Bimes pro
HTS high temperature chelate doping system

realizing ideas

in the field of special optical fiber fabrication equipment & processes

Sept 2023

MCVD processes for rare-earth doped fiber preform fabrication:

- most often by solution doping method:
    - invented in mid 1980's in Southampton University
    - complex handling and processing & limited core thickness

- other MCVD based methods:
    - chelate doping (high temperature sublimation of solid precursors), was reported by Tumminelli et al in 1990
    - flash vaporization (impulse evaporation from aerosols made from active precursor solutions), reported by Lenardic et al. in ECOC 2007
    - aerosol precursor delivery for MCVD or OVD deposition was proposed in 1986 by T.F. Morse (modified by Liekki for OVD deposition)

Vapor-phase doping methods

Advantages of vapor phase methods:

- well-controlled & repeatable precursor vapor generation
- stable, repeatable "in-situ" vapor phase layer deposition
- suitable for fabrication of LMA fibers
- allows doping of silica by all dopants used in specialty preform fabrication (Ge, P, B, F, Al, Bi, RE, other metal and transition elements)
- allows photodarkening mitigation by special rare earth co-doping
- allows gadient doping profiles to compensate differences betwen optical and acoustic fields
- allows fabrication of large core preforms, improving profile control
- provides means for fabrication of novel glasses and optical fiber materials
- allows use of pure chemicals (99.999% or better to metals)

Raw materials – Vapor pressure

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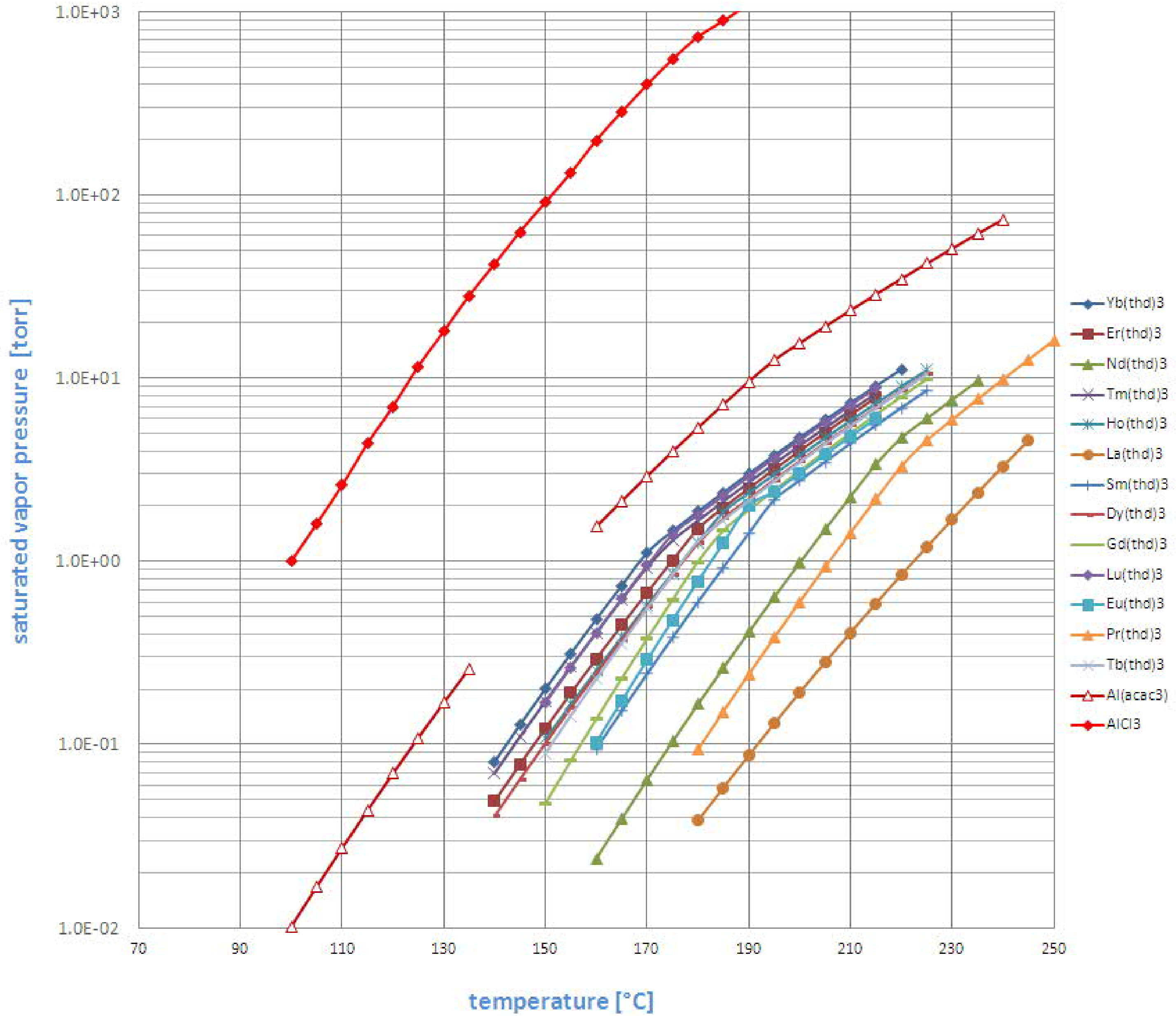
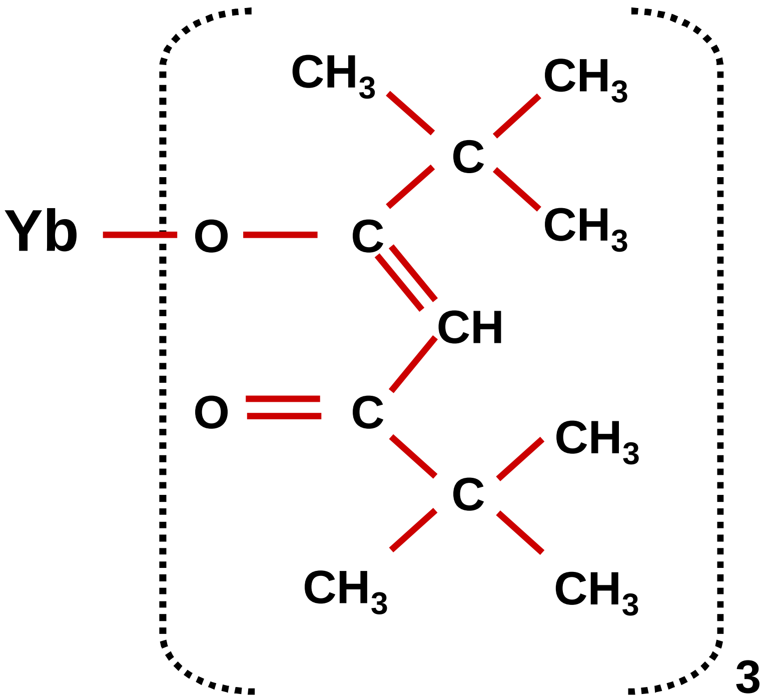
Lanthanide Chelate Vapor Pressure
(JE Sicre et al. JAmChemSoc 91:13 18.6.69)

