Sapphire crystal growth at ILM

K. Lebbou

Institut Lumière Matière (ILM), UMR5306 Université Lyon1-CNRS, Université de Lyon, Lyon 69622, Villeurbanne Cedex, France

E-mail: kheirreddine.lebbou@univ-lyon1.fr

ÉQUIPES LUMINESCENCE - MPN - NAVIER - (N)ME - SOPRANO

g-MAG

MATERIAUX POUR L'ASTRONOMIE GRAVITATIONNELLE

Outline

1- Introduction (Crystal growth at ILM Laboratory)

2- Sapphire crystal growth

3- Conclusion

1- Introduction

Crystal Growth

Process, Technology
- LHPG
- µ-PD
- Czochralski
- Kyroupolos

Materials
- YAG,LuAG, GAGG
- ASL, Sapphire,
- Oxides, Eutectic
- LGT,LGS,
- Perovskite (ABO_3)

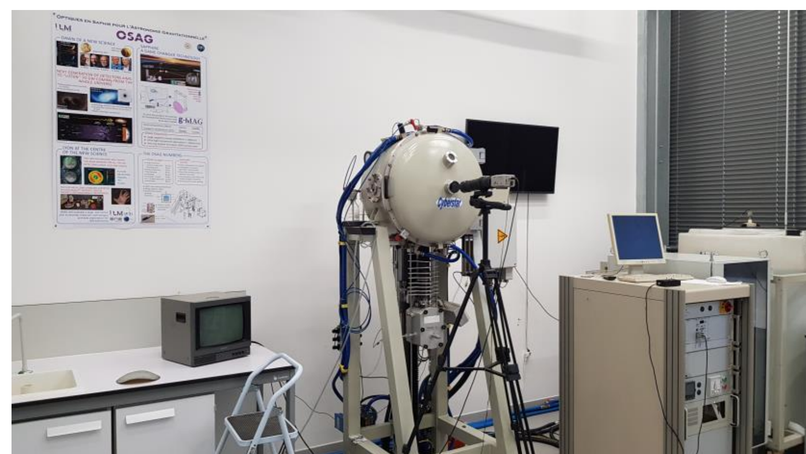
Applications
- Scintillation, Laser
- Aeronautic
- Piezoelectricity
- Gravitational wave detection(OG)
- 5G (2nd et 3rd generation)

INSTITUT LUMIÈRE MATIÈRE

Crystal growth research is localized in two sites

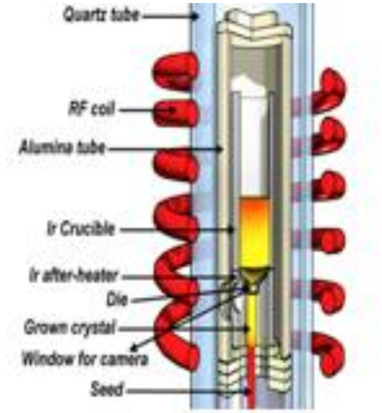
Raulin Building

Axel'one Building



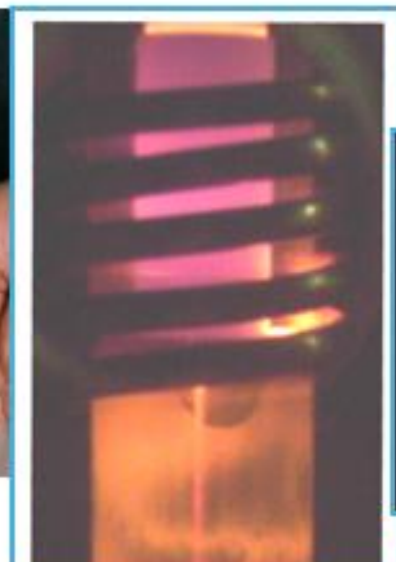
Machines for fibers crystal growth (4µPD +1LHPG)

RF Machine using metal chamber

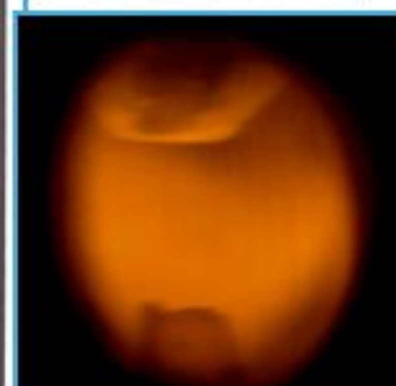


RF machine using Quartz tube chamber

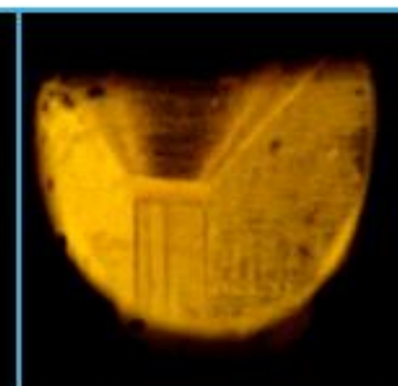
Seed, crucible and thermal insulation



RF Heating

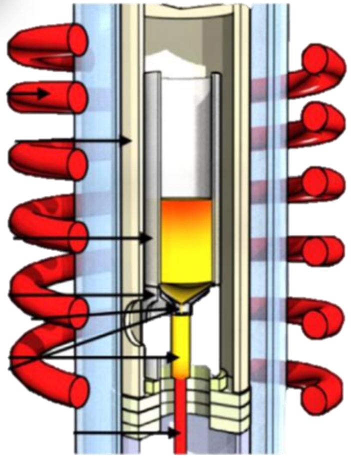


Seeding



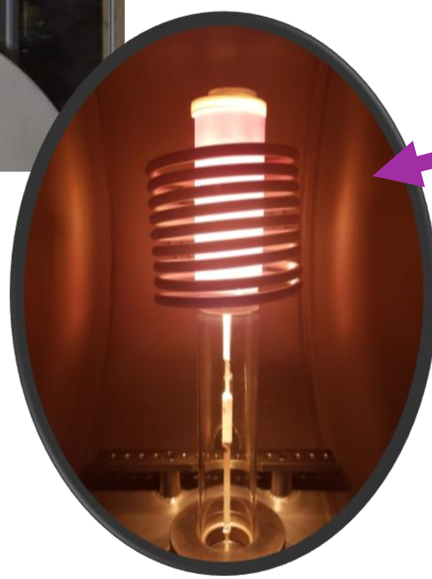
Connection and fiber growth

Sapphire fibers crystal growth by micro-pulling down technique



We use two RF machines

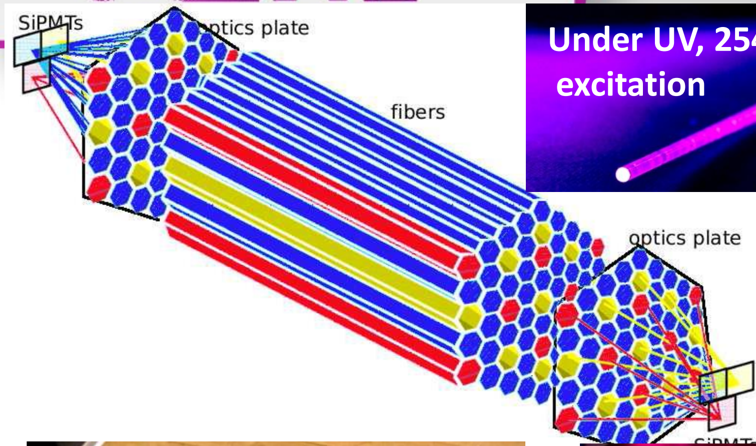
@ Raulin

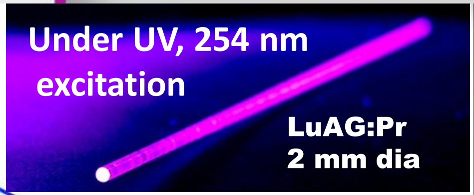


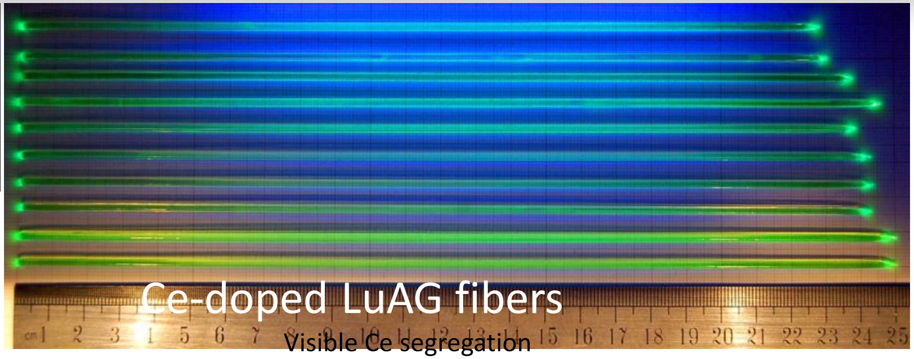
RF heating

@ Axel'one

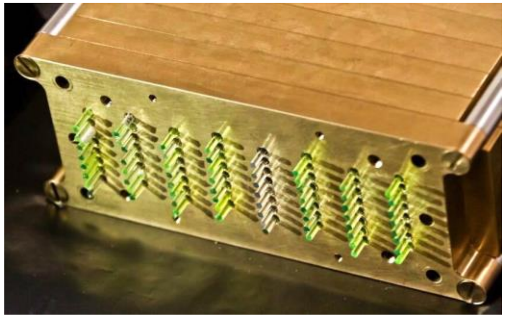
Some results: High energy physics scintillating fibers calorimeters design

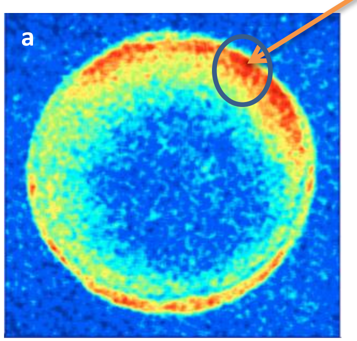


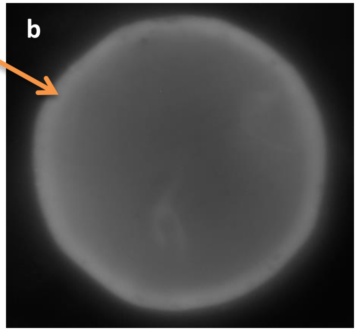




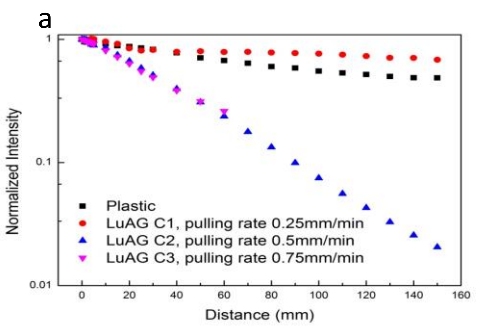
Visible Ce segregation
in the periphery of the fiber

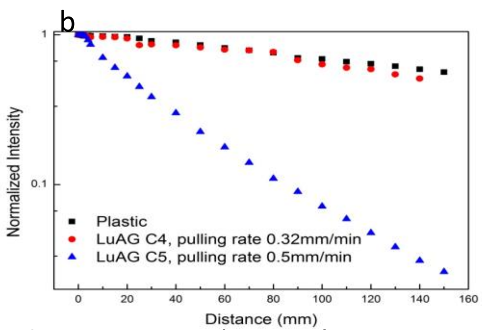






Cross section image of the Ce (0.12at%)-doped LuAG fibersexcited under X (a) and electron (b) showing a gradient concentration of Ce dopant well visible in the periphery.

