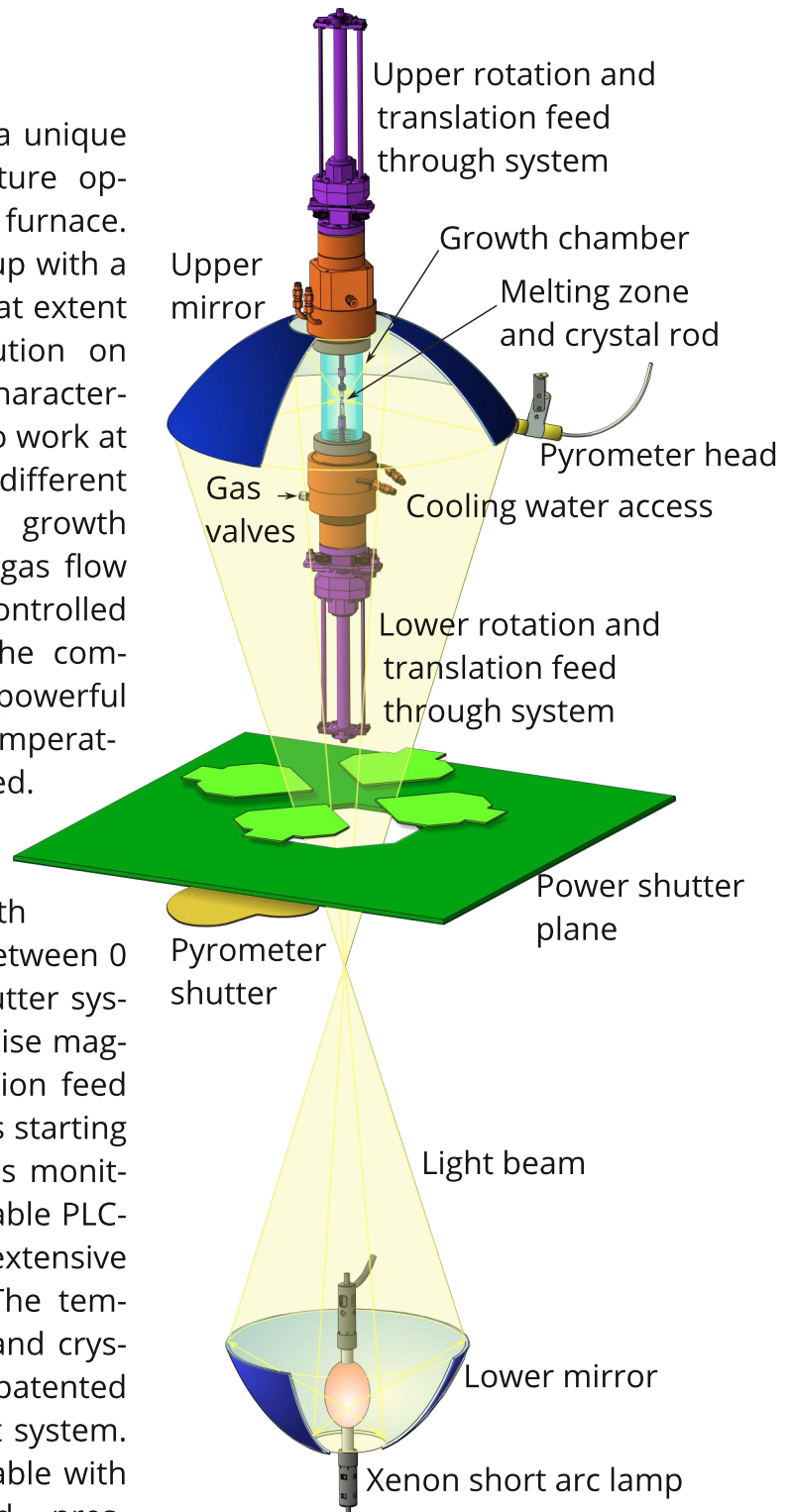
Advanced vertical 2-mirror image crystal growth furnace with unique features and components



SCIDRE
SCIENTIFIC INSTRUMENTS DRESDEN GMBH

HKZ — Hochdruck-Kristallzuchtungs-Anlage

The abbreviation HKZ stands for a unique high-pressure and high-temperature optical floating zone crystal growth furnace. It features a vertical 2-mirror set-up with a highly homogeneous and to a great extent controllable light power distribution on the sample rod. One of the key characteristics of the furnace is the ability to work at pressures of up to 300 bar with different pure and mixed gases in the growth chamber. Additionally, individual gas flow rates can be adjusted and controlled freely and independently over the complete pressure range. Using one powerful xenon short arc lamp, melting temperatures over 3000 °C can be achieved. While the light power tuning range of arc lamps is limited, the thermal energy in the growth chamber is step-less adjustable between 0 and 100 % thanks to a power shutter system in the light beam. Highly precise magnetically coupled linear and rotation feed through systems with pulling rates starting from 0.1 mm/h, advanced process monitoring technologies and a comfortable PLC-based user interface guarantee extensive control of the growth process. The temperature of feed rod, melt zone and crystal is measured directly via a patented in-situ temperature measurement system. An optional after-heater is applicable with all possible atmospheres and pressures—also with high-pressure oxygen atmospheres. This worldwide unique set-up allows the user to rule the growth of materials, which are difficult or impossible to handle at low pressures due to the higher volatility or higher partial pressure of their



elements. A very important characteristic is the highly developable and easily expandable, elaborated modular design of the HKZ system, which allows add-ons and upgrades in an easy and cost-efficient way.

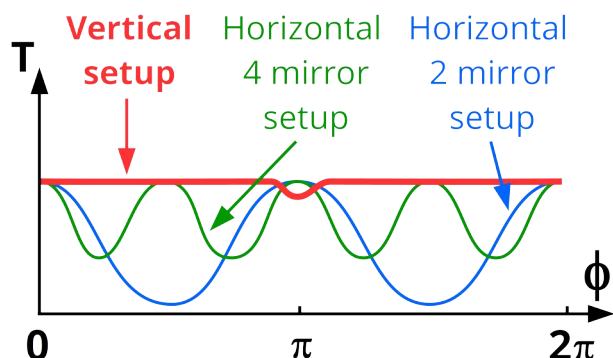
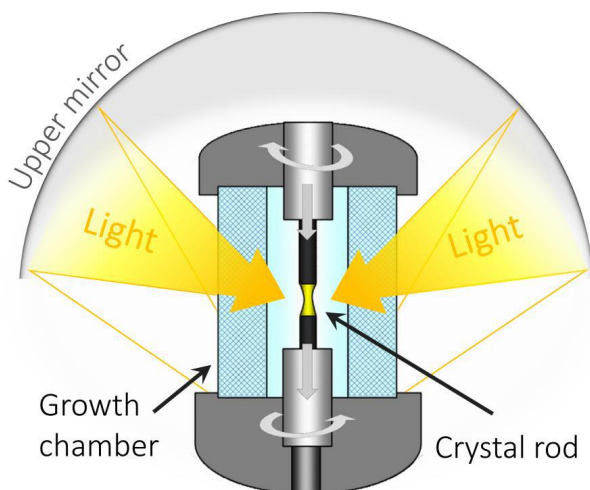
Optical Floating Zone Furnace model HKZ for floating zone melting operating temperature up to 3000°C

New HKZ furnace generation with modular set-up concept: Highly customizable and easily upgradeable in any direction by adding or exchanging a few module components.