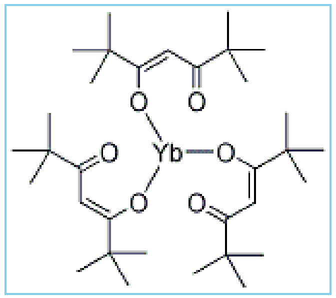


Raw materials - precursors

Chelates:



Tris(2,2,6,6-tethrametyl- 3,5-heptanedionato) ytterbium



$Yb(C_{11}H_{19}O_2)_3$

- Other rare earth chelates:
- Er(thd) 3
  - melting point (°C): 179-182
  - decomposes over 270 °C
- Ce(thd)3 (or Ce(thd)4)
  - melting point (°C): 265
  - decomposes above 280°C sublimates
- Yb(thd)3
  - melting point (°C): 167-169
  - decomposes over 300 °C

- Available also compounds with Eu, Sm, Th, Pr, Ho, Ce and other lanthanide ions, as well as most transition elements and heavy metals

Raw materials - precursors

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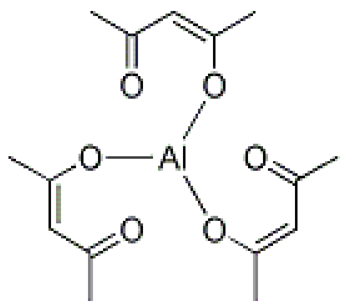


Aluminum tris actyl acetonate

$Al(C_5H_7O_2)_3$

Aluminum acetylacetonate Chemical Properties	
mp	190-193 °C(lit.)
bp	315 °C(lit.)
density	1,27 g/cm^3
Fp	314-316°C
Water Solubility	2.5 g/L (20 ºC)
BRN	4157942
Stability:	Stable. Incompatible with strong oxidizing agents.

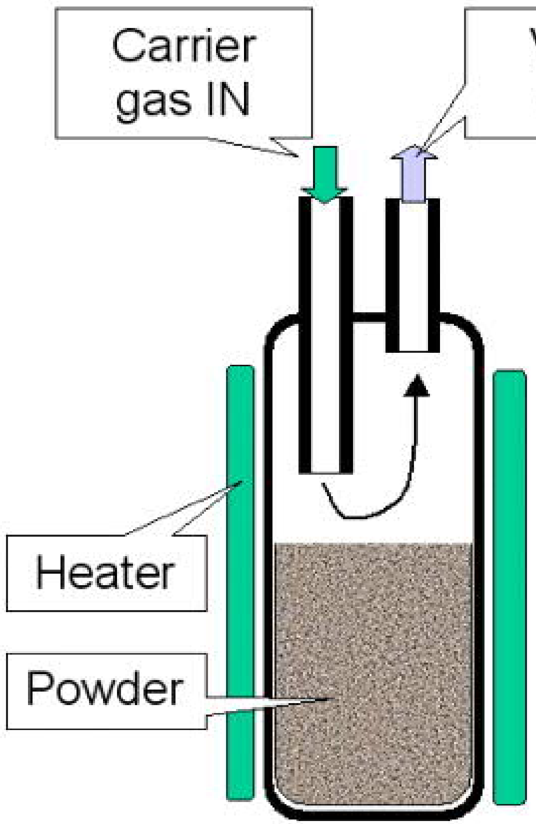
Aluminum trichloride

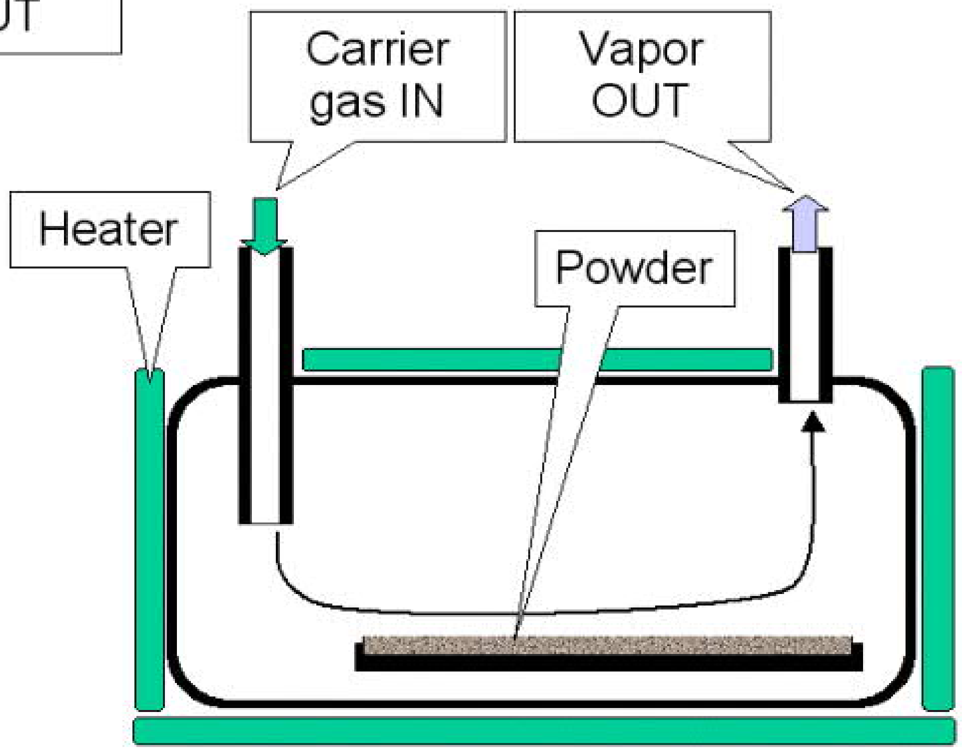
(hydrate or anhydrous)

$AlCl_3$

Aluminium chloride Chemical Properties	
mp	194 °C
bp	180°C
density	2.44
vapor pressure	1 mm Hg (100 °C)
Fp	88 °C
storage temp.	2-8°C
solubility	H_2O: soluble
form	powder
Water Solubility	reacts
Sensitive	Moisture Sensitive
Merck	14,337
Stability:	Stable, but reacts violently with water. Prolonged storage may lead to pressure build-up - vent container periodically. Incompatible with alcohols and a variety of other materials (see complete MSDS sheet for full list).