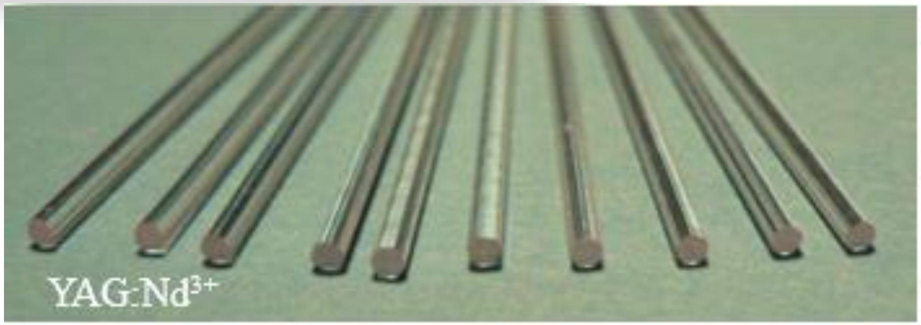


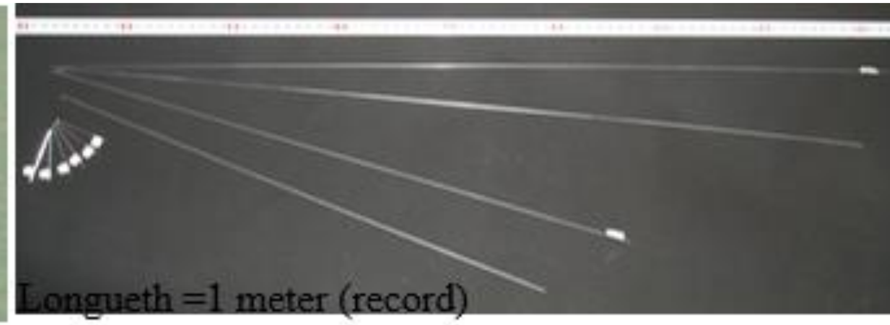


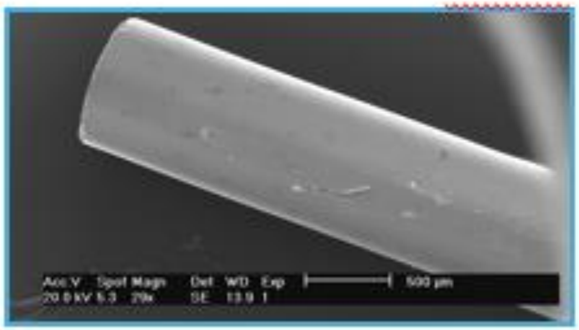
Attenuation curves for plastics fiber and Ce (<1at%) –doped LuAG fibers as a function of pulling rate

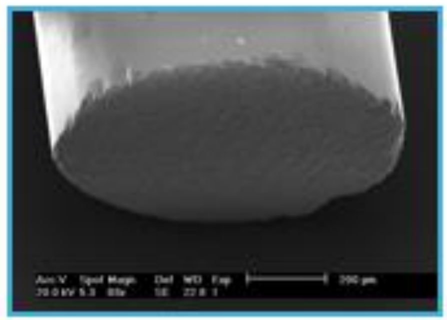
-X.Xu, K.Lebbou et al ,
Acta Materialia 67 (2014) 232–238
-A Belsky, K Lebbou et al,
Optical Materials 92 (2019), 341-346
-O. Sidletskiy, K. Lebbou, et al
CrystEngComm, 2019, 2019, 21 (11), 1728-1733

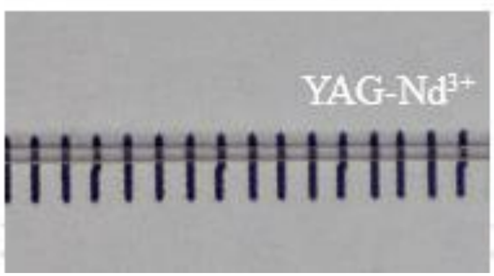
YAG-Nd fibers for laser application







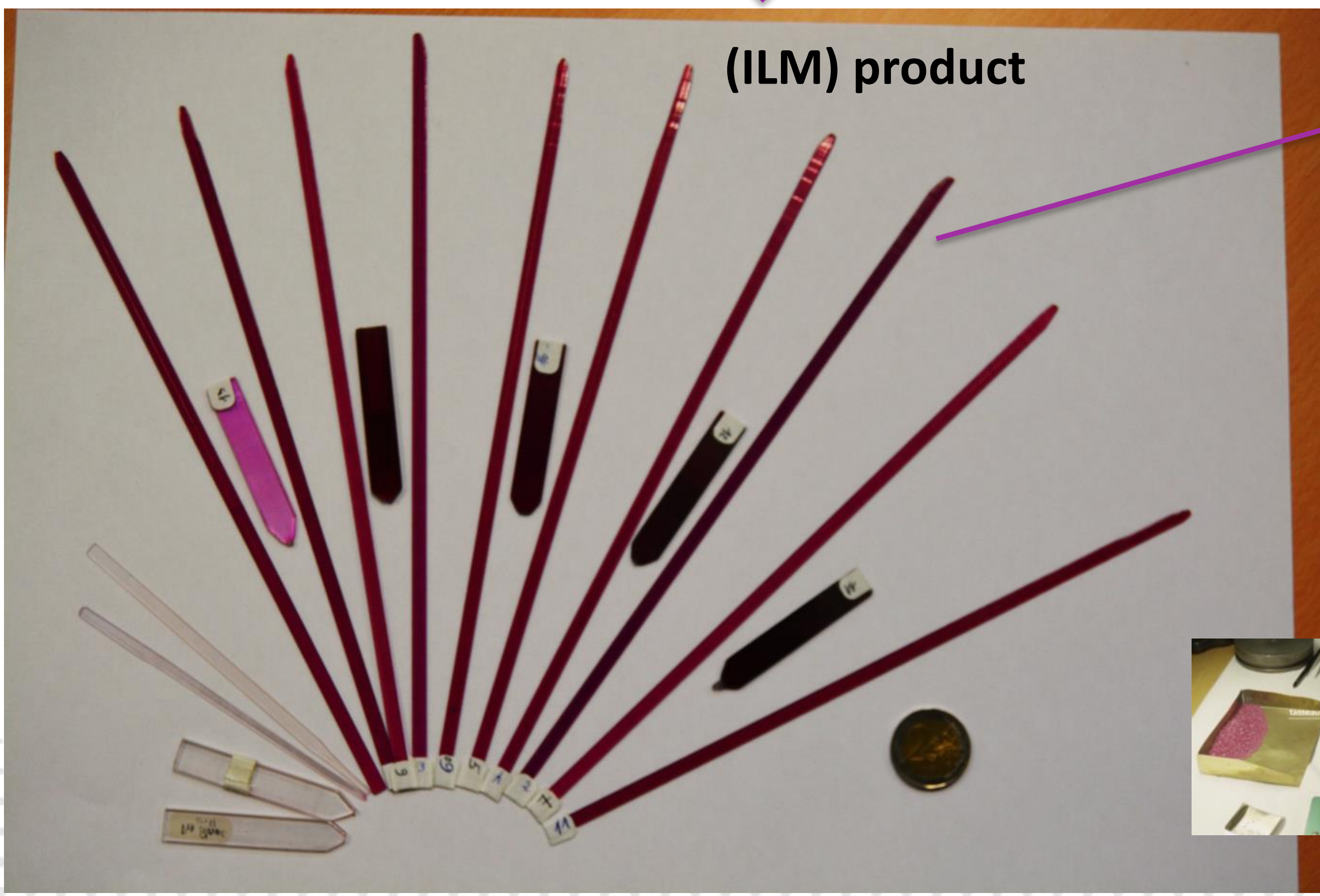


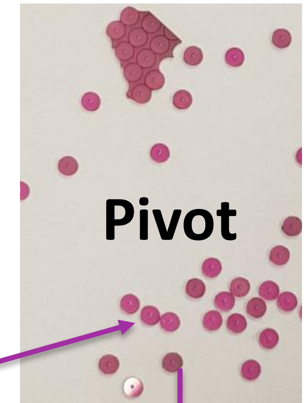


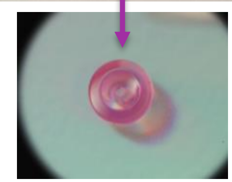
Taranis Laser Gain Module using YAG single crystal fibers as active element. This component is commercialised by fibercryst company created by LPCML Laboratory (ILM) in 2003.

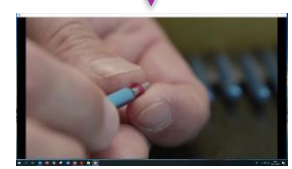
Sapphire for watch pivot (horology)

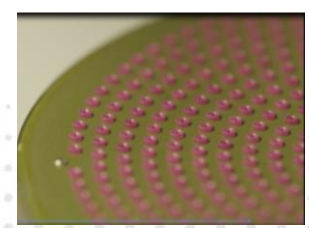
Watch designer



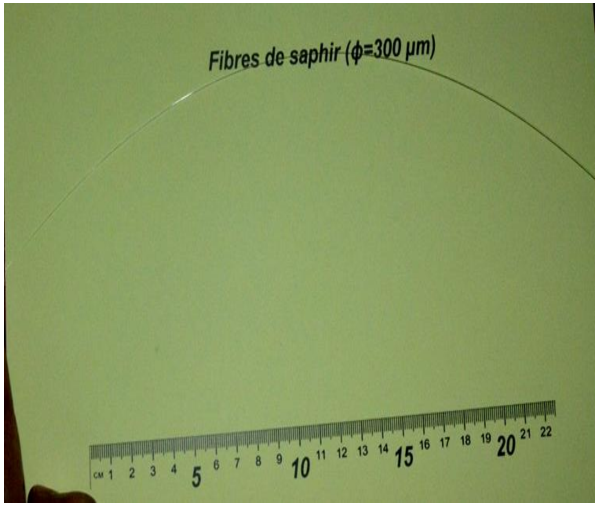


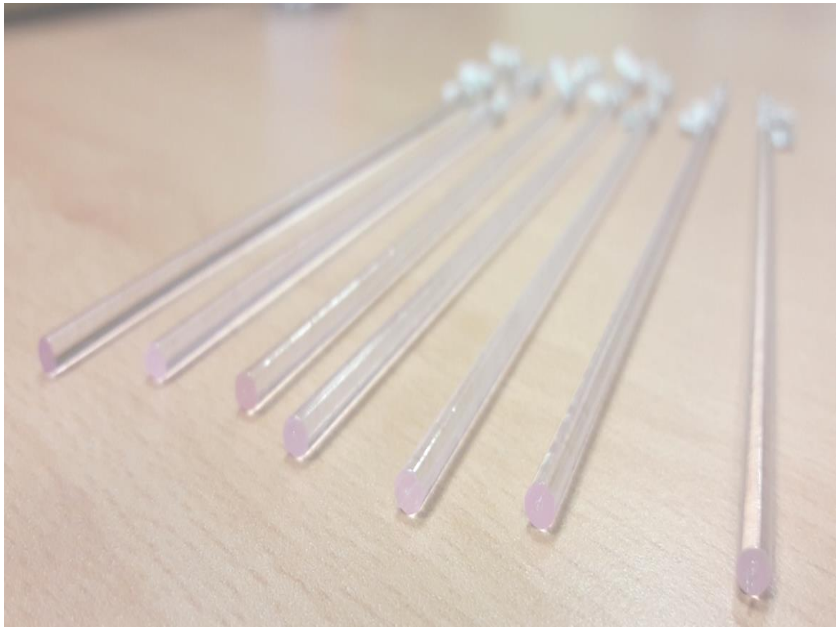






Fibers sapphire crystal growth

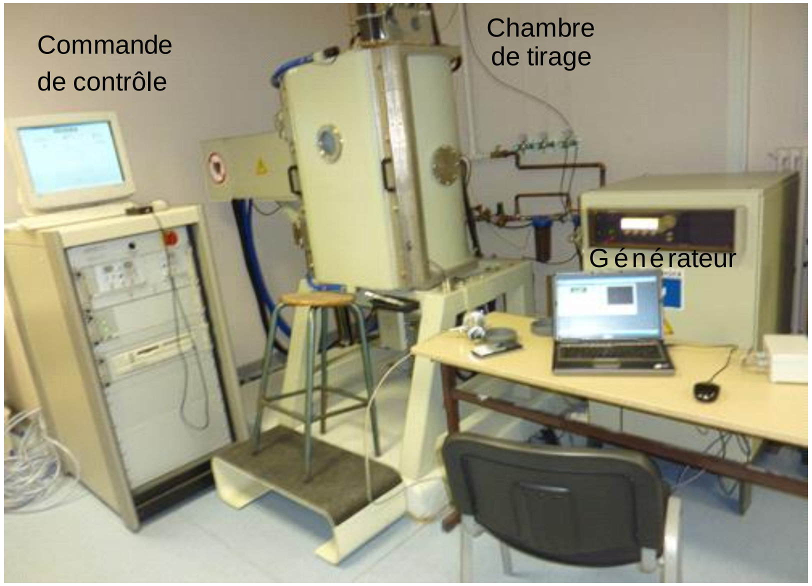


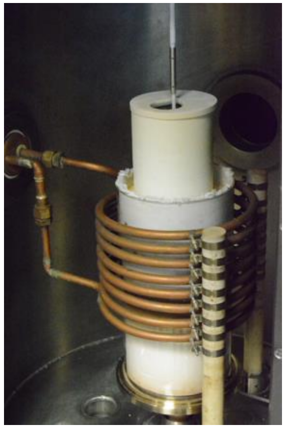


Sapphire fibers (diameter=1mm)