Highly homogeneous radial temperature distribution

The main feature compared to other optical floating zone devices is the 2-mirror set-up with (nearly) vertical light propagation. The axial symmetry of this illumination regime enables a homogeneous and highly controllable light distribution on the crystal rod in axial and radial direction and thus provides extremely uniform azimuthal heating and solidification behavior. In order to optimize the floating zone process, in particular the irradiation profile, the optical parameters were extensively analyzed and recalculated. Many details on the influence of light source geometry and position on the irradiation profile of the HKZ can be found in publications by D. Souptel et al. (2007) and M. Palme et al. (2004).



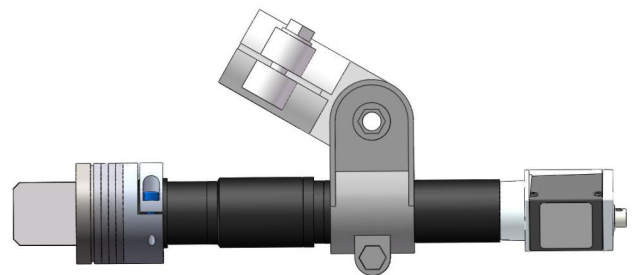
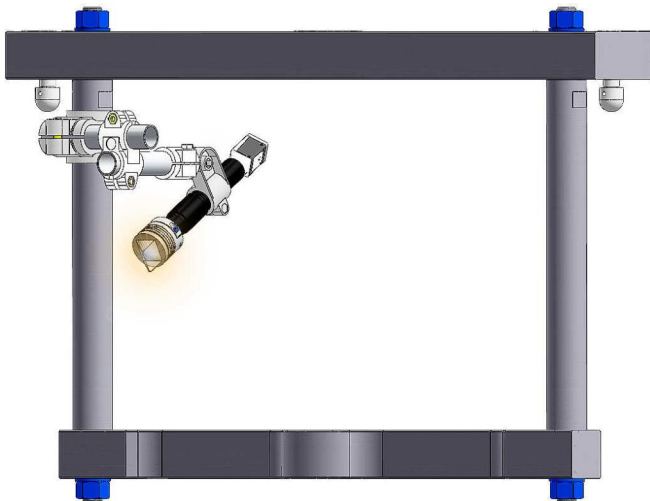
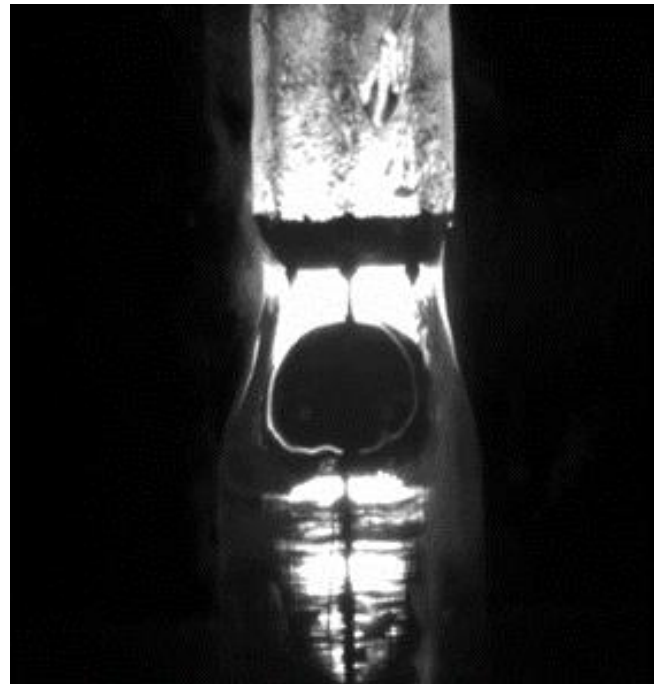
Control Unit – System control with 2 x 27" touch screen process control display

The entire set of experimental parameters such as linear and rotational movements of the pulling drives, gas mixture, mass flow and gas pressure, lamp position and irradiation power are controlled by a programmable logic controller, PLC. A comfortable software application displayed and operated via two 27" touch screens combines all relevant system information and process adjustments in one graphical user interface unit.



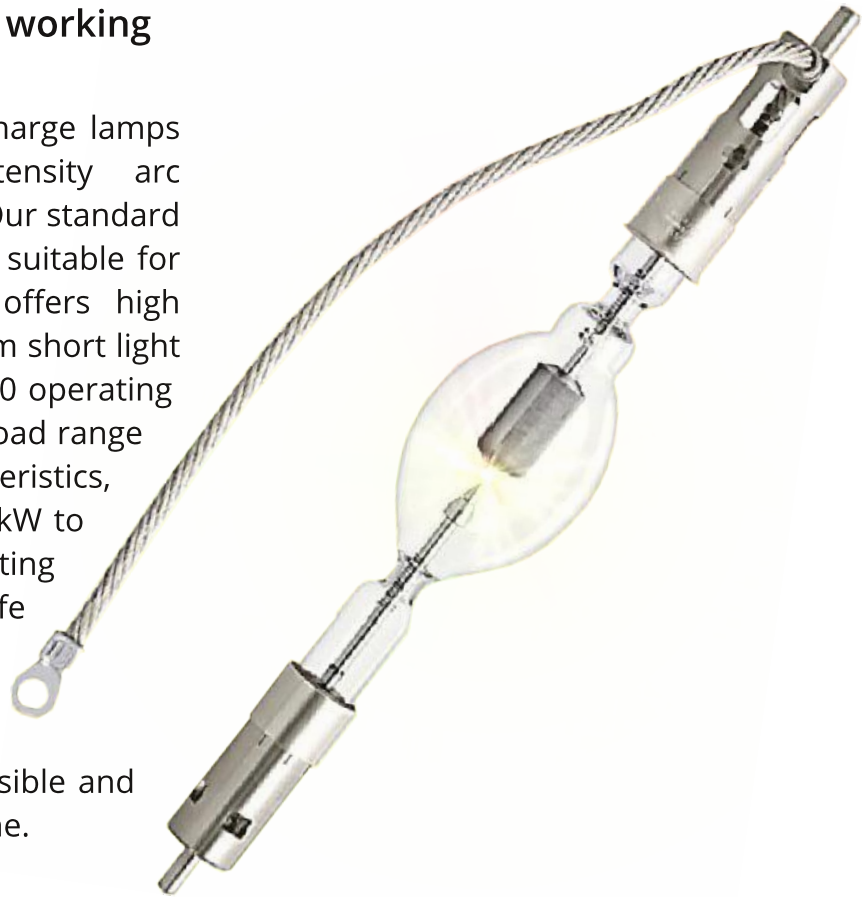
Process monitoring CCD camera; incl. PLC integration

To monitor the growth process the main facility is the CCD camera with specialised high quality optical lenses and filters. Together with the monitoring application, the HKZ user can comfortably perform observations, video recordings, snapshots or length measurements during the growth process. Through the window in the front door the growth can be also directly observed.