Fixed bed vs. flat bed sublimator



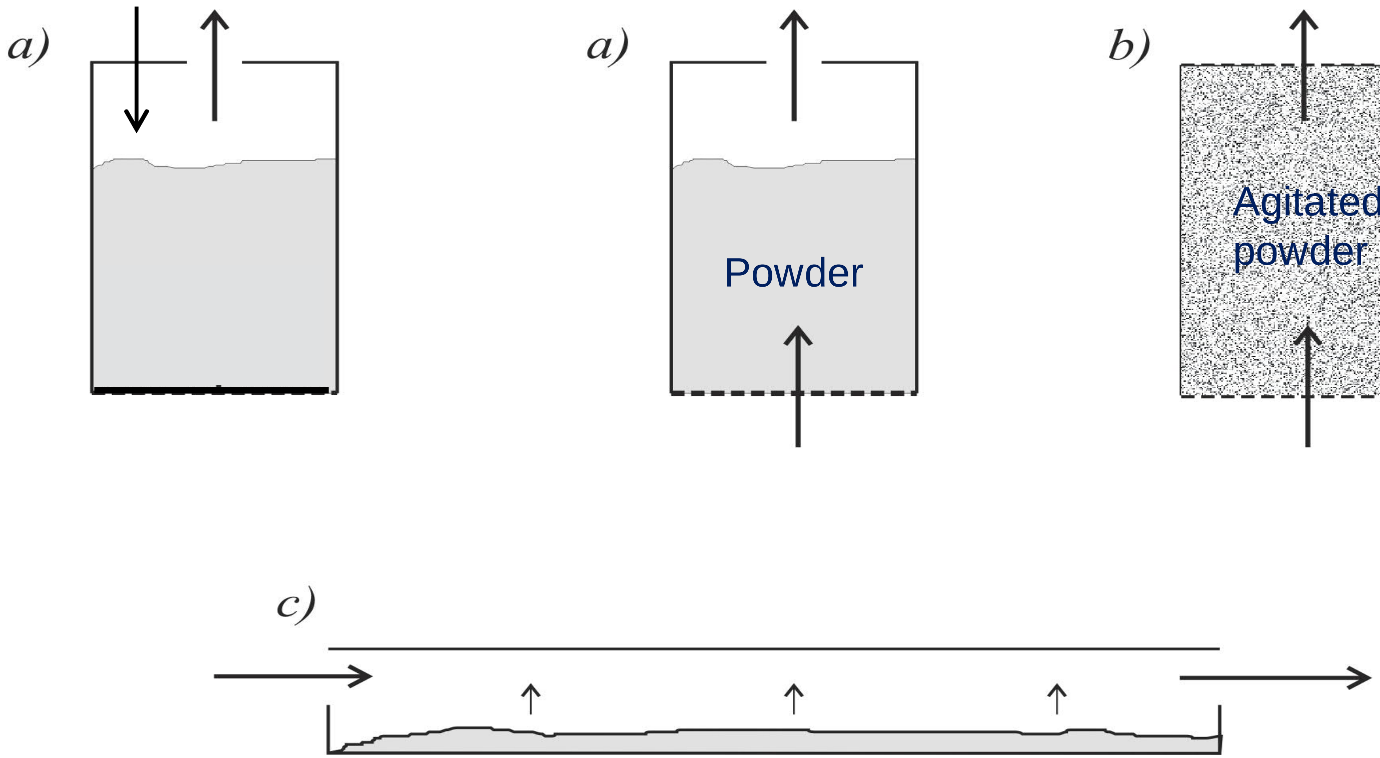


Fixed bed sublimator

Flat bed sublimator

For details pls see OFC2009 paper OThK6 (Lenardic et al)

typesAlYbCeF longitudinal homogeneity



a) Fixed bed evaporator (simple and flow-through)

b) Fluidized bed evaporator

c) „Flat bed“ evaporator

- From fixed bed to flat bed design:
  - fixed bed design was proven inadequate
    - low precursor flow
    - unstable delivery
    - changes to morphology of powder
    - possible agglomeration or lumping if temperature too close to melting point
  - fluidized bed design was proven unpractical (ultrasonic agitation)
    - too large carrier gas flow required than suitable for MCVD process
  - flat bed was developed:
    - permitting evaporation at melting point or above
    - providing sufficient precursor flow at moderate carrier gas flow
    - changes to sublimator design provide large surface and proper carrier gas flow guidance

HTS high temperature doping system design

- HTS Cabinet
  - steel cabinet, powder painted to prevent oxidation while providing clean look
  - isolated compartments: electrical, low temperature section, hot chamber, delivery tube, vapor soot box
  - whole cabinet mounted on precise sliding mechanism, for proper alignment of the delivery tube with the rotary seal and substrate tube
  - connection for gases, power and exhaust conveniently situated
  - HTS model with oil heated delivery tube includes oil circulation thermal bath system, heated main gas panel, leak tight doors and large windows for visual inspection
- HTS low temperature gas panel
  - made of SS316EL components with orbitally welded connections and VCR fittings for lowest leak rate
  - highest grade components are used in the panels, to ensure integrity and long term high temperature operation
  - Bronkhorst MFC, high precision (±0.5% setting), EtherCAT interface, digital, multicalibration
- HTS high temperature section
  - piping made of SS316EL or Ni-alloy components with orbitally welded connections and VCR fittings
  - custom developed low C_v, high temperature valves
  - hot chambers electrically heated up to 250°C
  - 3, 4 or 5 sublimators for precursor evaporation, with high temperature stop valves
  - one sublimator screened from others to be able to operate at lower temperature (for AlCl3)
- Delivery tube
  - delivery tube outer shell is SS316EL or Ni-alloy, electrically- or oil-heated
  - small diameter conduits in bundle are placed inside delivery tube to carry vapor and gases to the tube tip
  - tube tip is designed to correctly distribute and mix all gas and vapor constituents, preventing premature oxidation or decomposition
- HTS rotary seal
  - special design to operate at high temperature and ensure sealing to the delivery tube, with channel for MCVD gases

HTS key design features

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HTS high temperature doping system design

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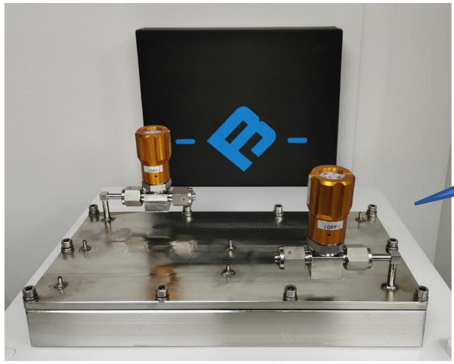
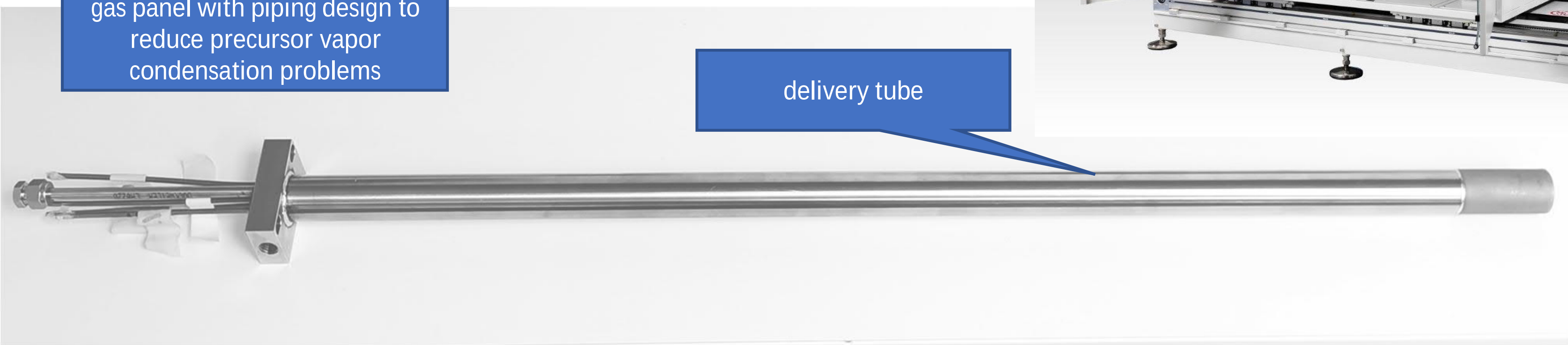