Czochralski crystal growth (ILM Laboratory)

Czochralski machine (CZ)



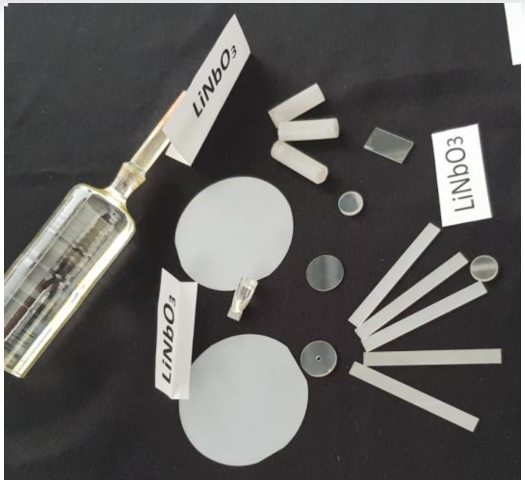


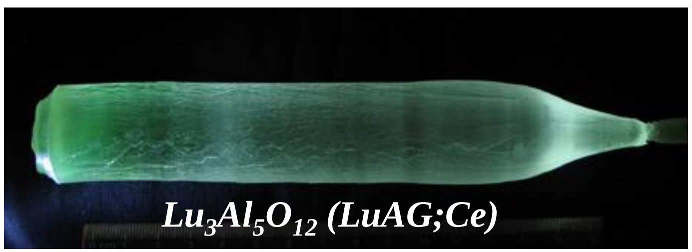
RF heating

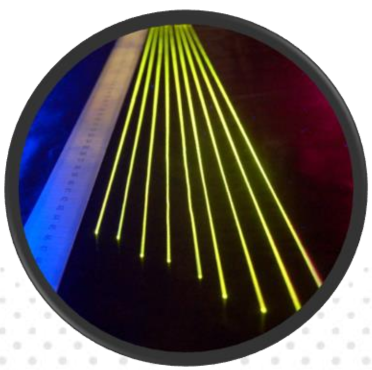
@ Raulin building

iLM INSTITUT LUMIÈRE MATIÈRE

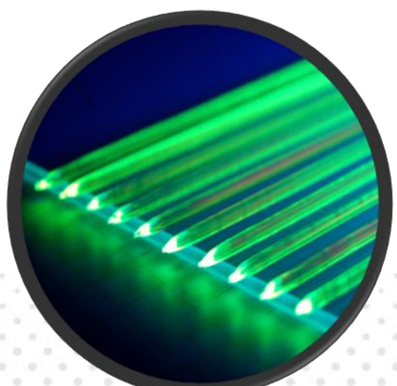
Crystal growth R&D research ILM



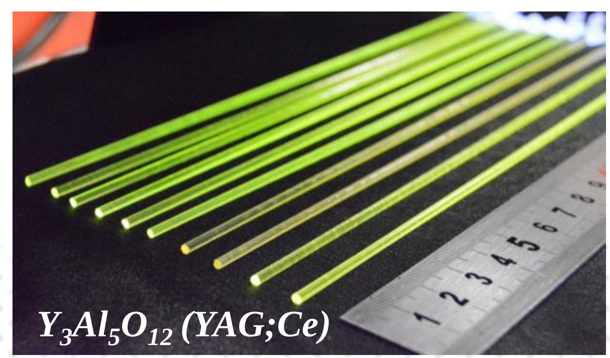




$Y_3Al_5O_{12}$ (YAG;Ce)

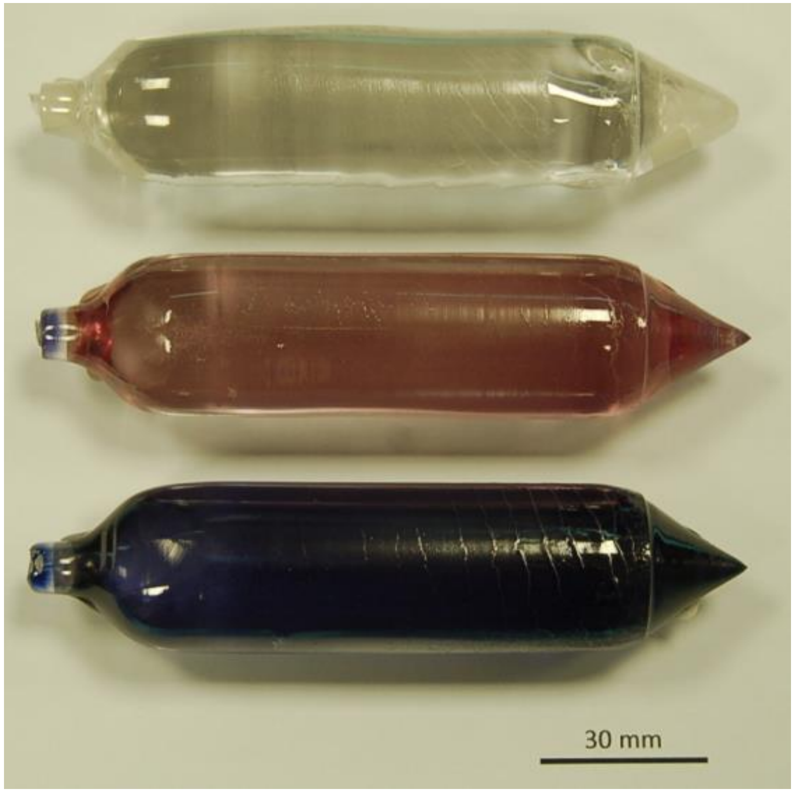


$Lu_3Al_5O_{12}$ (LuAG;Ce)



Bulk sapphire crystals growth by Czochralski technique

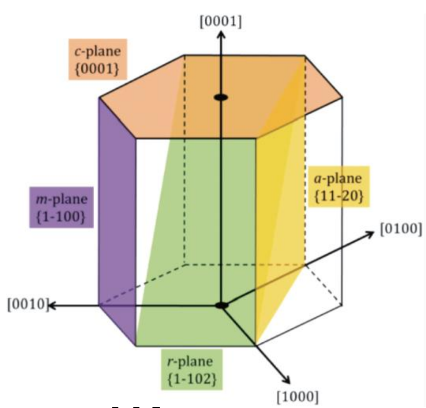
Different sapphire crystal and different dopants

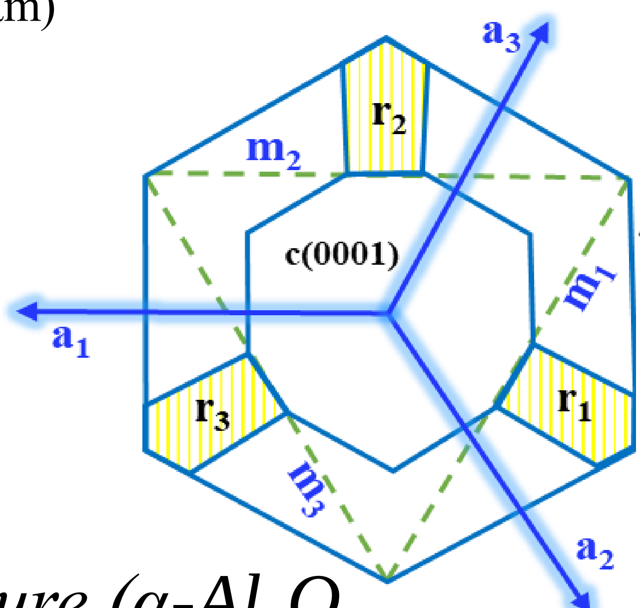


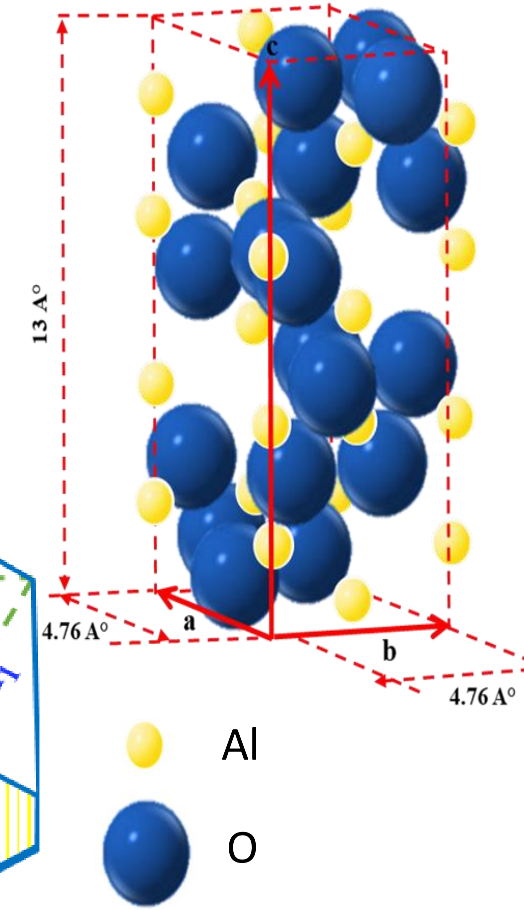
Growth conditions:
Atmosphere : Argon
V=2mm/h, Vr=10rpm,
Seed: A plane

2- Introduction to sapphire

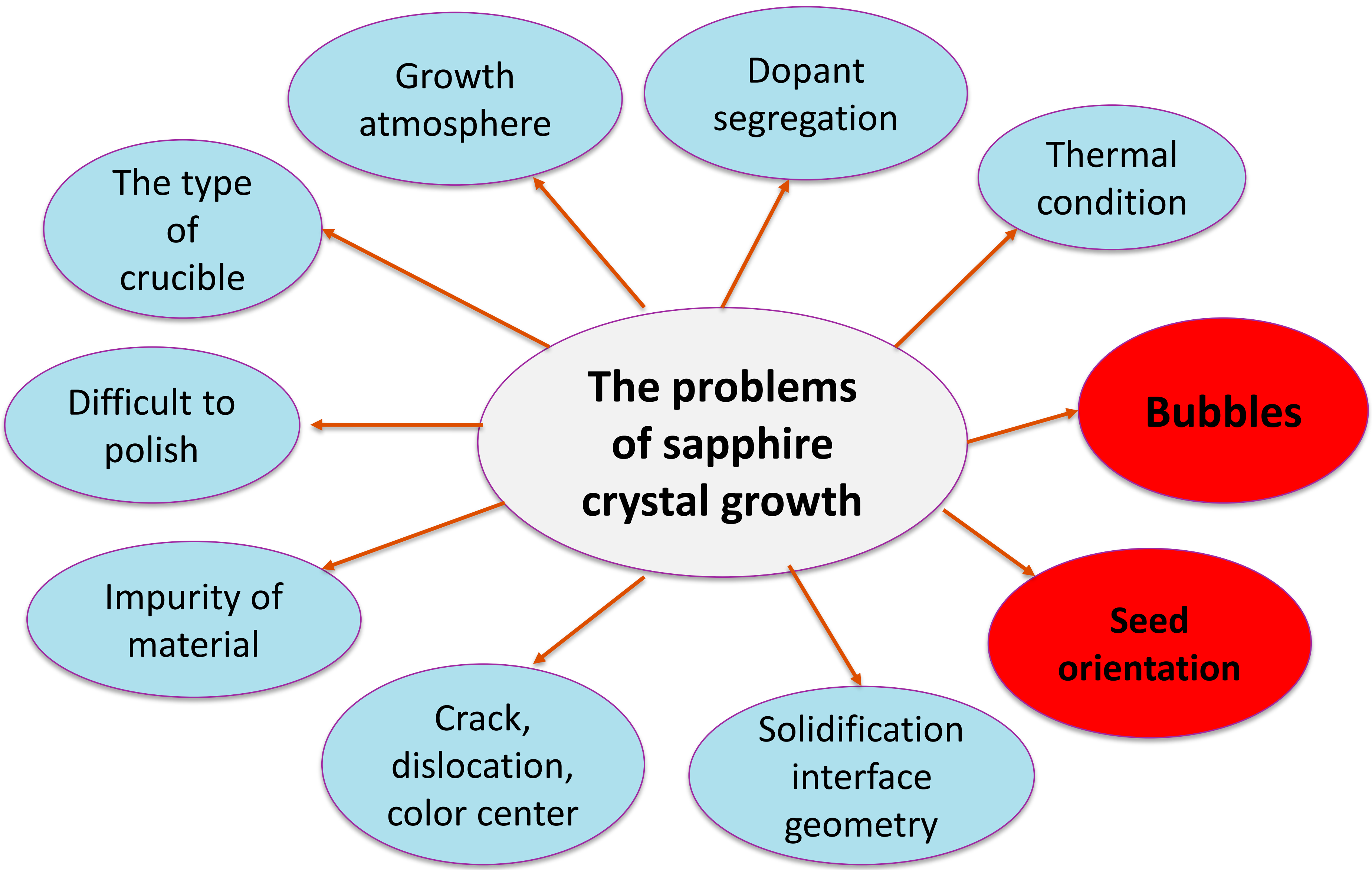
1. Simple chemistry formula (Al_2O_3)
2. Hexagonal-rhomboedric structure (a=4,376Å, c=13.00Å,12 atoms of Al and 18 atoms of O)
3. Melt congruently (2050°C)
4. Good traction resistance (400MPa)
5. High thermal conductivity(10W/mK)
6. Large transparency range (0.24 - 4µm)