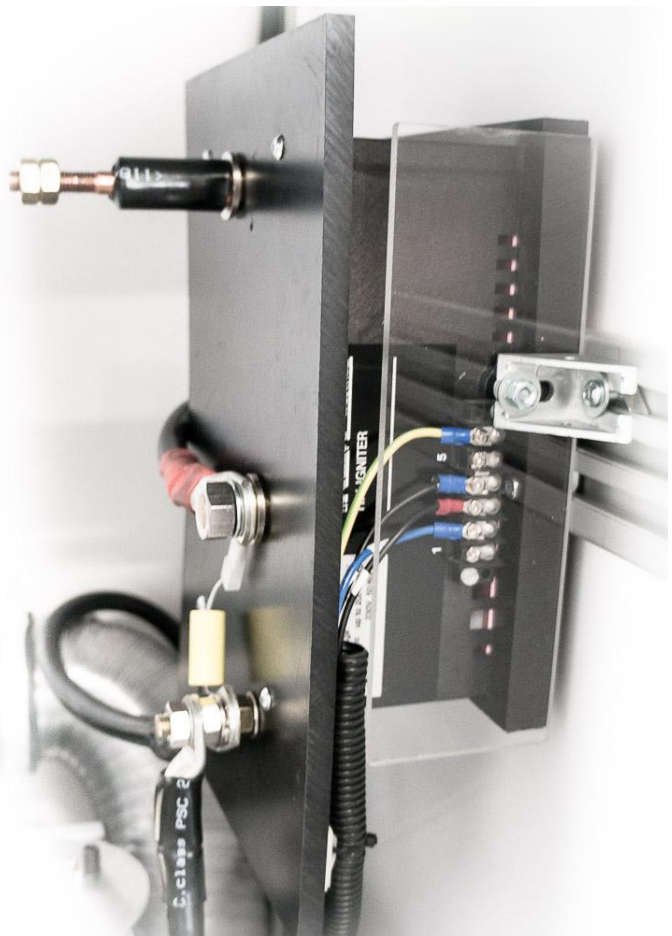
XBO lamp 5000 W, 1200 h working time

XBO lamps are short-arc discharge lamps based on steady high-intensity arc discharge in pure xenon gas. Our standard 5 kW xenon short arc lamp is suitable for most growing purposes. It offers high energy density due to a 7.5 mm short light arc and a long life time of 1200 operating hours. However, we offer a broad range of lamps with different characteristics, such as low or high power (2 kW to 10 kW), ultra short arcs (starting from 4 mm) and very long life time (up to 2200 h). All our lamps feature exceptionally high arc stability and continuous spectrum in the visible and IR range over the whole life time.



Lamp power supply, lamp power control; incl. PLC integration

High quality igniters and electromagnetic - electronic power supplies are accurately selected to ensure stable operation and long life to short arc lamps with different power characteristics, distinguished by an electronic control which manages the output current stability and allows to set and control the most important lamp parameters.



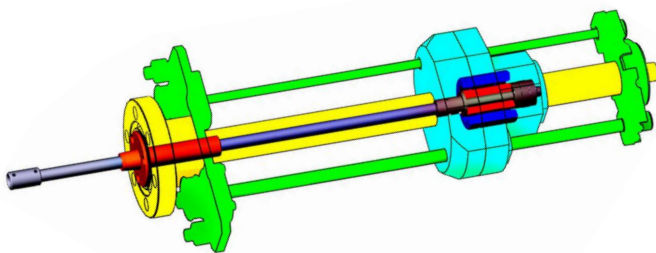
Mirror-set (upside and downside mirror)

The mirror geometry of the HKZ furnace was optimized especially for our purposes. In close cooperation with Fraunhofer Institute for Applied Optics and Precision Engineering IOF in Jena (Germany), the final mirror geometries were developed ready for manufacturing and application. In the HKZ furnace, the mirror positions relative to each other and the position of the upper mirror relative to the molten zone can be adjusted easily.



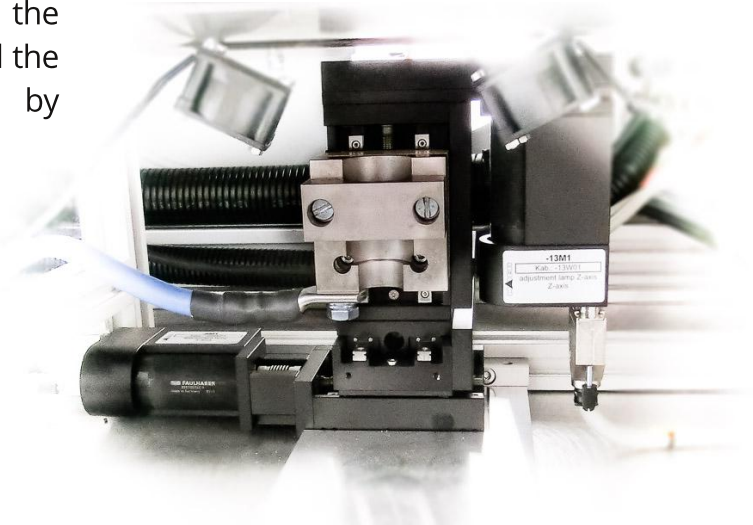
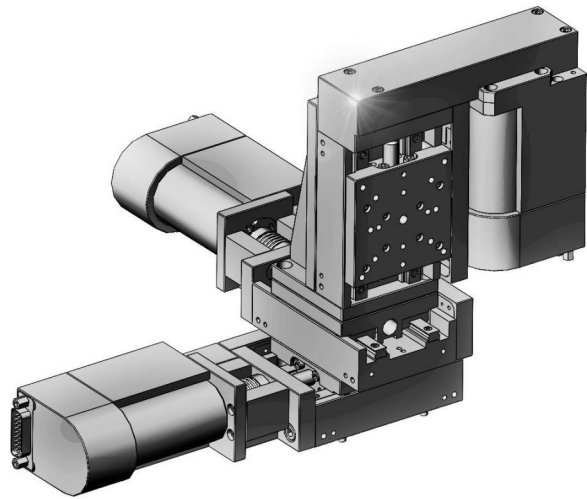
Lower and upper pulling drive with fast gear; incl. PLC integration, position sensor, sample holder

In order to move the upper and lower part of the sample rod, we use two magnetically coupled feed through systems. They allow highly precise synchronous or independent rotations and pulling with rates starting from 0.1 mm/h. A fast gear is implemented for rod set-up. The whole movement system is completely capsuled and is employable at all possible pressure regimes without pressurized bearings.