flat bed sublimator for large precursor material charge and large vapor flow

rotary seal for delivery tube and MCVD gases inside lathe headstock

gas panel with piping design to reduce precursor vapor condensation problems



delivery tube

Core deposition process (MCVD) - HTS vapor-phase doping

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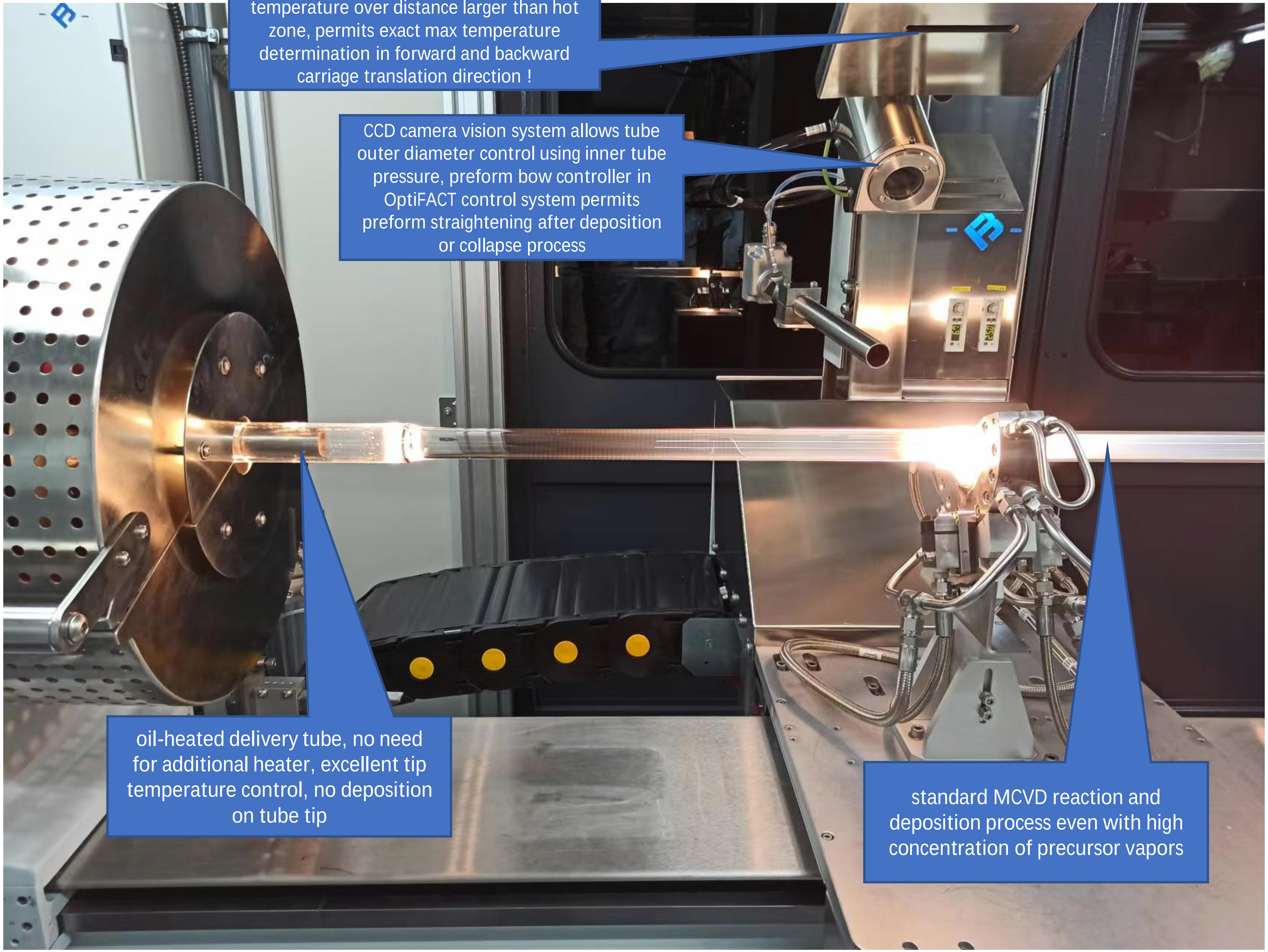

Longitudinal temperature profile of delivery tube

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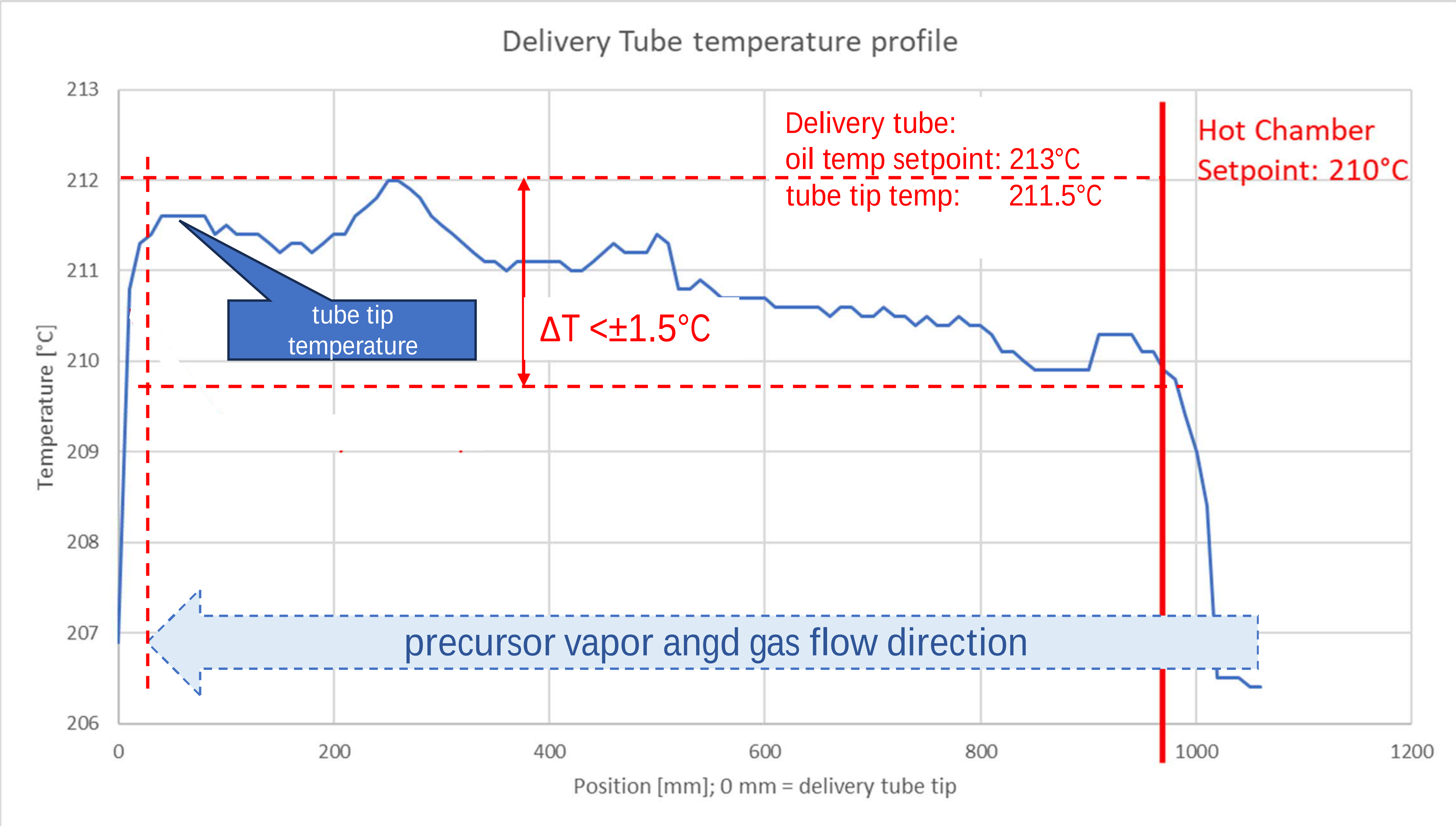
Delivery tube profile is critical for stable and repeatable vapor delivery process. Oil heated delivery tube provides excelent longitudinal temperature profile, with minimal temperature variations from set temperature !