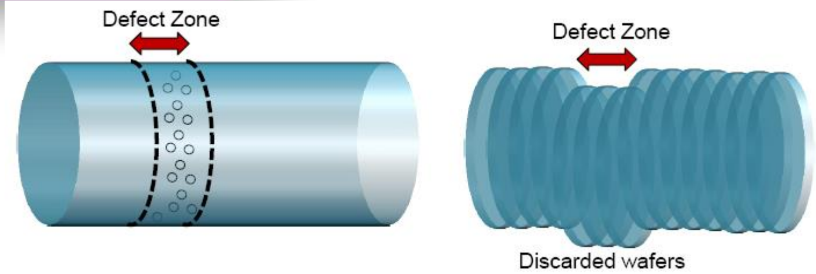




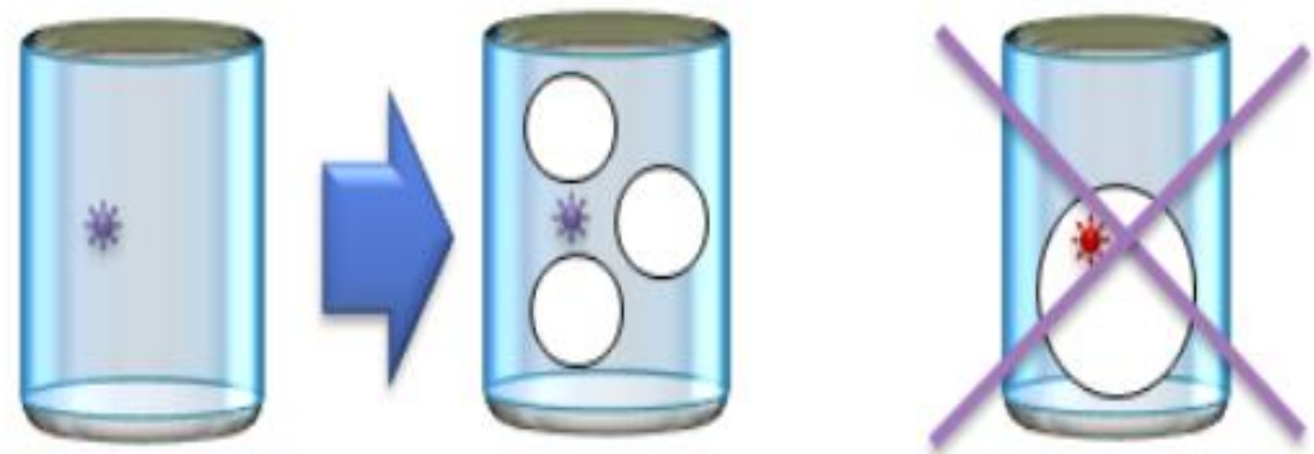
Saphir crystalline structure (α-Al_2O_3


The problems of sapphire crystal growth
Growth atmosphere
Dopant segregation
Thermal condition
The type of crucible
Bubbles
Difficult to polish
Impurity of material
Seed orientation
Crack, dislocation, color center
Solidification interface geometry

Bubbles defect



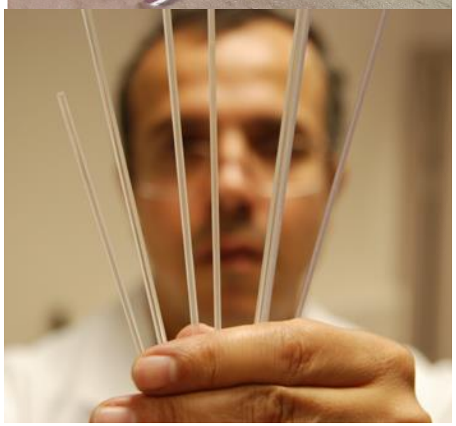
Impact of bubbles presence on sapphire cutting and polishing



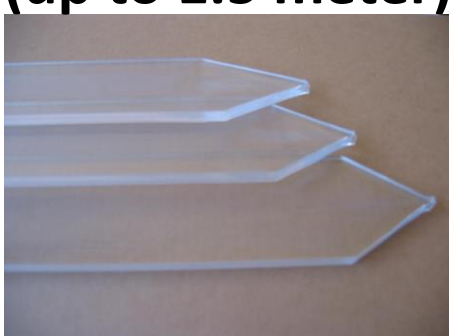
Boule with defects (bubbles) OK for small diameter, but lost for large diameter

iLM INSTITUT LUMIÈRE MATIÈRE

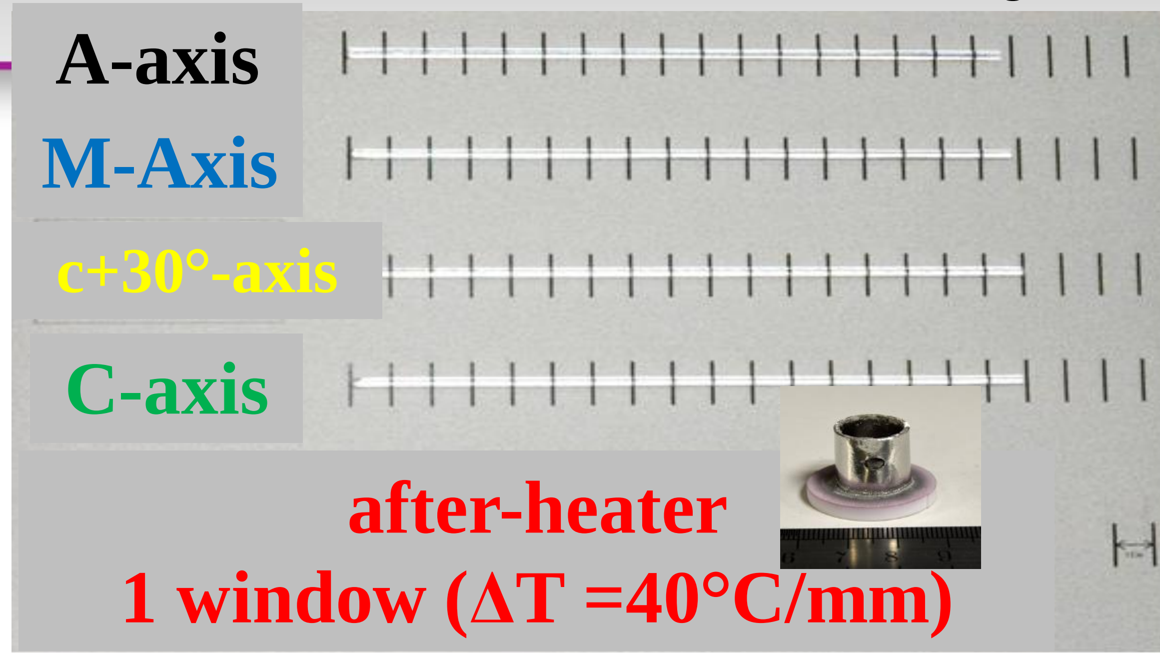
Fibers growth as a function of seed orientation and thermal gradient

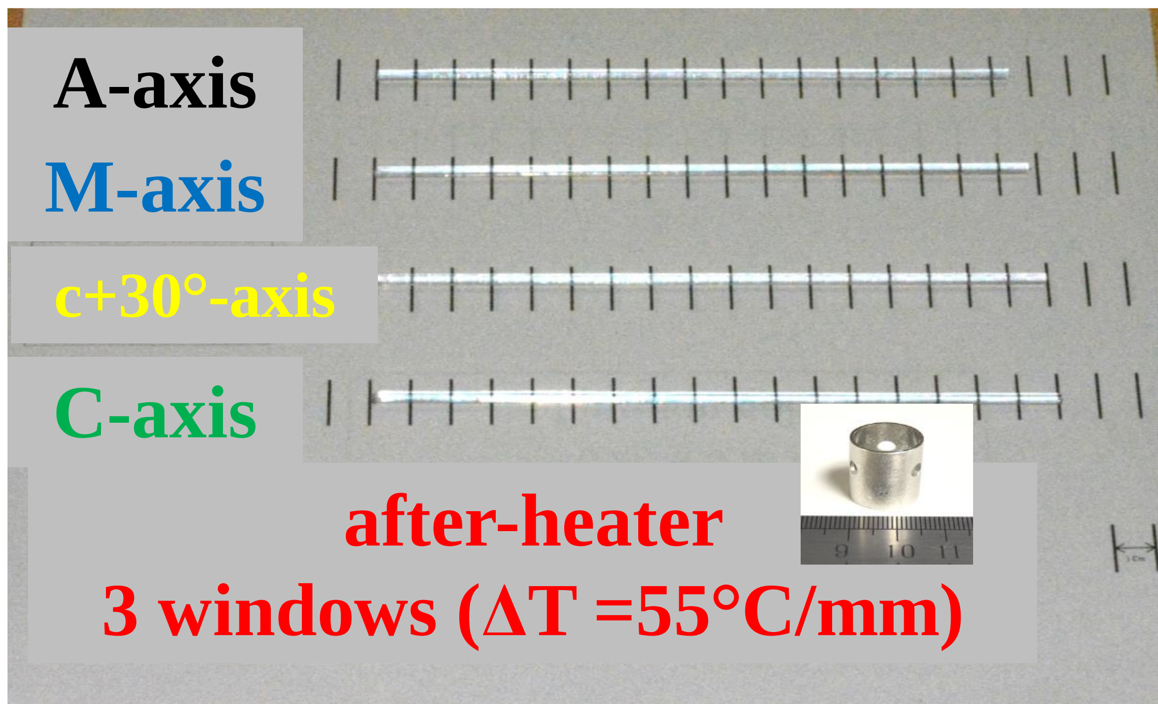


Fibers
(up to 1.5 meter)