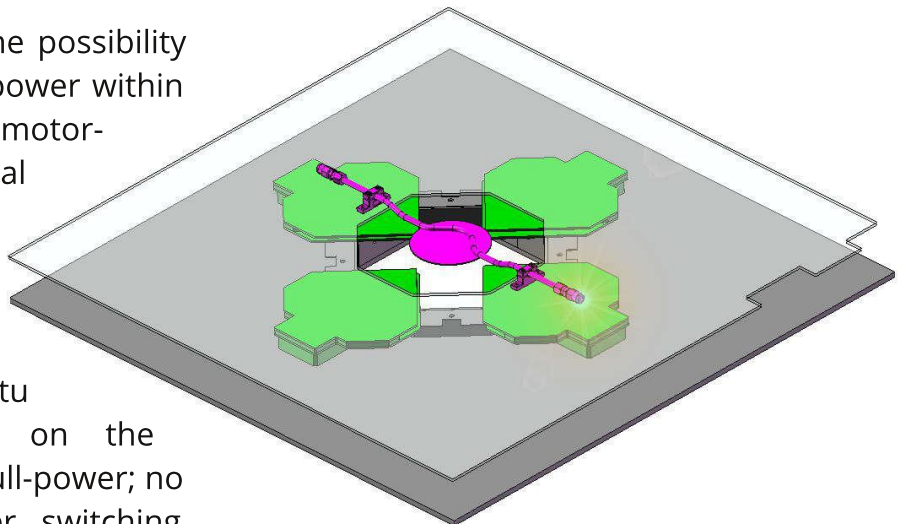
Lamp moving system / focusing system, motorized x-, y-, z-axis with precision linear drives; incl. PLC integration

As the lamp position is crucial for the irradiation distribution and the temperature profile on the sample, the lamp can be comfortably moved in all directions at any time of the experiment. This can be necessary if you want to change the melting zone dimensions and/or perform focus fine tuning before or during the growth. Thus, the floating zone can be custom-tailored and the growth process individually regulated by tuning the lamp position.



Light power shutter system to control the light energy; incl. PLC integration

A very important feature is the possibility to fully control the radiation power within the growth chamber by a motor-driven step-less mechanical shutter, which can rapidly open and close the light beam channel. This allows to perform automatically or manually any kind of in-situ irradiation power changes on the sample surface from zero to full-power; no thermal shocks appear after switching on/off the lamp.



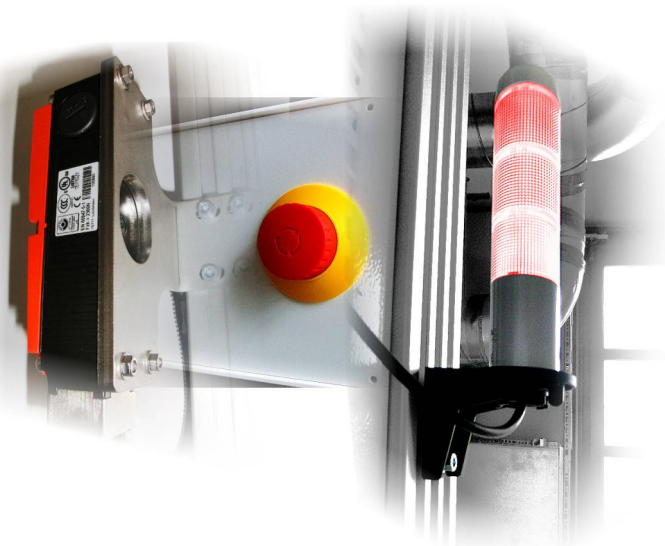
Process window in the upper door for direct view of the growth process

Window with special protective glass for direct view inside the process chamber during the growth process. Direct observation of the the furnace components and the process at any stage through the window in the front door.



Safety system with protection housing, door lock system, automatic shut-down function

The sophisticated protection and safety system manages the relevant interlocks according to the hazard potentials of high pressure and high irradiation. Passive safety is ensured by covering the optical system as well as the growth chamber against unexpected burst of pressurized items. Various precautionary measures are implemented in the controlling software in order to avoid faulty operation to a great extend.



Protective clothing for lamp changing

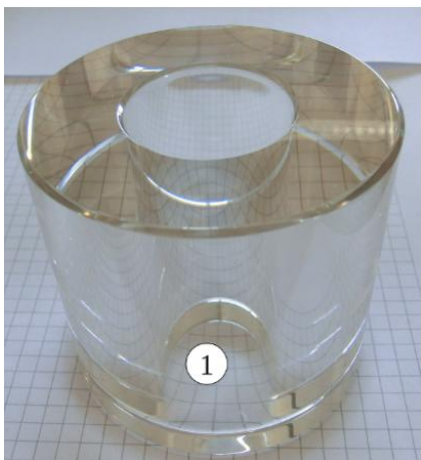
Special leather protective clothes and a face shield are provided as personal safety equipment for the lamp changing process and any work in the lamp room with an installed lamp. The clothing will effectively protect the operator in the unlikely event of a lamp burst.



Pressure application with pressure control up to 10, 50, 150 or 300 bar, incl. process chamber; chamber supporting system; basic item to use gases with the intended pressure

Pressure proved and vacuum stable transparent crystal growth chamber

The "heart" of the device is for sure the process chamber, which can reliably keep pressures between 10^{-5} mbar and 300 bar. The main part of the chamber is a pressure proved and vacuum stable transparent cylinder. For protection against contamination we provide simple inner protective quartz tubes. Due to the furnace geometry, there is an easy access to the crystal growth chamber, even during the growth process, and sufficient space for mounting of auxiliary functional components in the neighbourhood of the growth chamber (e.g. after-heater for growing crystal, pyrometer, camera, etc.) without any appreciable radiation absorption.