AlYbCeF longitudinal homogeneity

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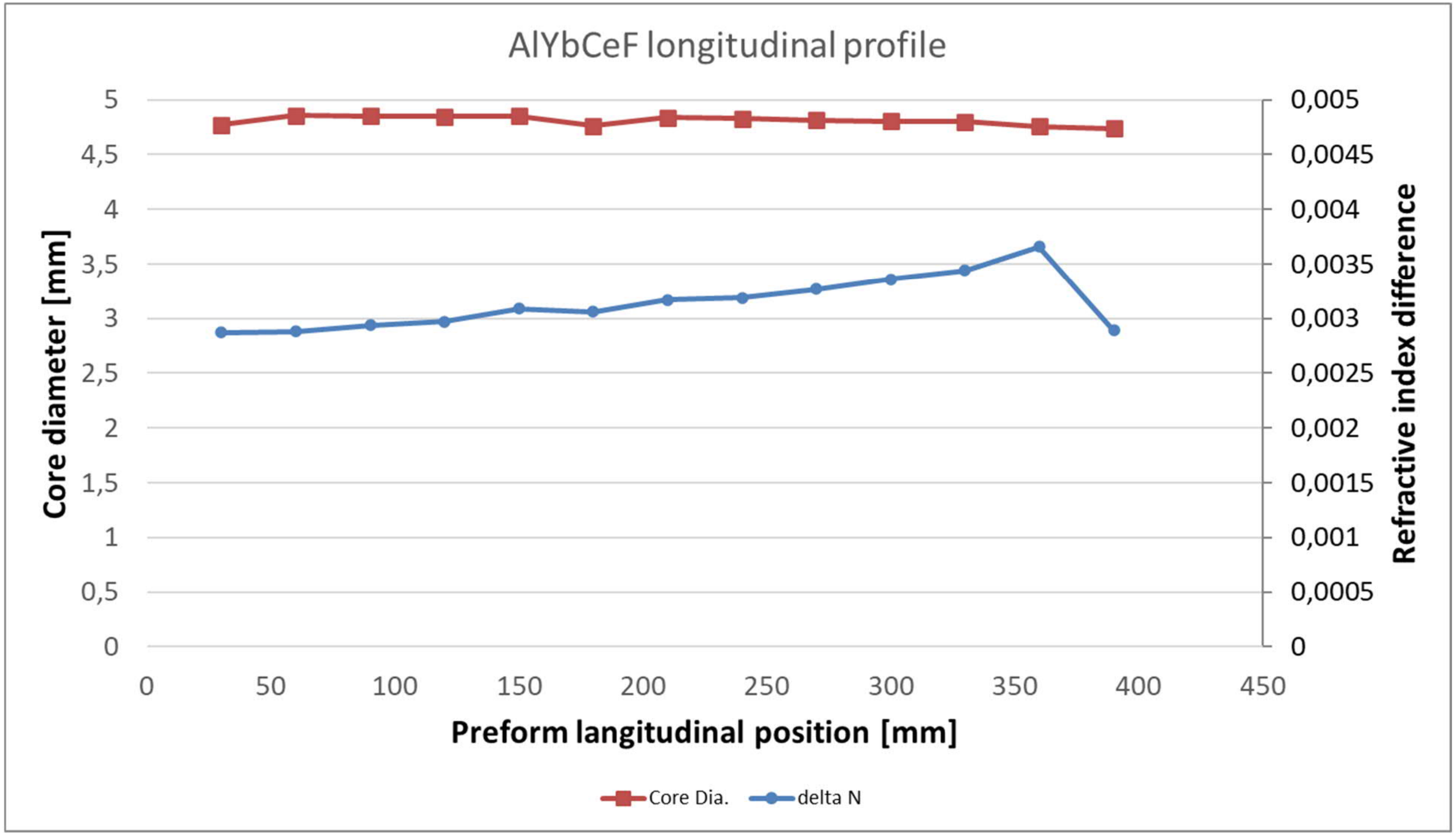
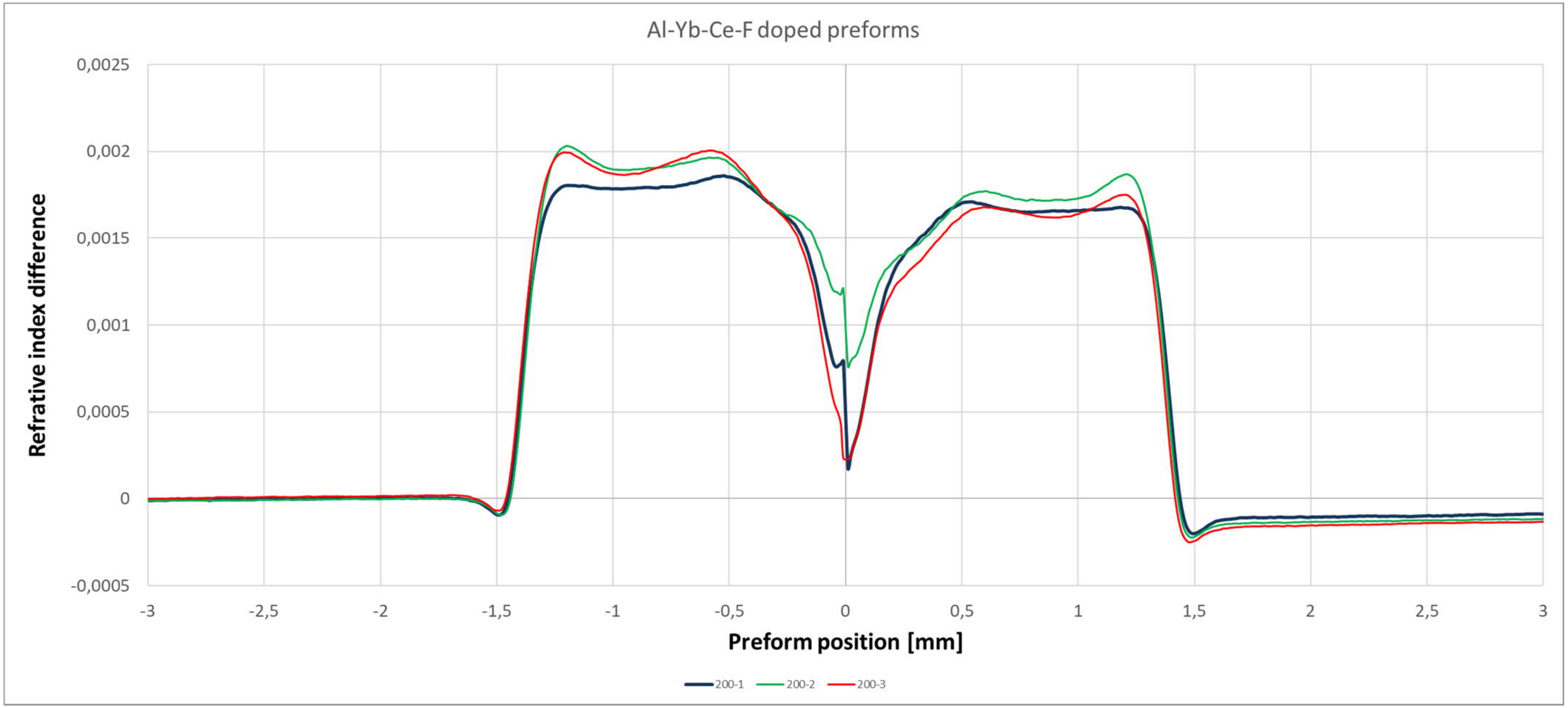
- Below is a longitudinal plot of core diameter and core deltaN for AlYbCeF preform.
- Glass structure: Al_2O_3 3.0mol%; Yb_2O_3 0.30mol%, Ce_2O_3 0.15mol%, C_2F_6 used for down-doping
- Deposited layers: 40 (low efficiency due to use of C_2F_6)