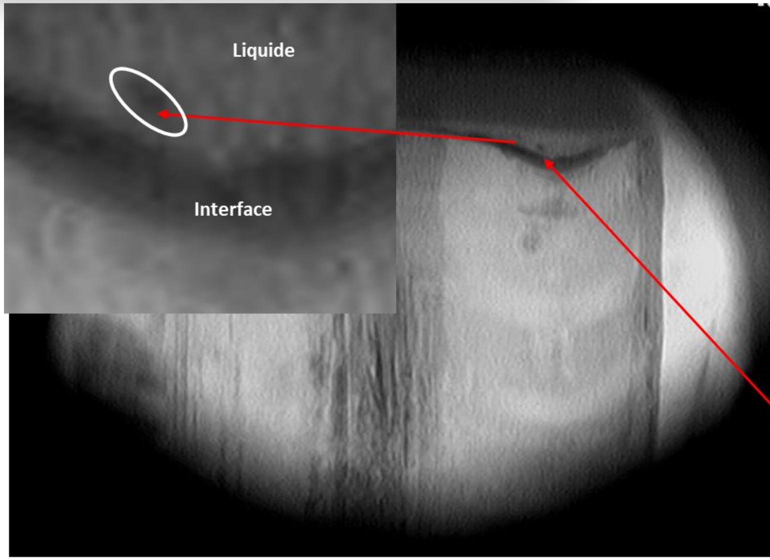


Ruban

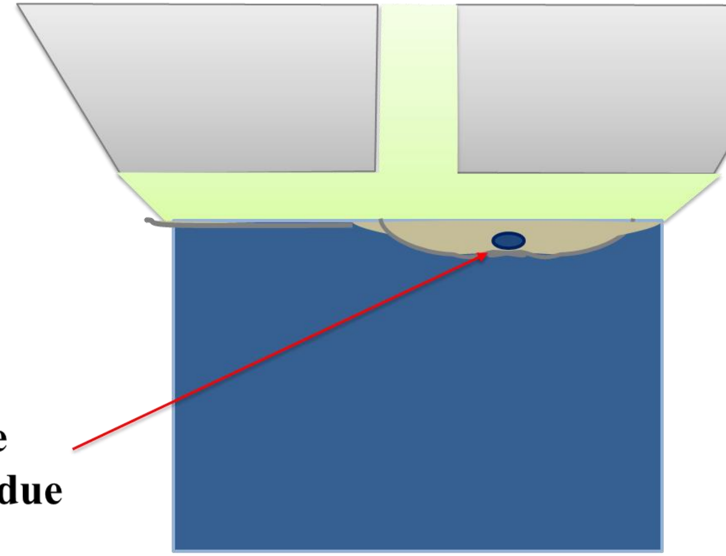


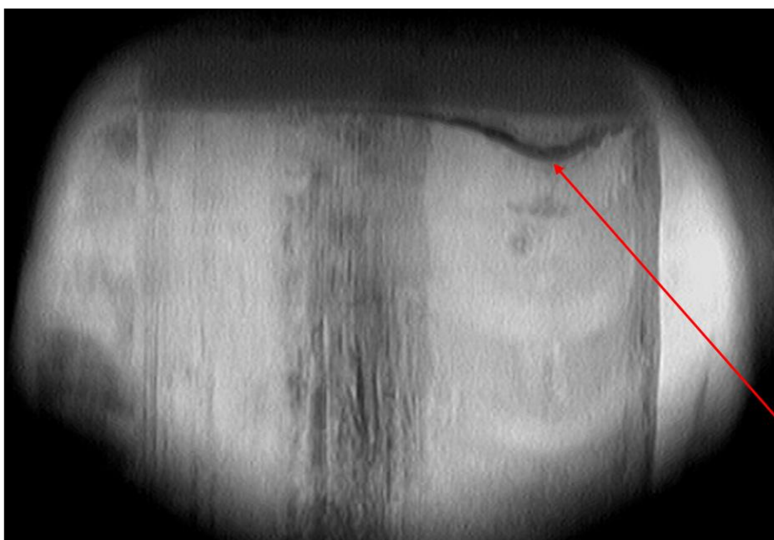


Bubbles incorporation during sapphire crystal growth

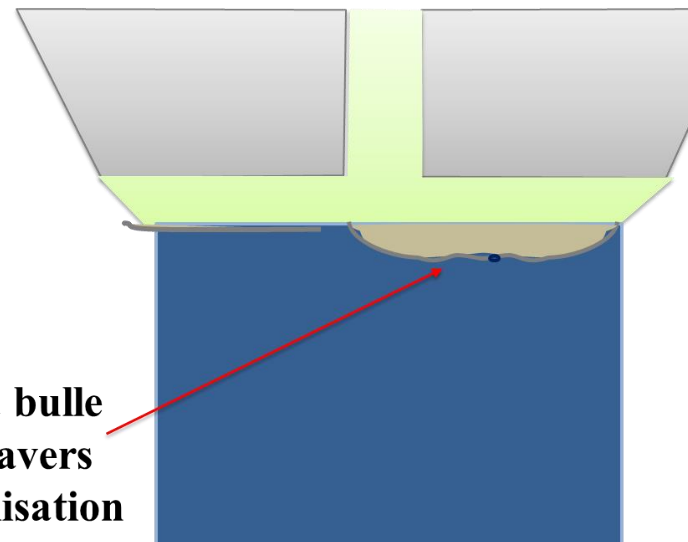


Bulle observée dans la zone fondue

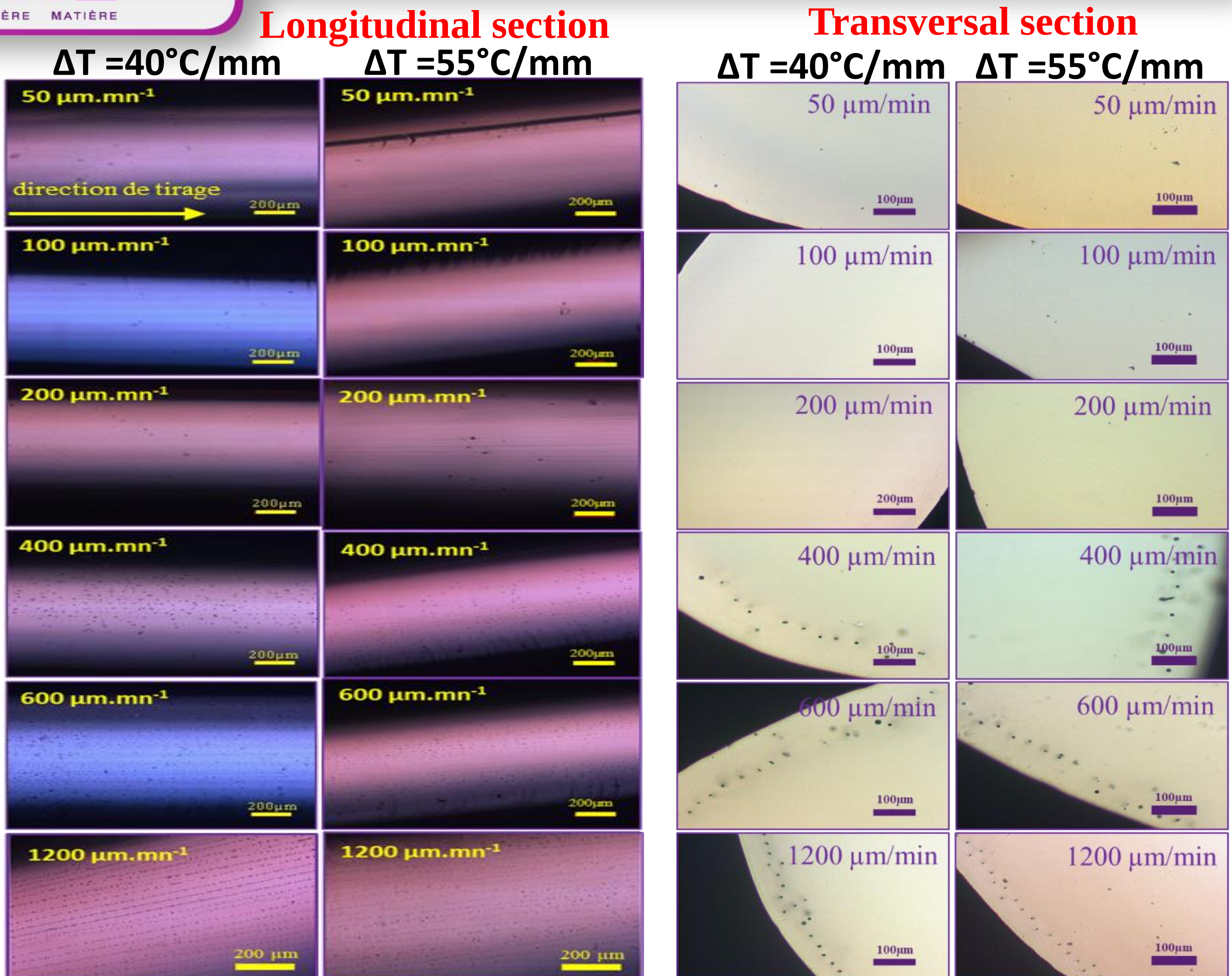




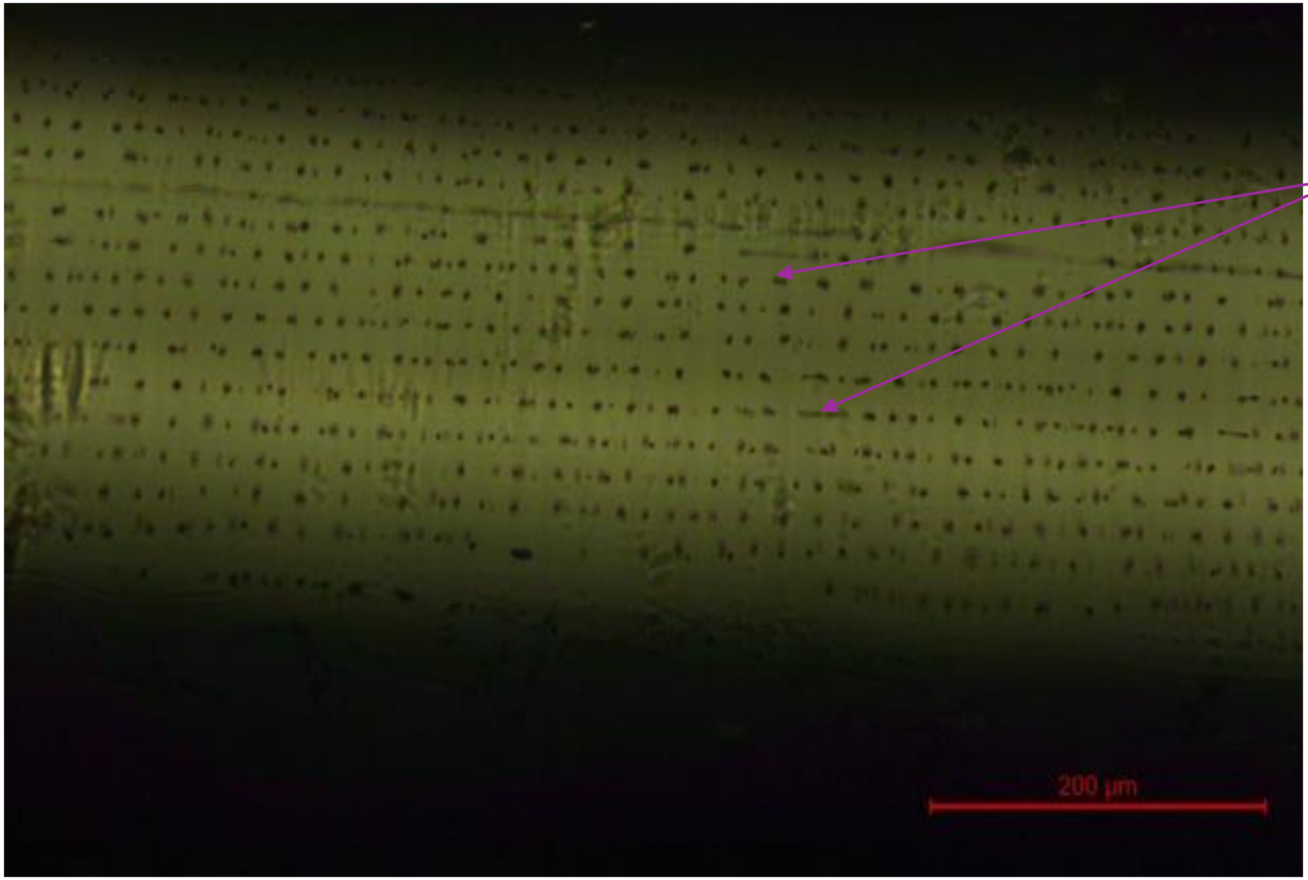
Incorporation de la bulle dans le cristal à travers l'interface de cristallisation



Effect of pulling rate and thermal gradient (ΔT) on bubbles distribution



High pulling rate (high bubbles density)