Sapphire cylinder (single crystal) for pressures up to 150 bar



Quartz cylinder (thick-walled) for pressures up to 50 bar



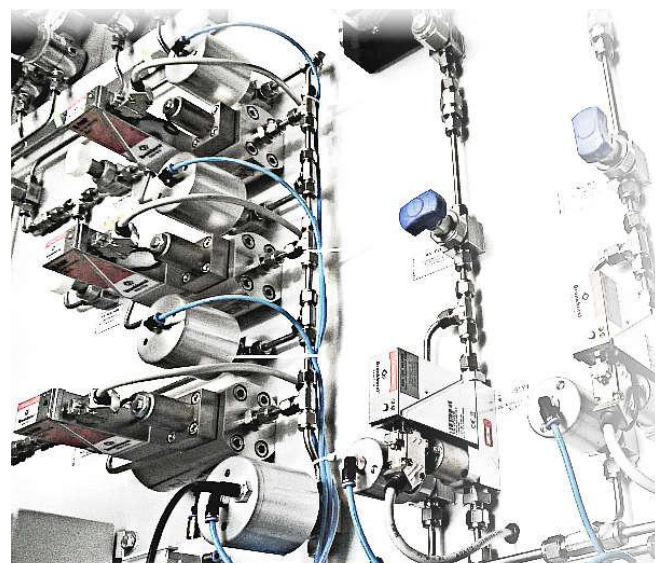
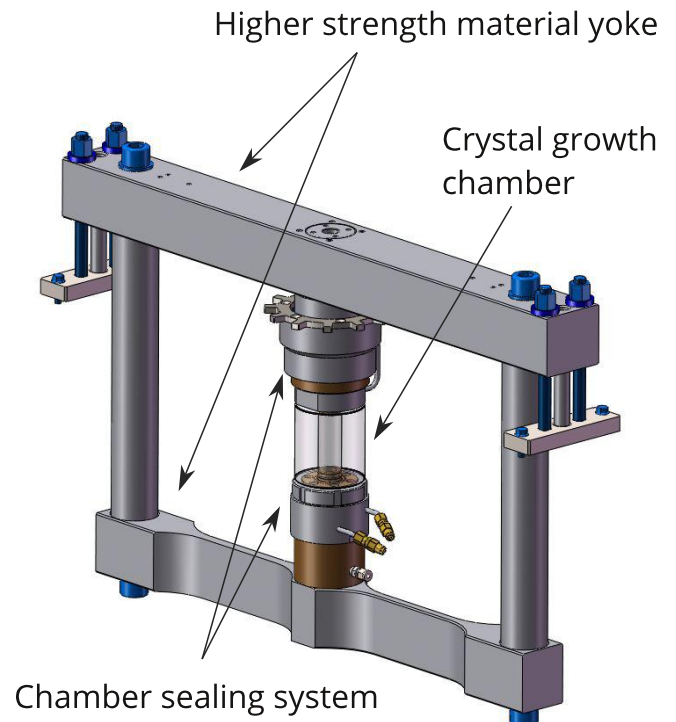
Quartz cylinder (thin-walled) for pressures up to 10 bar

Chamber supporting system

The chamber supporting system has two important components. One of them is the yoke of higher strength material around the process chamber and the upper mirror. It is designed to absorb and withstand the substantial axial forces, which arise from the high pressure atmosphere inside the process chamber. The other component are the upper and lower chamber sealing systems. They provide the pressure-proof and gas-tight connections between the sapphire or quartz cylinder and the yoke with feed through systems. Nevertheless, the sealing components are easy to mount and unmount to guarantee an easy access to the growth chamber and fast sample installation and removal.

Full pressure and flow control for different gases

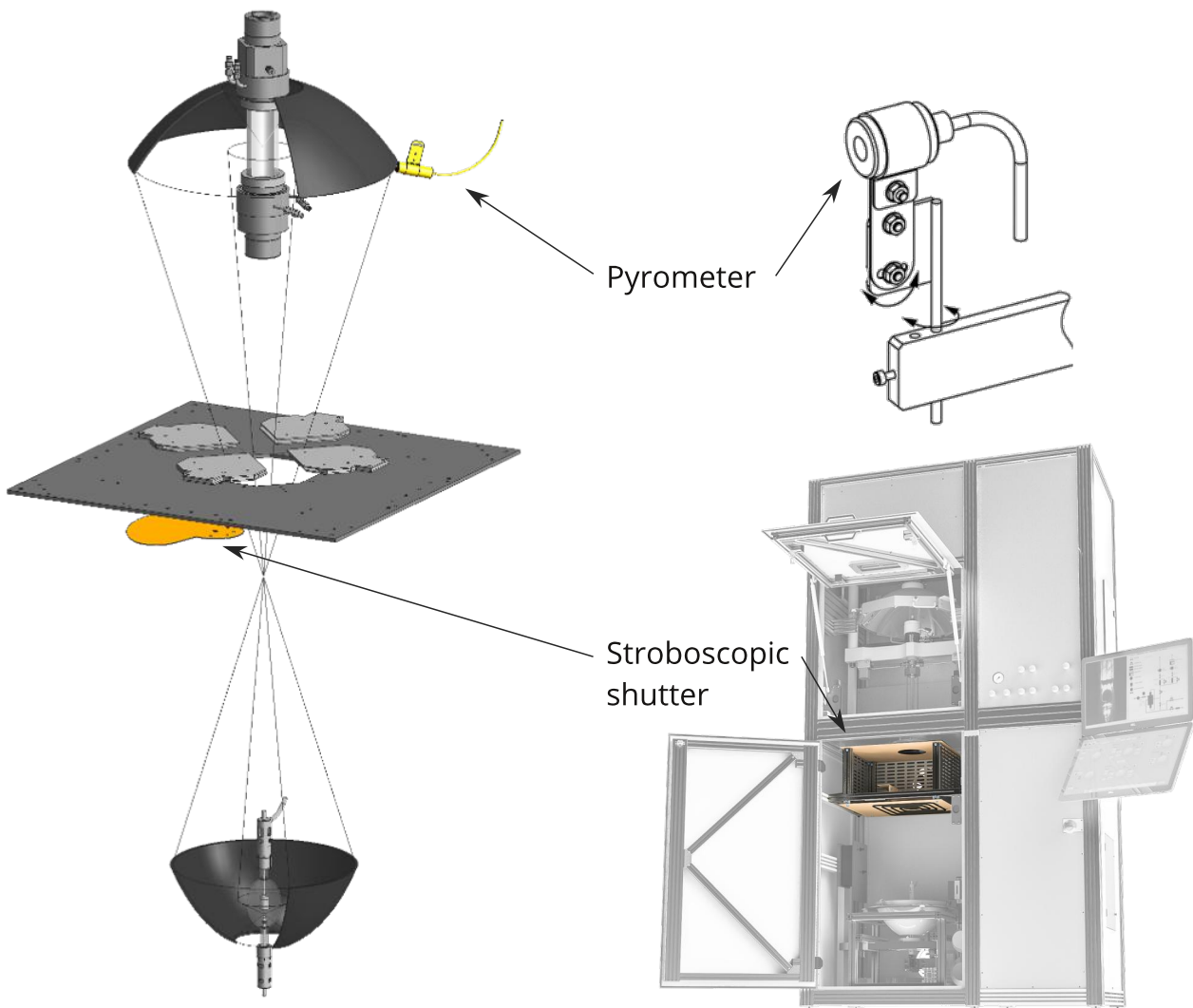
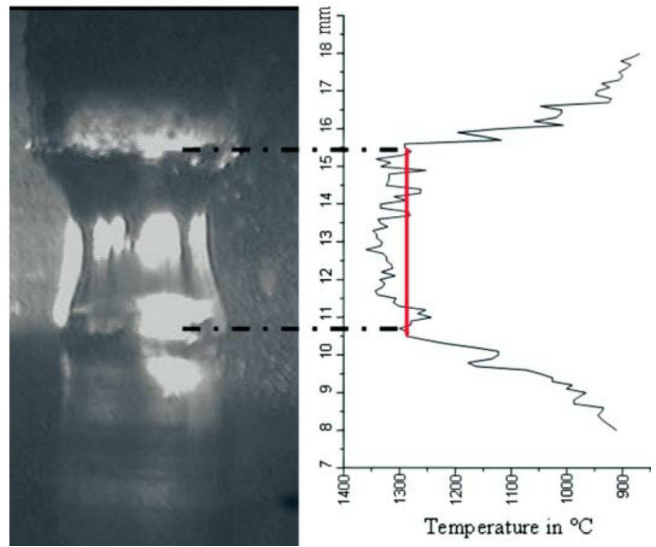
The gas management system enables in-situ adjustable pressures, flow rates and process atmospheres. Typically used gases are pure O_2 , Ar or variable mixtures of both, but almost any other gas could be used, also other reactive gases which interfere with the melt. As there are independent mass flow control systems for each gas, the individual mass flow rates can be adjusted manually or automatically at any time of the growth process.