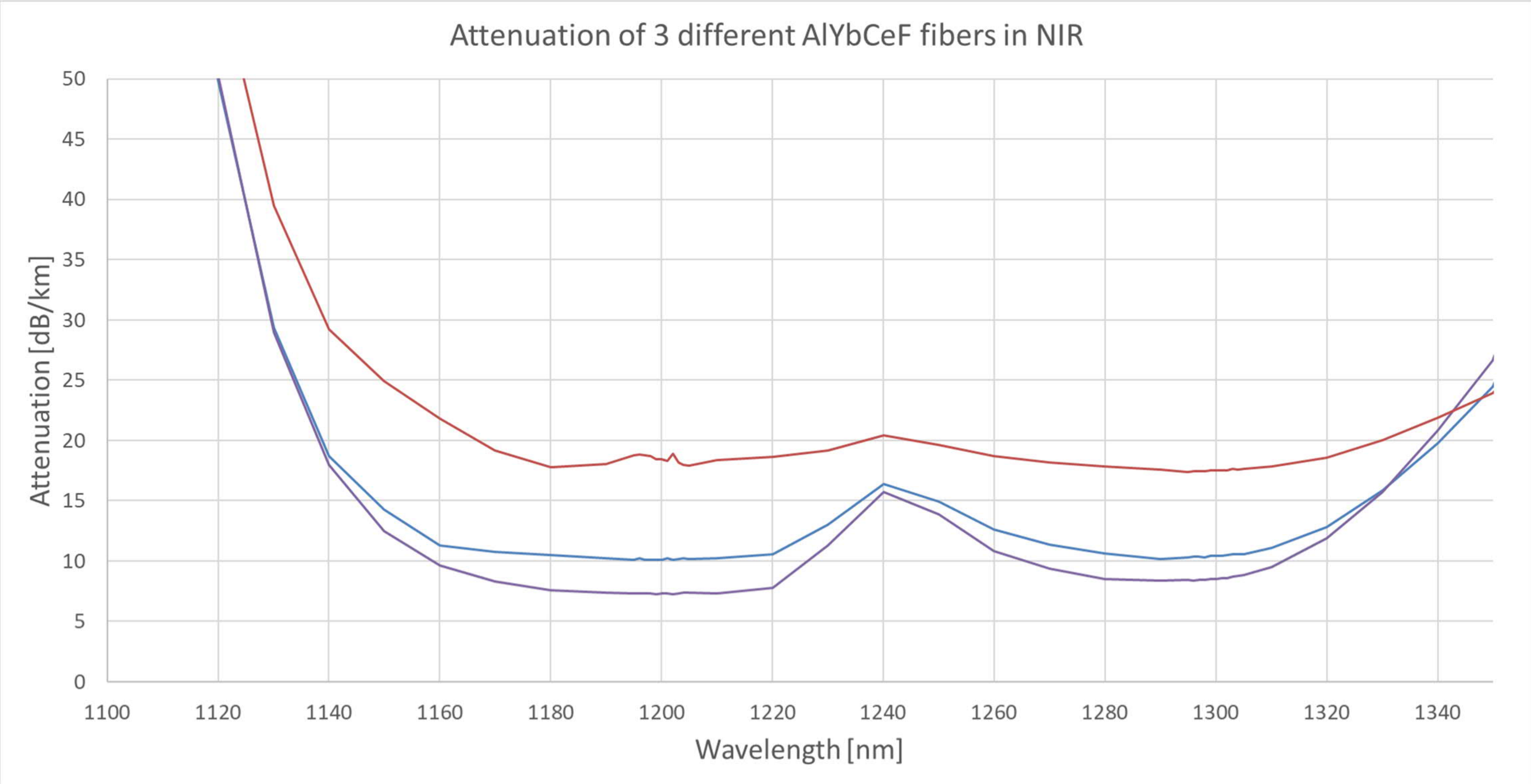
AlYbCeF preform-to-preform repeatability

- Three preforms were made using identical recipe.
- Glass structure: Al_2O_3 1.2mol%; Yb_2O_3 0.12mol%, Ce_2O_3 0.05mol%, SiF_4 used for down-doping
- Core diameter: 2.8mm, 7 deposition layers.



