Croissance et caractérisation de cristaux de saphir dopés Ti pour lasers de puissance

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José Baruchel, Thu Nhi Tran Caliste

Nicolas Barthalay

TITANSAPHIR Project

RSA Le Rubis SA

RSA, a French company, is one of the world largest producer of synthetic sapphire, ruby and spinels.

TITANSAPHIR Project

Growth of Titanium-doped Sapphire single crystals for power laser applications.

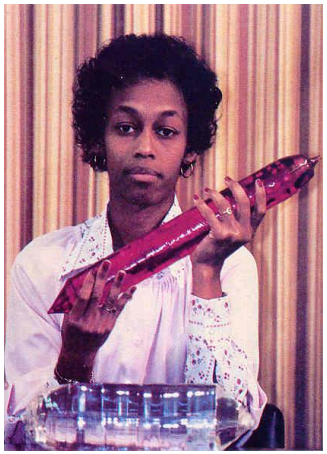
*Find **growth process improvements***

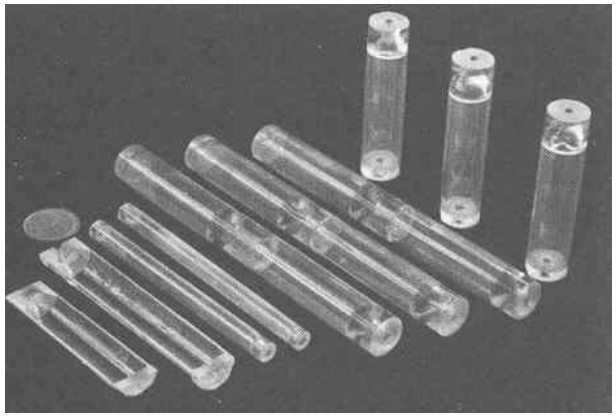
***Characterization** of the defects inside the crystals*

Industrial partners			*Research Labs*	
RSA	Cyberstar	Amplitude TECHNOLOGIES	SIMaP	iLM INSTITUT LUMIÈRE MATIÈRE
Crystal Growth	*Growth Furnace*	*Laser Optics*	*Defect Characterisation & Growth process*	*Optical Characterisation*

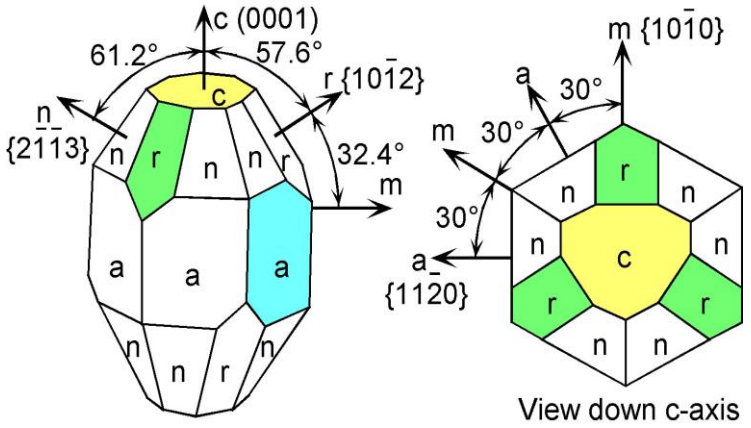
Sapphire: Al_2O_3

Natural sapphire





Artificial grown sapphire



Melting point: 2050 ºC

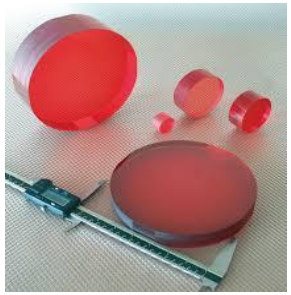
Mohs hardness: 9

Kyropoulos Crystal Growth

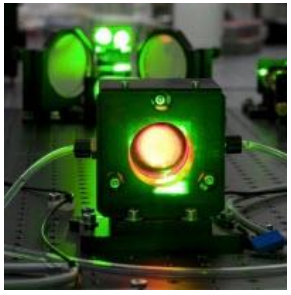
Industrial furnace

Kyropoulos growth technique advantages

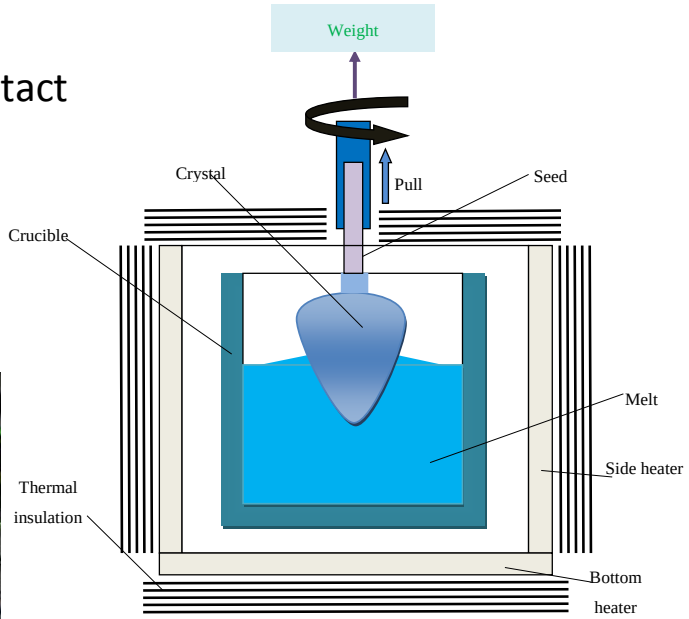
- Possibility to grow crystals with large diameter (≥ 30 cm).
- Growth inside melt and without contact with crucible.
- Low dislocation density.