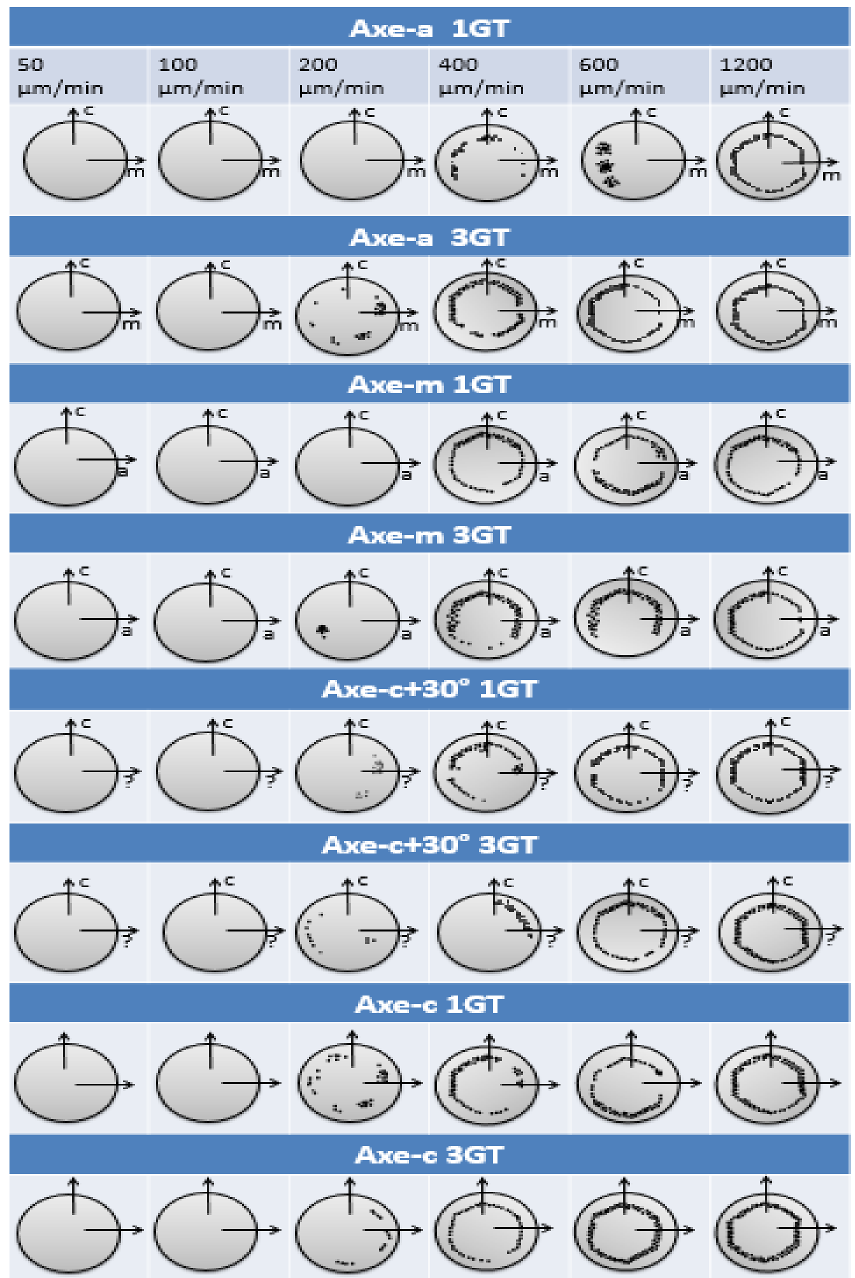
Bubbles defects

LuAG:Ce single crystal fiber , same behavior as sapphire fibers

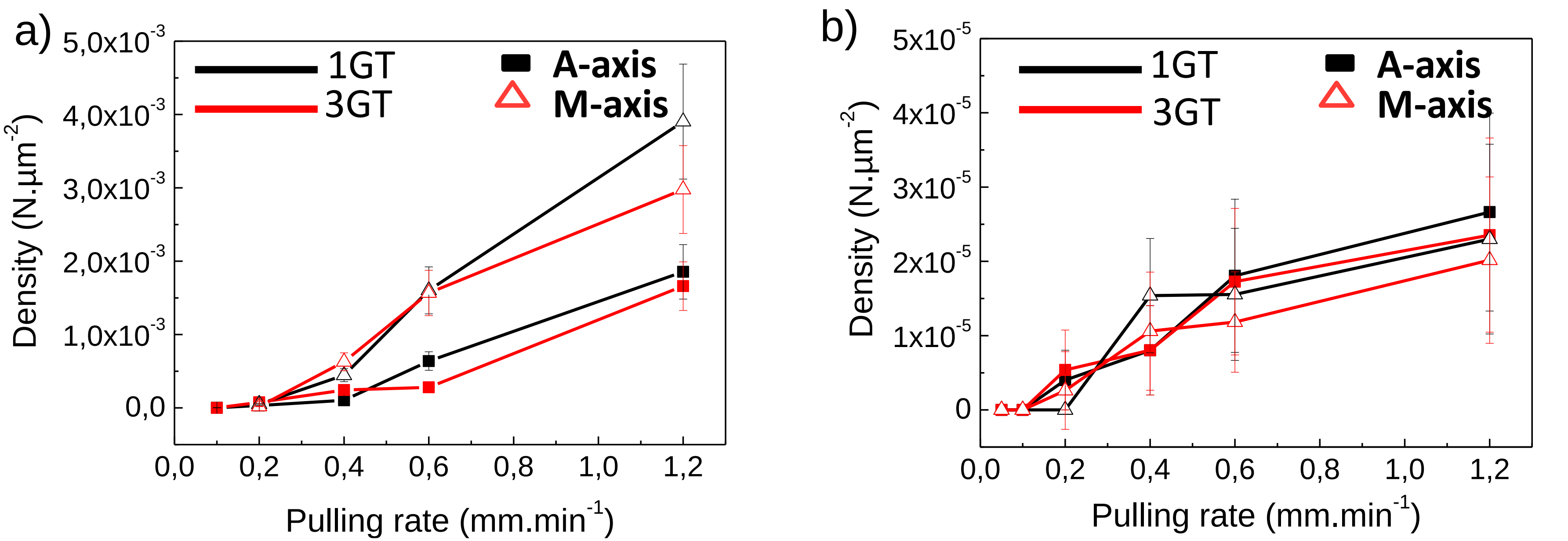
Impact of the crystallographic orientations on density and distribution of the bubbles

- The bubbles are organized on hexagonal shape

- The edge of the bubbles are perpendicular to the crystallographic axis



Effect of pulling rate and thermal gradient on bubbles density



1GT≈ 40°C/mm and 3GT≈55°C/mm)

- **V <200µm/min** (Crystals are bubbles free)
- **GT high (ΔT =55°C/mm , bubbles density decrease)**

R.BOUAITA et al, CrystEngComm. Vol.21 , (2019), 4200-4211
R.Bouita, PhD Thesis (defended 20 October 2019)

ILM INSTITUT LUMIÈRE MATIÈRE

Bubbles origin is expected to be connected to the alumina decomposition in the melt

$$Al_2O_3 \leftrightarrows AlO + AlO_2$$

$$Al_2O_3 \leftrightarrows Al_2O + 2O$$

$$Al_2O_3 \leftrightarrows Al_2O_2 + O$$

$$AlO_2 \leftrightarrows AlO + O$$

$$AlO_2 \leftrightarrows Al + 2O$$

$$Al_2O \leftrightarrows 2Al + O$$

$$Al_2O_2 \leftrightarrows Al_2O + O$$

E.A. Ghezal, H. Li, A. Nehari, A. Brenier, K. Lebbou et al, Cryst. Growth Des. 12 (2012) 4098–4103

H. Li, E.A. Ghezal, G. Breton, J.M. Ingargiola, A. Brenier, K. Lebbou, Opt. Mater. 2 (2014) 1-7

R.Bouiata , K.Lebbou et al, CrystEngComm. Vol.21 , (2019), 4200-4211

2- OSAG Project (Gianpietro Talk)
ILM –LMA collaboration
National project
(Idex breakthrough)

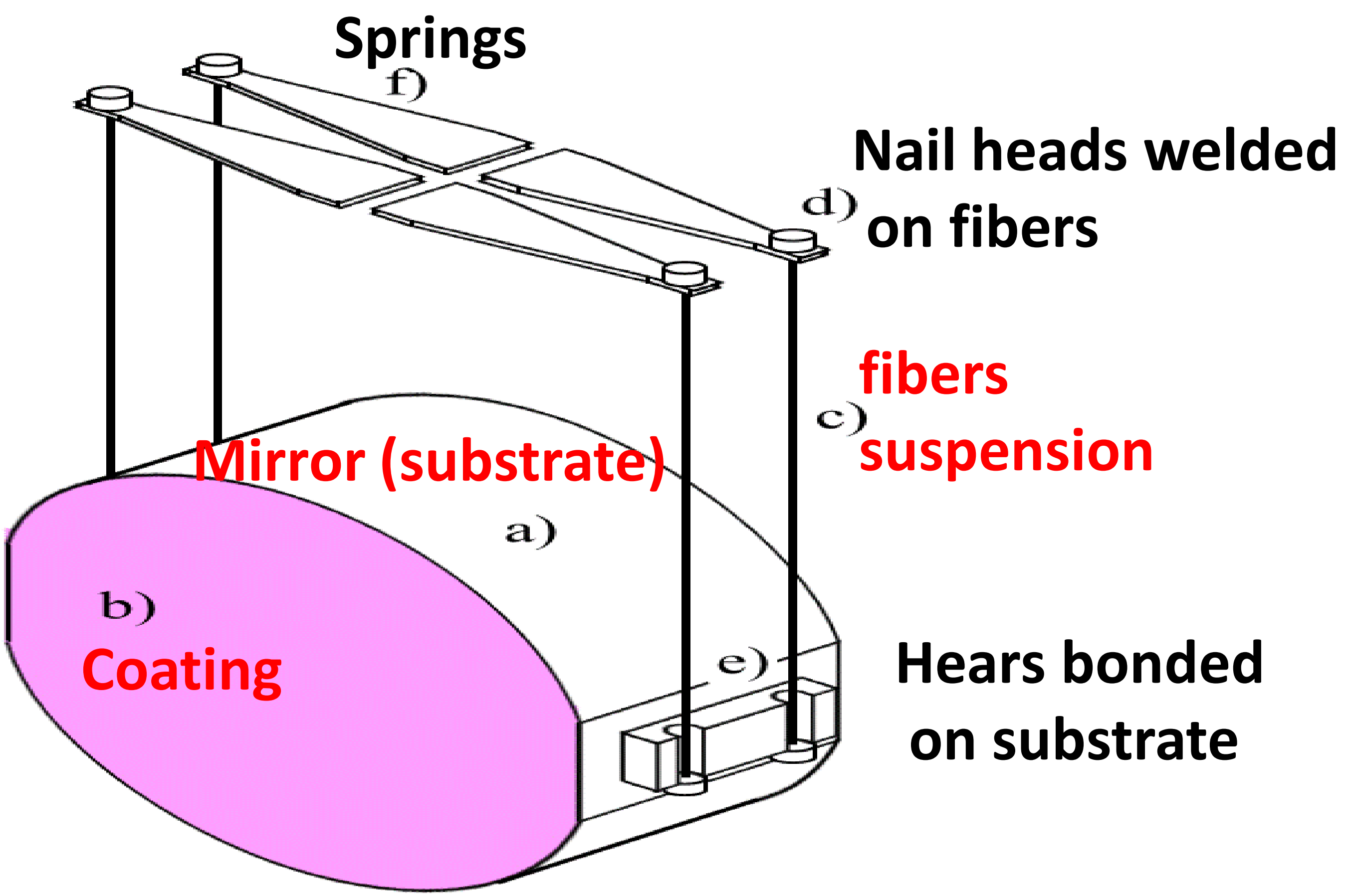
Mains targets of OSAG project

- Design and conception machine of large sapphire crystal (ϕ≈450mm)
- Sapphire crystal growth and finding the optimal protocol to pull performed sapphire without defects
- Analyze the material at the atomic level, in particular:

o Luminescence and Induced Breakdown Spectroscopy Laser for chemical composition analysis and mapping (in close connexion with the OPTOLYSE platform)

o Raman for the short scale structure and stress detection

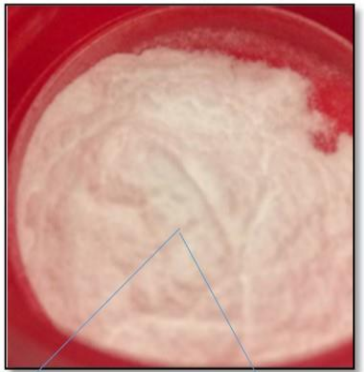
- Optical and mechanical characterization bench (fibers)
- Characterize the samples according to their properties: o elastic and thermal o density of bubbles and dislocations
- Metrology and coating deposition
- Development and characterization of ultra-low optical loss sapphire mirrors