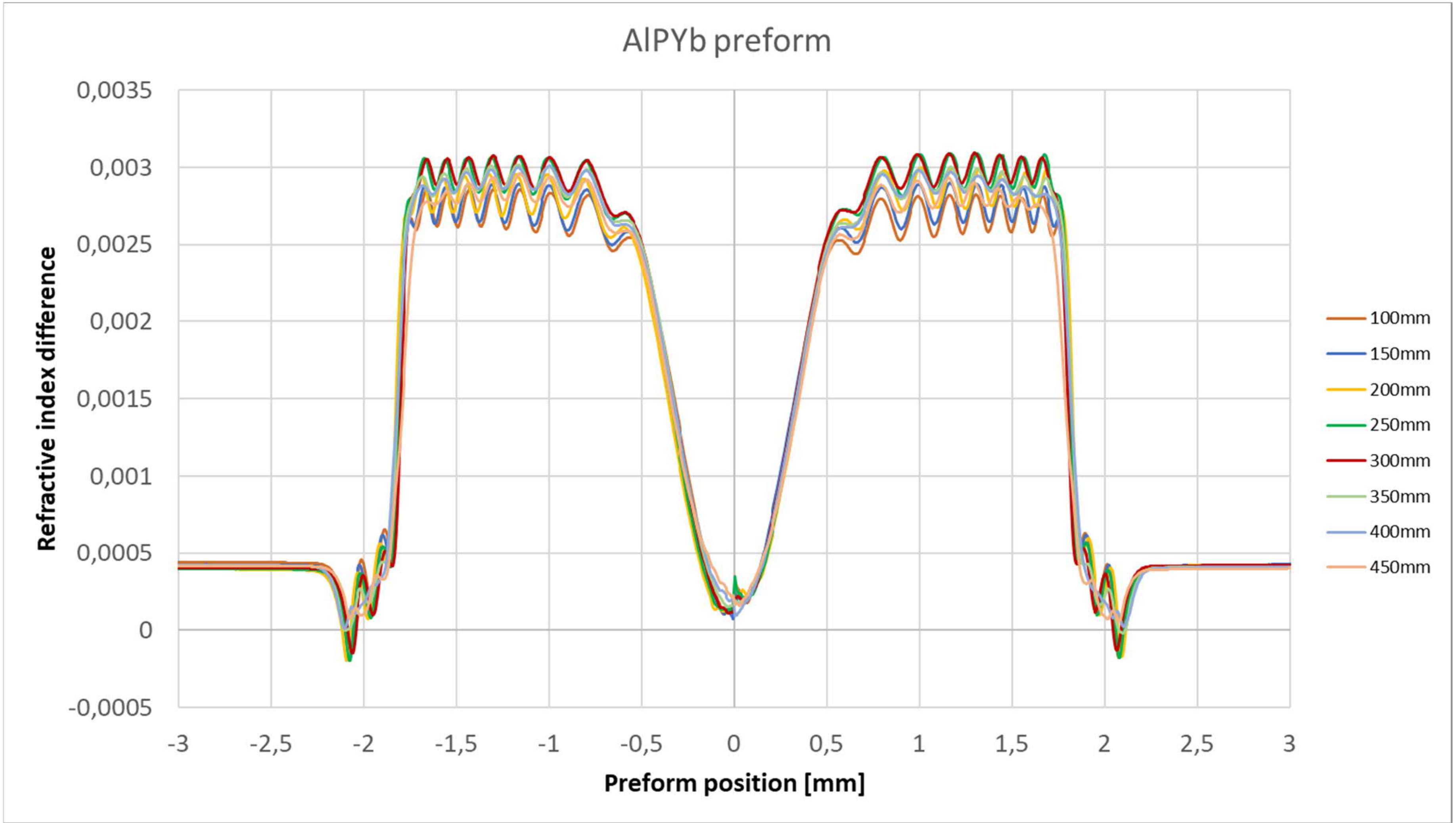
AlYbCeF core attenuation

- Active fiber attenuation strongly depends on the glass composition and drawing conditions.
- Below is a graph showing core attenuation of three differenet AlYbCeF fibers:



AlYbCeF profile longitudinal results

- Three preforms were made using identical recipe.
- Glass structure: Al_2O_3 2.5mol%; P_2O_5 4.5mol%; Yb_2O_3 0.20mol%
- Core diameter: 3.6mm, 9 deposition layers; deposition length 600mm.



OptiFACT control system for HTS doping

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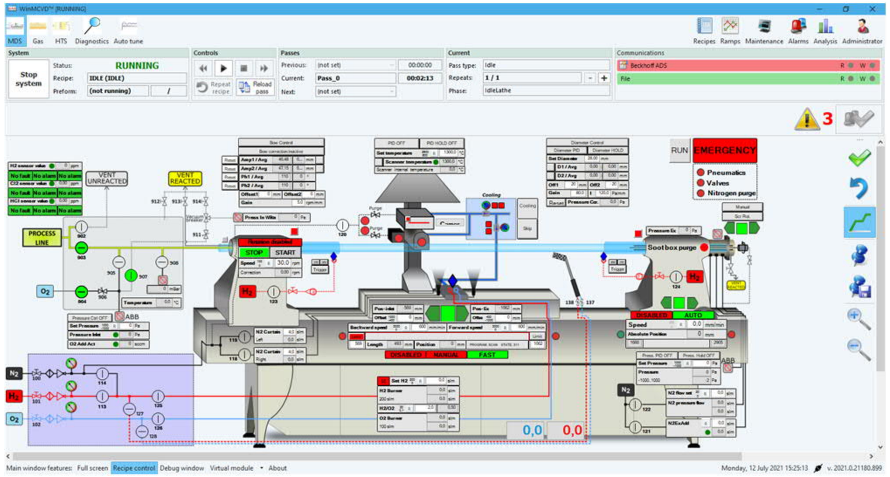
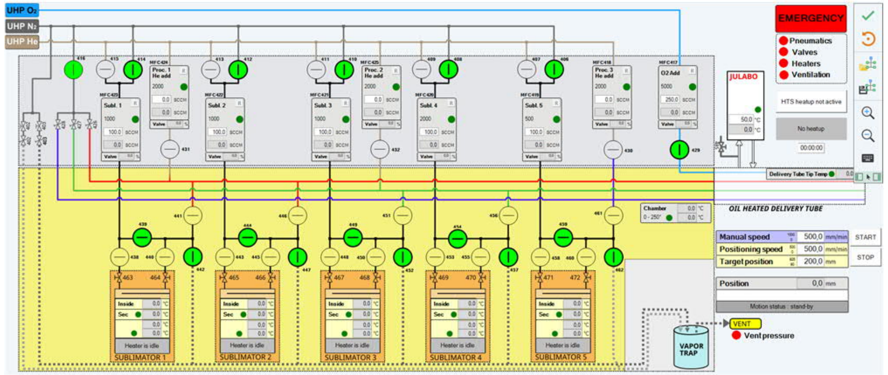
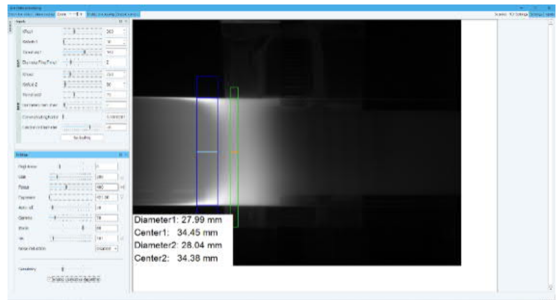
- Control system features
  - HTS control system is fully integrated into MCVD system controller, using common GUI interface (see OptiFACT add link),
  - robust industrial PC computer with Windows 11, running OptiFACT program, with multiple control screens, monitor capability,
  - automation components from Beckhoff including PLC, TwinCAT controls, fast peripheral I/O units, digital interfaces (EtherCAT,..), safety functions