Patented in-situ temperature measurement system with bi-colour pyrometer; incl. PLC integration

Pyrometric temperature measurement system

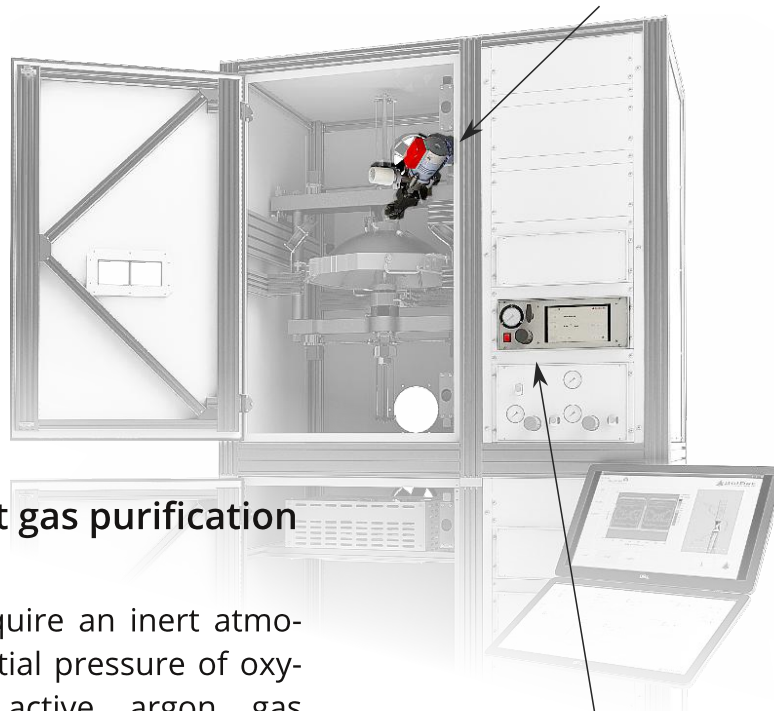
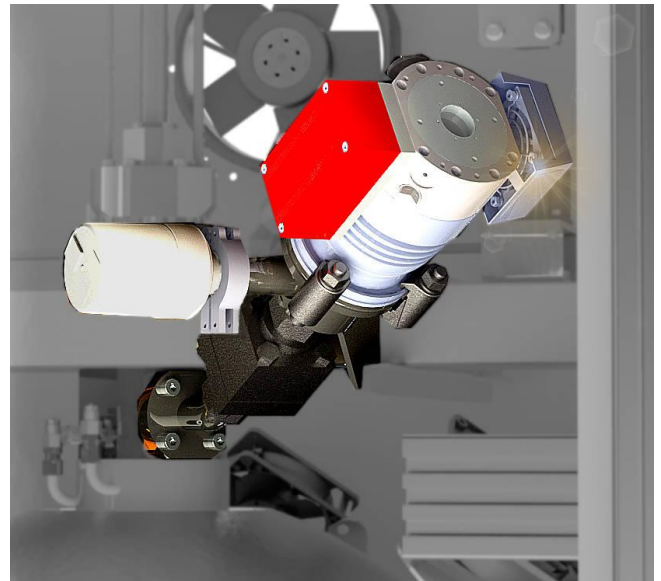
A patented technique permits temperature measurements on the rod and melt surface during the growth process. The light flux is interrupted for some ms, and a two-color pyrometer measures the unbiased radiation. Due to a moveable pyrometer head, temperature profiles can be recorded vertically (sample rod-melt-crystal) and horizontally (along the molten zone). The figure on the right shows a typical vertical temperature profile parallel to the rod axis.



Vacuum and gas cleaning application

Advanced vacuum connections for high pressure system with special valve and wide diameter pipe up to 10^{-5} mbar

The HKZ gas and vacuum measurement sensors and the turbo pump are located close to the process chamber and connected with wide diameter tubes. Thus, the vacuum which is achieved and measured directly represents the conditions within the growth chamber. The pump and sensor controls as well as all other units are comfortably integrated into the main controlling system.



High pressure inert gas purification system

A lot of processes require an inert atmosphere with a low partial pressure of oxygen. The optional active argon gas purification system GRS removes reliably oxygen particles from flowing argon and achieves O_2 concentrations down to 10^{-12} ppm under high pressure conditions. The clear arrangement and visibility of the controls provide an easy handling. The GRS can be customized in a wide range.