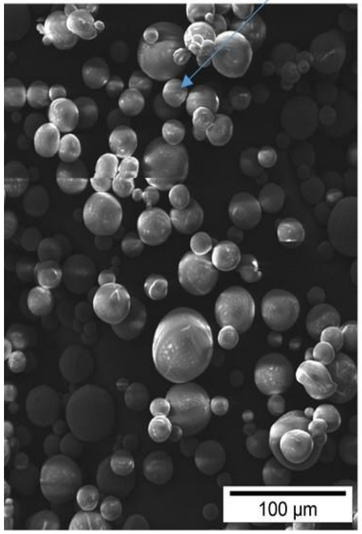
A typical test mass of a GW detector

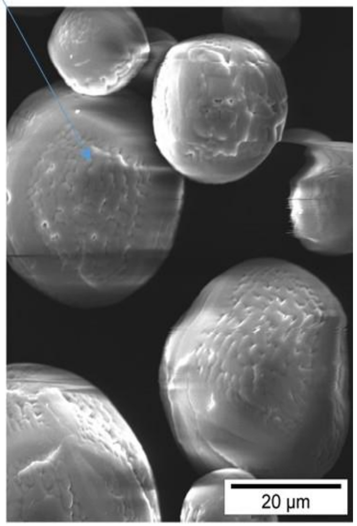
Raw materials optimization

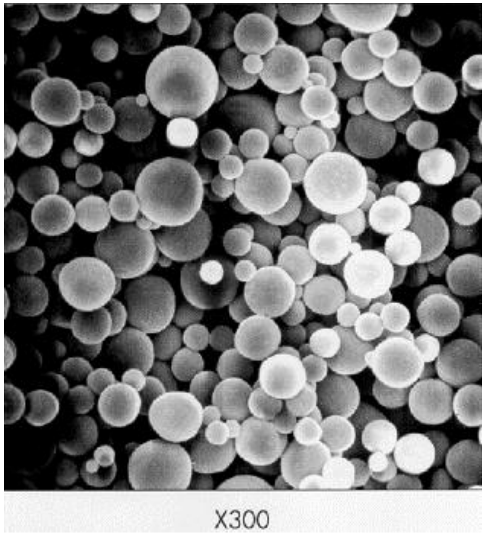
Dense alumina raw materials



Different sources

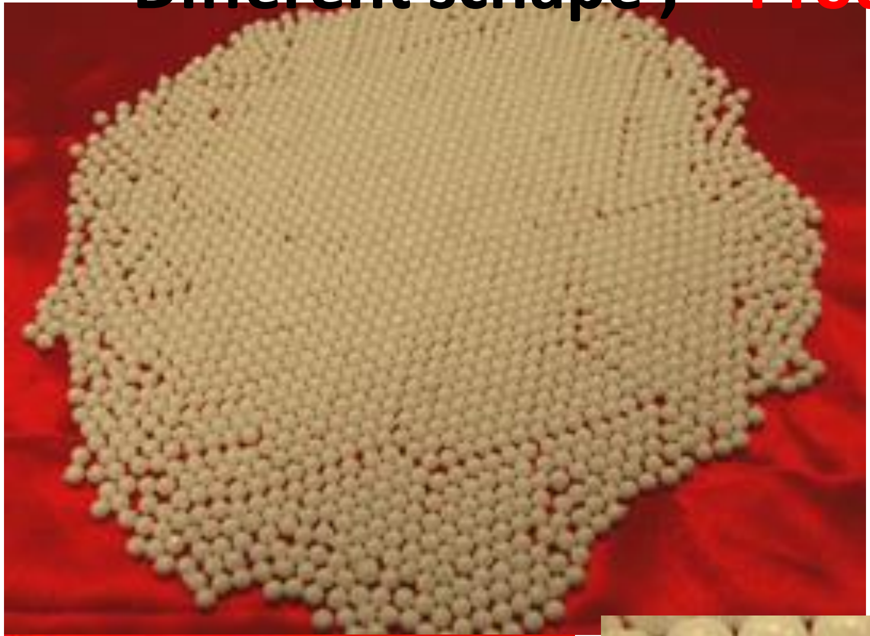


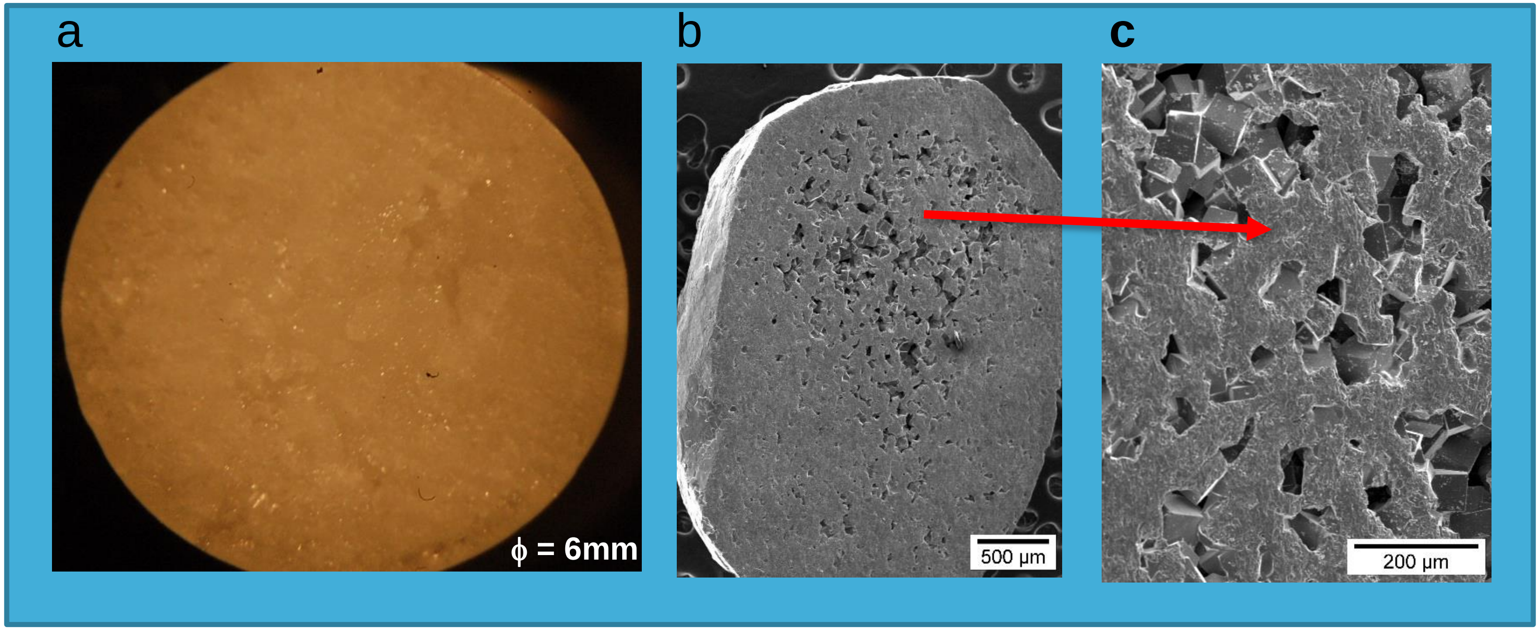




α-alumina developed in ILM Laboratory (99.9999%)

Different schape , Production 1Kg/hour



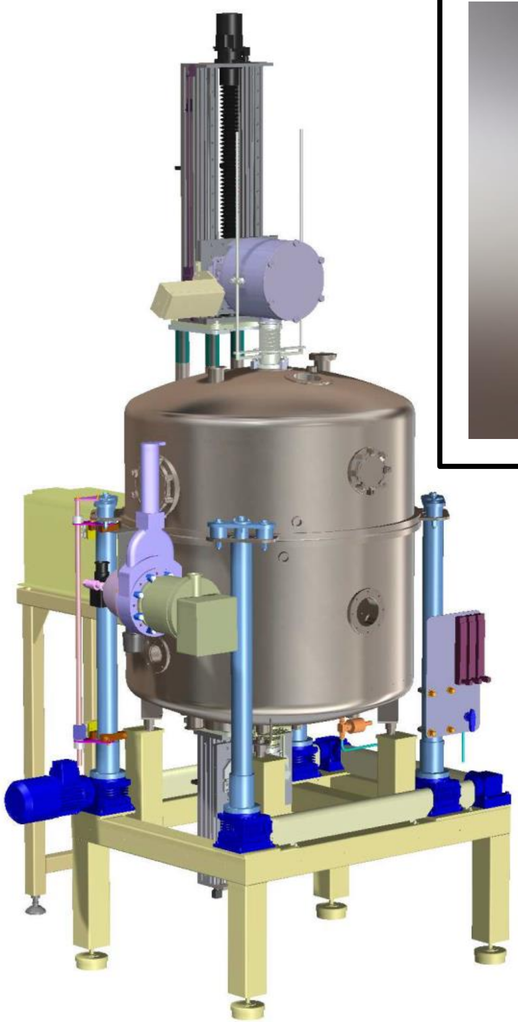


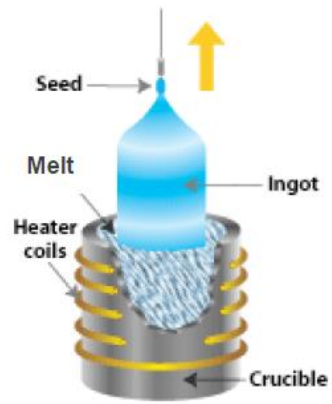
Morphology of ILM Alumina raw material

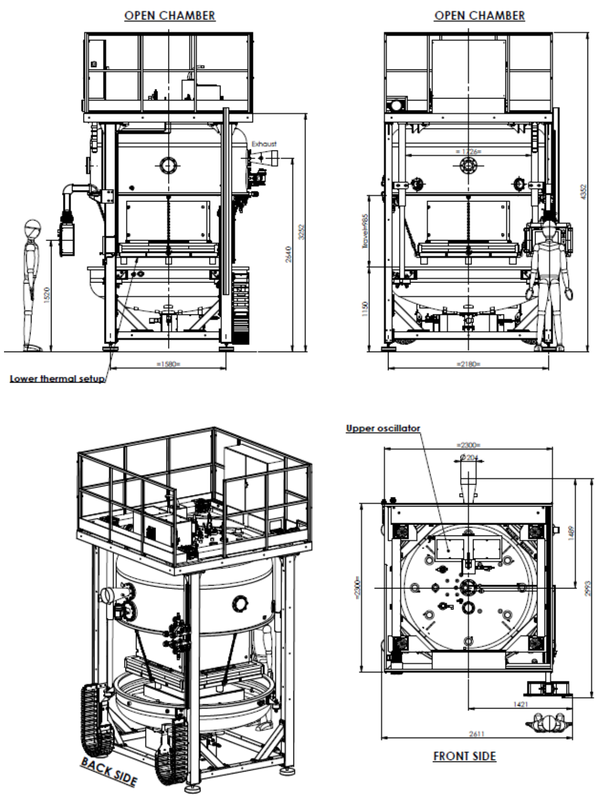
New growth Technology for sapphire mirror

Furnace design for large sapphire growing (diameter 500mm)