- OptiFACT software features
  - runs under Windows 11 (other version on request)
  - password protected access level (operator, engineer, system administrator)
  - recipe system with import/export to spreadsheet
  - fully automatic recipe execution, with full-time manual control, chain recipe execution
  - recipe parameter ramp generator and editor
  - extensive logging options
  - on-line process analyzer (parameter graphing)
  - PID controllers with auto optimization
  - remote desktop access
  - DEMO version can be used off-line for training or off-line recipe preparation







HTS services and support

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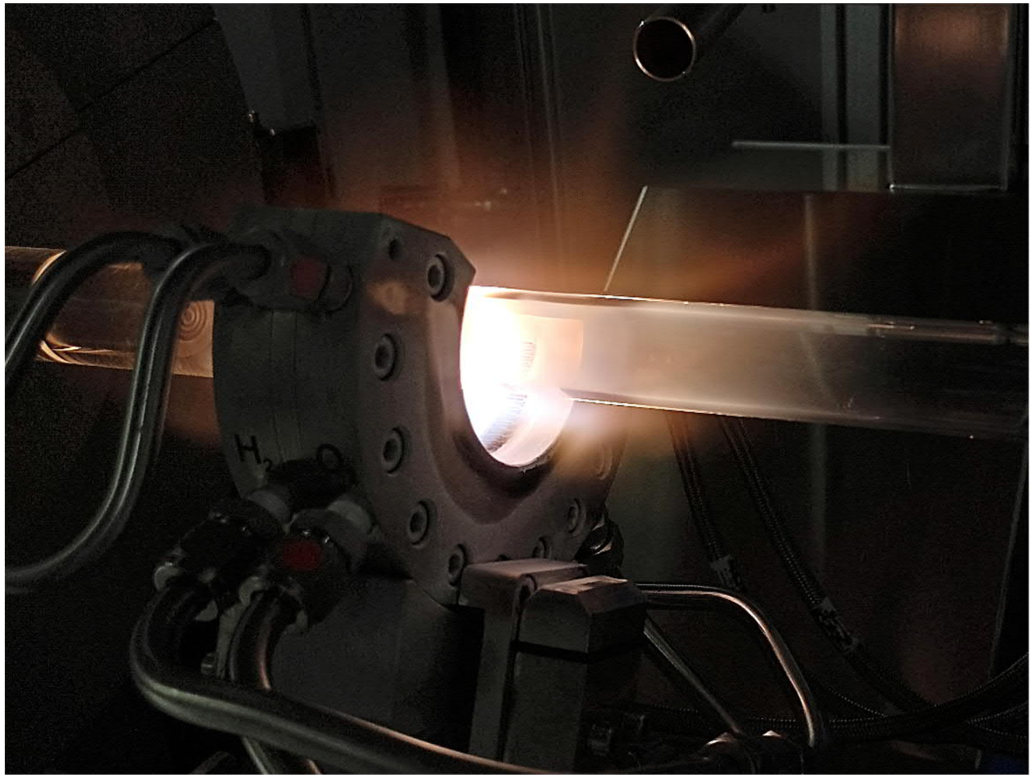
- Bimes services and support
  - service provided from Bimes site in Slovenia
  - manned support desk for quick and efficient customer support, using ticketing system and centralized mail account
  - on-line support using communication services over internet
  - spare part and consumable service
  - raw material recommendations, material delivery on request
- OptiFACT services
  - year free upgrades inside same product version during warranty
  - free 20 hours of programming service during warranty
  - remote update, version upgrade (according to customer's preference)
  - semi-open system allows customers to make changes, add or remove component
  - functionalities added or developed on requestother interfaces built-into
- HTS and optical fiber process support
  - service provided by Plasil d.o.o., Bimes partner from Slovenia
  - operator and engineer training for MCVD and high temperature doping processes
  - process support for fabrication of basic optical fiber types (active or passive)
  - on-line process support using communication services over internet



OPTION: MIF Furnace for MDS system

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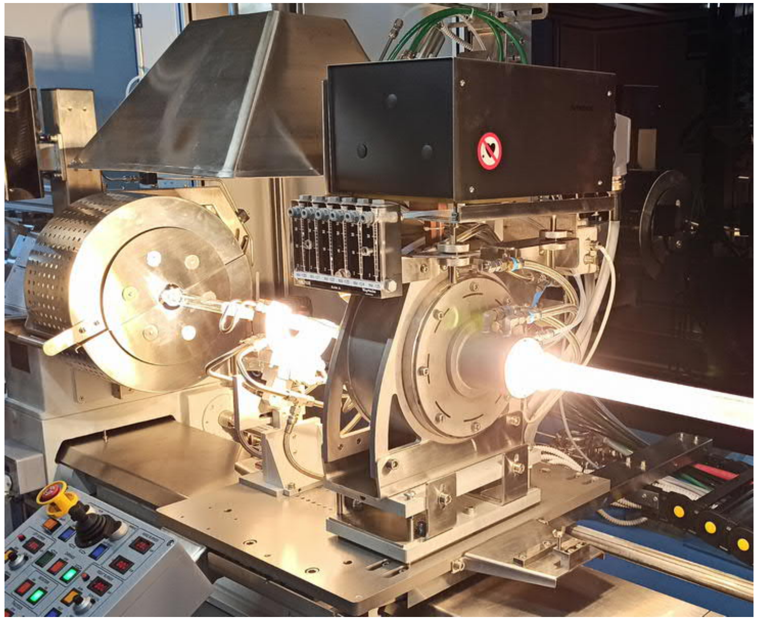
JUTRONIC d.o.o.



Furnace process and equipment

- FCVD process advantages
  - installed in parallel with H_2/O_2 burner
  - furnace used for collapse process and jacketing
  - with furnace collapse time is reduced to 40-50% of time required with burner use
  - reduced internal diffusion and diffusion from outside
  - improved preform geometry due to fast process and symmetric heat source
  - large preform capability
- MCF furnaces
  - graphite induction principle
  - simple graphite heating element and insulation shape, reduced cost of operation
  - maintenance and operation similar to draw tower furnaces
  - for substrate tube OD up to 45 mm
  - low graphite burn-out due to gas window design
  - furnace can be removed from the carriage when used
  - best performance of combined burner/furnace system is achieved with long bed preform lathes
- Accessories
  - integration into existing or new MCVD systems
  - support for technology transfer
  - inductive generators from 40 to 60 kW
- Recommendation
  - use of MIF furnace in fabrication of rare earth-doped fiber preforms is strongly recommended for semi-industrial or industrial fabrication environments

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