Software application and additional features

Data logging system

Includes logging and transfer of all process data to ext. USB

Video application system

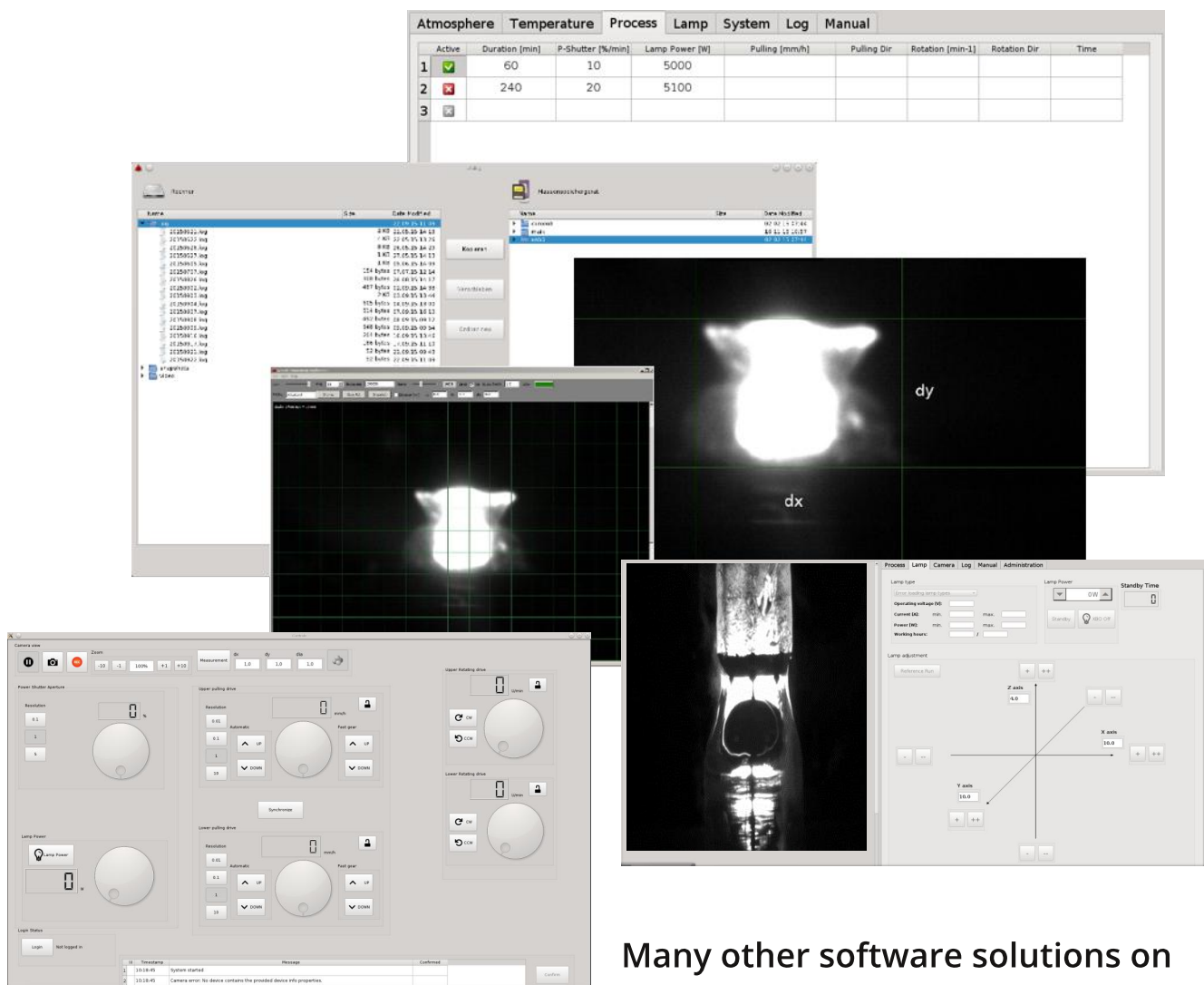
Video recording, zoom, grid, snapshot, measuring of the sample during the growth process

Power ramp function for power-shutter and lamp power control

Create time based power sequences

Travelling ramp function for pulling drive control

Rotation and translation speed of both pulling drives to create time based travelling sequences



Many other software solutions on request

Please ask—we can easily integrate your software handling and process monitoring desires!

HKZ technical specifications overview

Atmosphere

- Argon and oxygen (pure and in any mixture)
- Many other gases also possible
- Gas pressure inside the growth chamber: up to 300 bar
- PLC controlled gas flow up to 1 l/min (higher flow rates possible on request)
- Each gas adjustable individually and independently
- Vacuum: down to $1 \cdot 10^{-5}$ mbar
- Turbo pump close to the process chamber, wide diameter connections
- Active argon gas cleaning (removes O_2 traces in argon down to 10^{-12} ppm with titanium gettering, applicable under high pressure)
- Oxygen content measurement system

Growth chamber

- Highly transparent, pressure-proved between 0 and 300 bar
- Long life span due to protective tube
- Sample holder with different diameters

Optical heating

- Xenon short arc lamp, available between 2 kW and 10 kW
- Temperature: up to 3000 °C
- Power shutter in the light beam, motor-driven step-less adjustable between 0 and 100 % — full temperature control and avoidance of thermal shocks
- Upper and lower elliptical mirrors, specifically optimized geometry
- Mirror position adjustment system
- Motor-driven in-situ lamp positioning and focusing unit, workable during the experiment
- Lamp power control

Sample rod moving

- Precise magnetically coupled linear and rotation feed through systems
- Pulling rate: 0.1 mm/h to 200 mm/h
- Fast service gear

- Pulling length: up to 200 mm
- Rotation rate: 0 to 70 rpm

Temperature measurement

- Precise two-color pyrometer with a patented in-situ stroboscopic method
- Adjustable in x- and z-position, PLC-controlled
- Temperature profile recordings (vertically: rod-melt-crystal and horizontally along the molten zone)

After-heater

- Applicable with all possible atmospheres and pressures
- Easily exchangeable heater coils
- Modular, highly customizable unit
- Highly adaptable to special needs

Process control

- High resolution CCD camera with specialized lenses and filters
- Monitoring software application: visual control, video recordings, snapshots and length measurements during the growth process
- Several advanced power ramp and travelling ramp functions adaptable
- Front window for direct observation of the growth process
- Comfortable GUI with two 27" touch screens for all PLC-based adjustments
- Safety system with protection housing, door lock system, automatic shut down function

Required lab connections

- Gas supply with the intended pressure
- Exhaust air system
- Energy supply: 3-phase AC, 50 Hz, 400 V, 63 A
- Cooling water