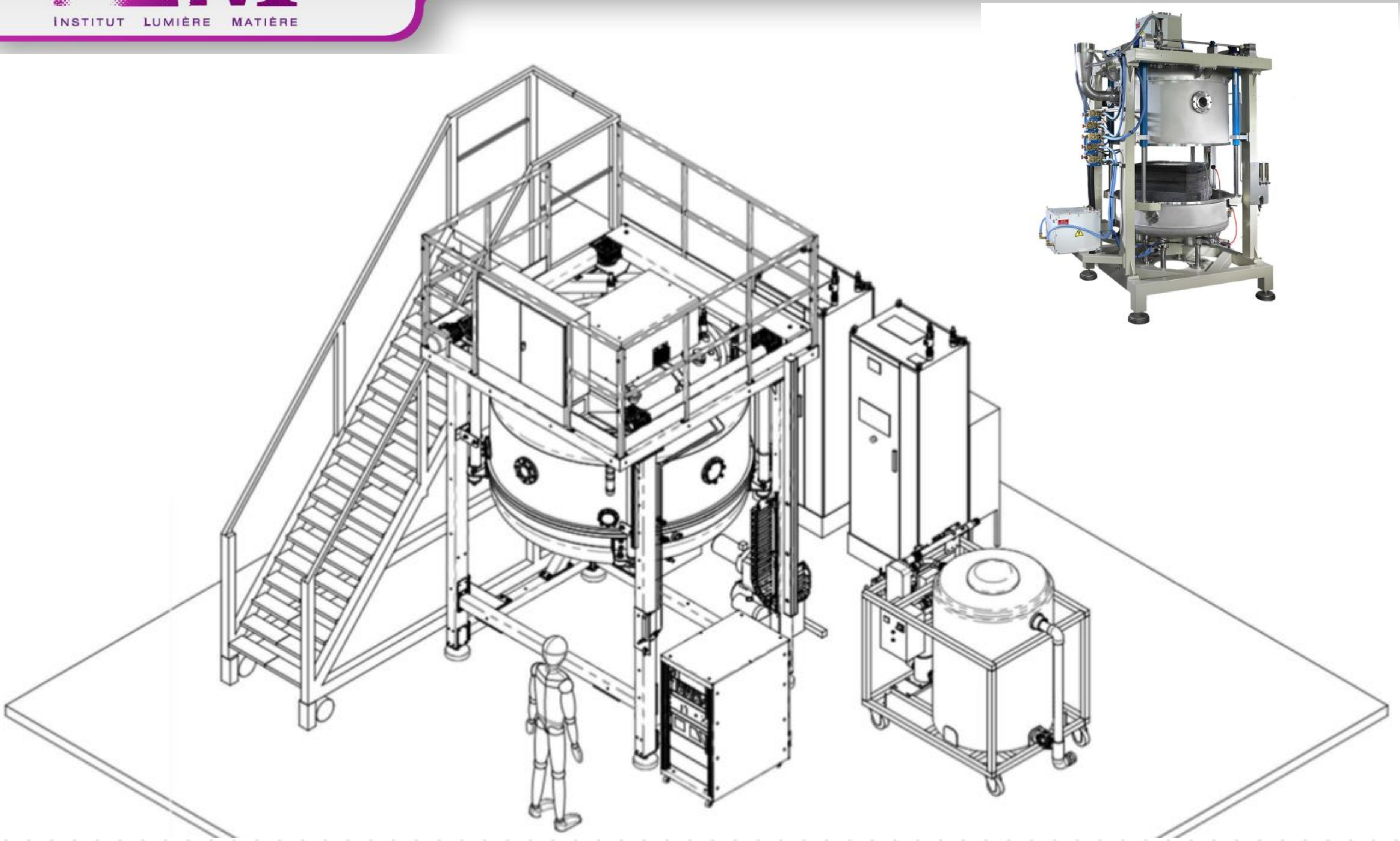
We will use RF and resistive heating at the same time!







Machine configuration

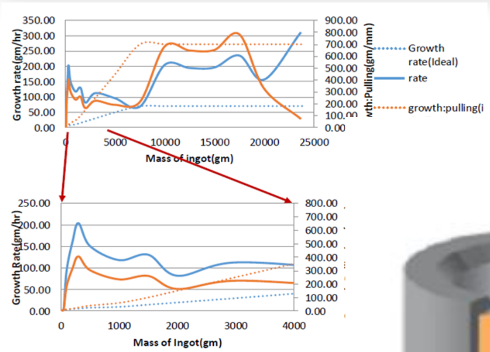


OSAG machine for large sapphire crystal growth

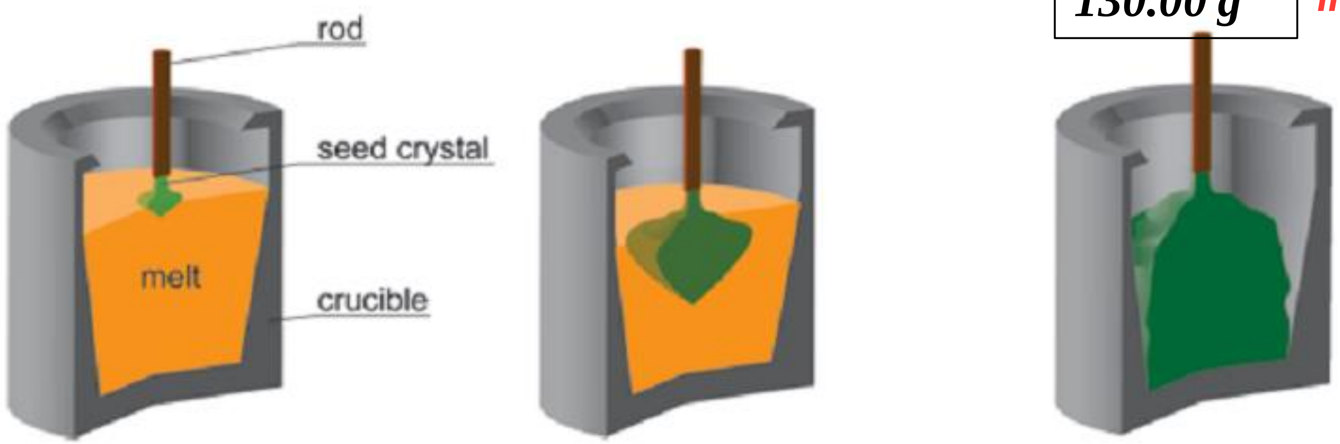
INSTITUT LUMIÈRE MATIÈRE

ILM technology for growing large sapphire ingot

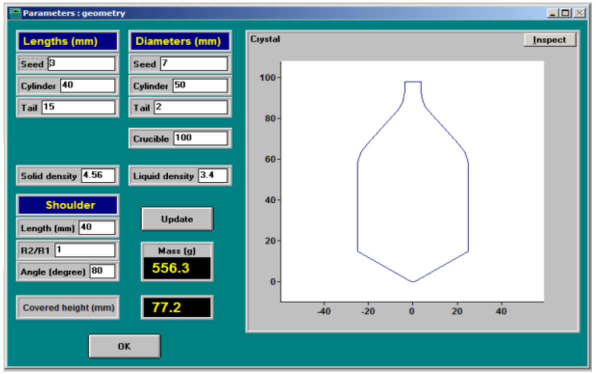
Compilation of two techniques (Cz+Ky)



150.0 kg	*m > 80 Kg*
130.00 g	*m < 80 Kg*



Czochralski + *Kyropoulos*



For controlling the sapphire ingot geometry

Automatized control ⬄ Knowing the level of the melt at any time during the process

In situ reconstruction of crystal shape grown in an axisymmetric Kyropoulos system

Gourav Sen[a], Wenqing Jia[b], Yoann Malier[a], Liliana Braescu[c], Thierry Duffar[a,*]

[a] Univ. Grenoble-Alpes, CNRS, Grenoble INP, SIMaP 38000 Grenoble, France
[b] Suzhou Nuclear Power Research Institute, Suzhou 215004, China
[c] Institut National de la Recherche Scientifique – Centre Énergie, Matériaux et Télécommunications, 1650 Boulevard Lionel-Boulet, Varennes, J3X 1S2, Québec, Canada

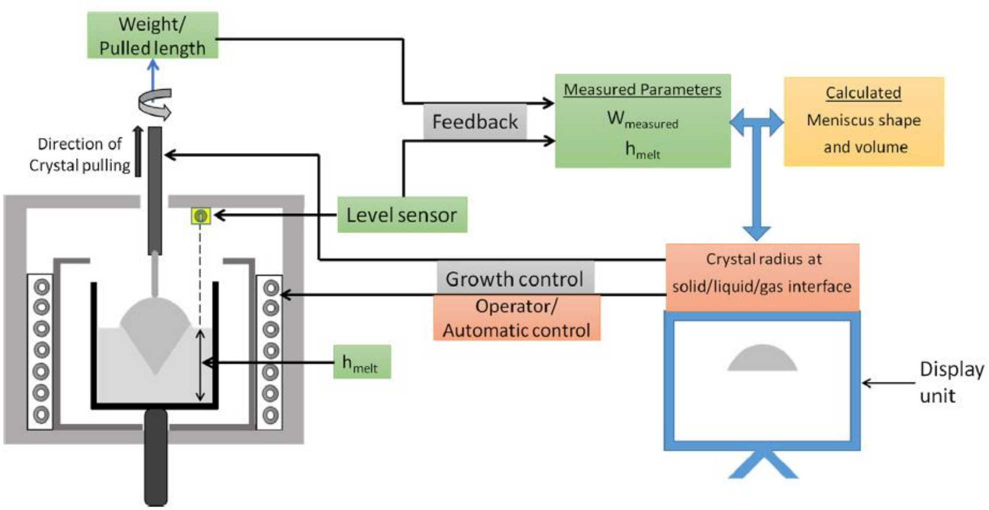
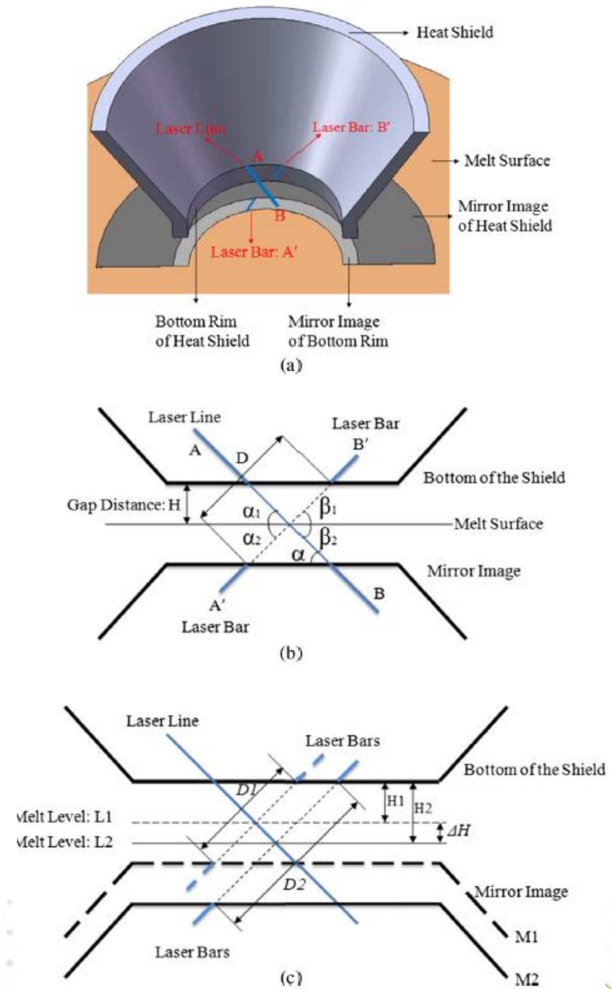


Fig. 1. Scheme for crystal shape monitoring system with melt level sensor.

Melt level measurement for the CZ crystal growth using an improved laser triangulation system

Senwei Xiang[a,*], Feng Pan[b], Ke Xiang[a], Xuanyin Wang[a]

[a] Zhejiang University, State Key Laboratory of Fluid Power and Mechatronic Systems, Mechanical Engineering, 38 Zheda Road, Hangzhou 310027, China
[b] Zhejiang Sunny Optical Intelligence Technology Co., Ltd., 525 Xixi Road, Hangzhou 310027, China



3- Conclusion

- Big challenge to develop a new technology to grow performed inexistent sapphire crystals for GW

- Development of sapphire suspension (fibers, ruban)

- Tools development to prepare (cut, polishing) large sapphire crystals

- Characterize and understand the different complex phenomenon involved during sapphire crystals growth

- Optical characterization through different techniques (spectroscopy, TEM, mechanic…) to reach OSAG target

Thank you for your attention