Furnace dimensions

- Height: 3020 mm , width: 1630 mm, depth: 945 mm; slightly smaller



References

Materials grown with the HKZ furnace

- Zhang, J., Chen, Y. S., Phelan, D., Zheng, H., Norman, M. R., & Mitchell, J. F. (2016). Stacked Charge Stripes in Quasi-Two Dimensional Trilayer Nickelate $\text{La}_4\text{Ni}_3\text{O}_8$. arXiv preprint:1601.03711.
- Li, Z. W., Liu, C. F., Skoulatos, M., Tjeng, L. H., & Komarek, A. C. (2015). Floating zone growth of Ba-substituted ruthenate $\text{Sr}_{2-x}\text{Ba}_x\text{RuO}_4$. *J. Cryst. Growth*, 427, 94-98.
- Cao, H. B., Zhao, Z. Y., Lee, M., Choi, E. S., McGuire, M. A., Sales, B. C., Zhou, H. D., Yan, J.-Q., and Mandrus, D. G. (2015): High pressure floating zone growth and structural properties of ferrimagnetic quantum paraelectric $\text{BaFe}_{12}\text{O}_{19}$. *APL Mat.* 3, 062512.
- Zhang, J., Zheng, H., Malliakas, C. D., Allred, J. M., Ren, Y., Li, Q. A., Han, T.-H. & Mitchell, J. F. (2014). Brownmillerite $\text{Ca}_2\text{Co}_2\text{O}_5$: Synthesis, Stability, and Re-entrant Single Crystal to Single Crystal Structural Transitions. *Chem. Mater.*, 26(24), 7172-7182.
- Bauer, A., Regnat, A., Blum, C. G., Gottlieb-Schönmeier, S., Pedersen, B., Meven, M., Wurmehl, S., Kuneš, J. & Pfeleiderer, C. (2014). Low-temperature properties of single-crystal CrB_2 . *Phys. Rev. B*, 90(6), 064414.
- Omar, A., Blum, C. G., Löser, W., Büchner, B., & Wurmehl, S. (2014). Effect of annealing on spinodally decomposed Co_2CrAl grown via floating zone technique. *J. Cryst. Growth*, 401, 617-621.
- Brasse, M., Chioncel, L., Kuneš, J., Bauer, A., Regnat, A., Blum, C. G. F., Wurmehl, S., Pfeleiderer, C., Wilde, M. A. & Grundler, D. (2013). de Haas-van Alphen effect and Fermi surface properties of single-crystal CrB_2 . *Phys. Rev. B*, 88(15), 155138.
- Omar, A., Dimitrakopoulou, M., Blum, C. G. F., Wendrock, H., Rodan, S., Hampel, S., Löser, W., Büchner, B. & Wurmehl, S. (2013). Phase Dynamics and Growth of $\text{Co}_2\text{Cr}_{1-x}\text{Fe}_x\text{Al}$ Heusler Compounds: A Key to Understand Their Anomalous Physical Properties. *Cryst. Growth Des.*, 13(9), 3925-3934.
- Blum, C. G., Ouardi, S., Fecher, G. H., Balke, B., Kozina, X., Stryganyuk, G., Ueda, S., Kobayashi, K., Felser, C., Wurmehl, S. & Büchner, B. (2011). Exploring the details of the martensite-austenite phase transition of the shape memory Heusler compound Mn_2NiGa by hard x-ray photoelectron spectroscopy, magnetic and transport measurements. *Appl. Phys. Lett.*, 98(25), 252501.
- Wizent, N., Behr, G., Löser, W., Büchner, B., & Klingeler, R. (2011). Challenges in the crystal growth of Li_2CuO_2 and LiMnPO_4 . *J. Cryst. Growth*, 318(1), 995.
- Behr, G., Löser, W., Wizent, N., Ribeiro, P., Apostu, M. O., & Souptel, D. (2010). Influence of heat distribution and zone shape in the floating zone growth of selected oxide compounds. *J. Mater. Sci.*, 45(8), 2223-2227.
- Blum, C. G. F., Jenkins, C. A., Barth, J., Felser, C., Wurmehl, S., Friemel, G., Hess, C, Behr, G, Büchner, B, Reller, A, S. Riegg, S., Ebbinghaus, S. G., Ellis, T., Jacobs, P. J., Kohlhepp, J. T. & Swagten, H. J. M. (2009). Highly ordered, half-metallic Co_2FeSi single crystals. *Appl. Phys. Lett.*, 95(16), 161903.
- Wizent, N., Behr, G., Lipps, F., Hellmann, I., Klingeler, R., Kataev, V., Löser, W., Sato, N. & Büchner, B. (2009). Single-crystal growth of LiMnPO_4 by the floating-zone method. *J. Cryst. Growth*, 311(5), 1273-1277.
- Behr, G., Löser, W., Souptel, D., Fuchs, G., Mazilu, I., Cao, C., Köhler, A., Schultz, L. & Büchner, B. (2008). Crystal growth of rare earth-transition metal borocarbides and silicides. *J. Cryst. Growth*, 310(7), 2268-2276.

Methodical considerations concerning the HKZ system

- Wizent, N., Behr, G., Löser, W., Büchner, B. & Klingeler, R. (2011). Challenges in the crystal growth of Li_2CuO_2 and LiMnPO_4 . *J. Cryst. Growth*, 318(1), 995-999.
- Behr, G., Löser, W., Wizent, N., Ribeiro, P., Apostu, M. O., & Souptel, D. (2010). Influence of heat distribution and zone shape in the floating zone growth of selected oxide compounds. *J. Mater. Sci.*, 45(8), 2223-2227.
- Behr, G., Löser, W., Souptel, D., Fuchs, G., Mazilu, I., Cao, C., Köhler, A., Schultz, L. & Büchner, B. (2008). Crystal growth of rare earth-transition metal borocarbides and silicides. *J. Cryst. Growth*, 310(7), 2268-2276.
- Souptel, D., Löser, W. & Behr, G. (2007). Vertical optical floating zone furnace: principles of irradiation profile formation. *Journal of crystal growth*, 300(2), 538-550.
- Behr G, Voigtländer R, Horst A, Morgner R, Fischer F: Patent DE 10 2006 019 807.7 (21 April 2006) and PCT/EP2007/05157 (7 March 2007)
- Palme, M., Riehemann, S., Horst, A., Souptel, D. & Behr, G. (2004). Optical simulation of a radiation heating. *Fraunhofer IOF Annual Report 2004*, 70-71.

This data sheet is elaborated with utmost diligence. We reserve the right of error and technical modifications. All illustrations, descriptions and technical specifications are subject to change without prior notice.

Dresden, 05 July 2016

Quotation request HKZ

**www.scidre.de/quot/hkz
(please use the message field)**

**High-Pressure
High-Temperature
Optical Floating Zone Furnace**

**Scientific Instruments Dresden GmbH
Gutzkowstraße 30
01069 Dresden
Germany**

The Scientific Instruments Dresden, in short SciDre GmbH, is a globally acting research technology company located in Dresden, Germany. We are a team of engineers and scientists specialized in developing and manufacturing of scientific equipment. One of the main activity fields of SciDre is the production of unique tools and systems for material research and crystal growth based on the requests and requirements of scientists. All our devices can be custom-tailored in almost any specification.

Phone

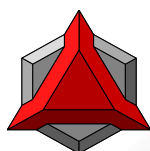
+49 351 821 131 40

Home page

scidre.com

E-mail

info@scidre.de



SCIDRE
SCIENTIFIC INSTRUMENTS DRESDEN GMBH