Sapphire disks



High power Lasers



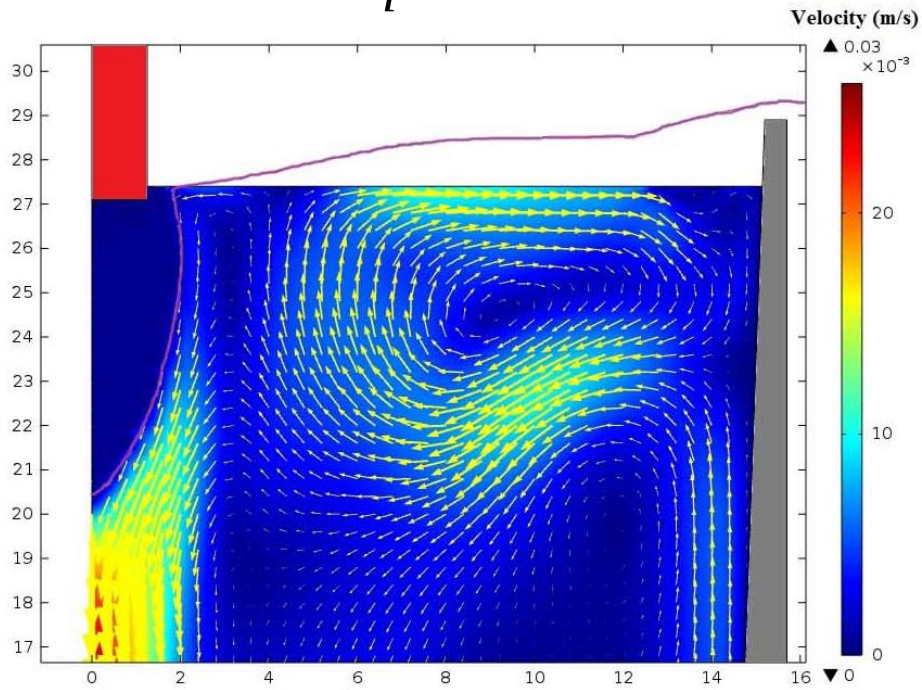
Crystal Morphology

20 cm

Plate formation at crystal head

$$t_i = 0.15\,h$$

Crucible diameter 30 cm



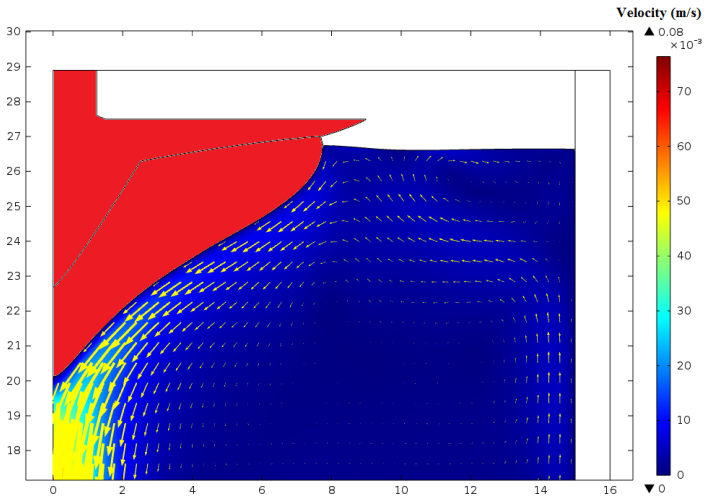
Global modeling of thermal and velocity field
(COMSOL)

Plate formation at crystal head

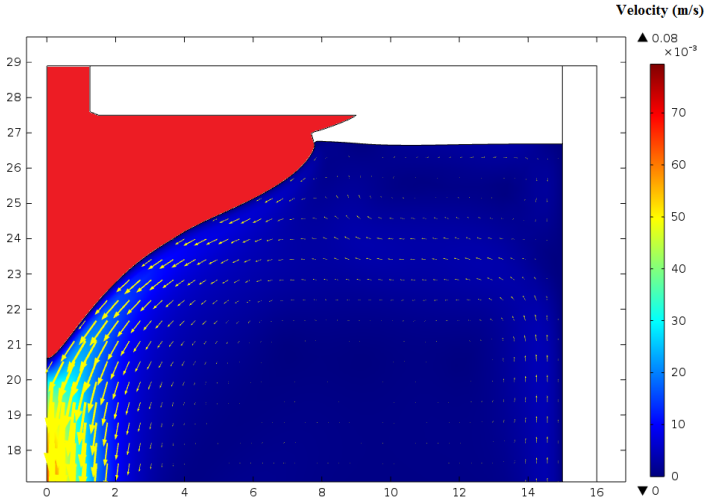
Temporal concave shape of the crystal

Effect of the Marangoni convection

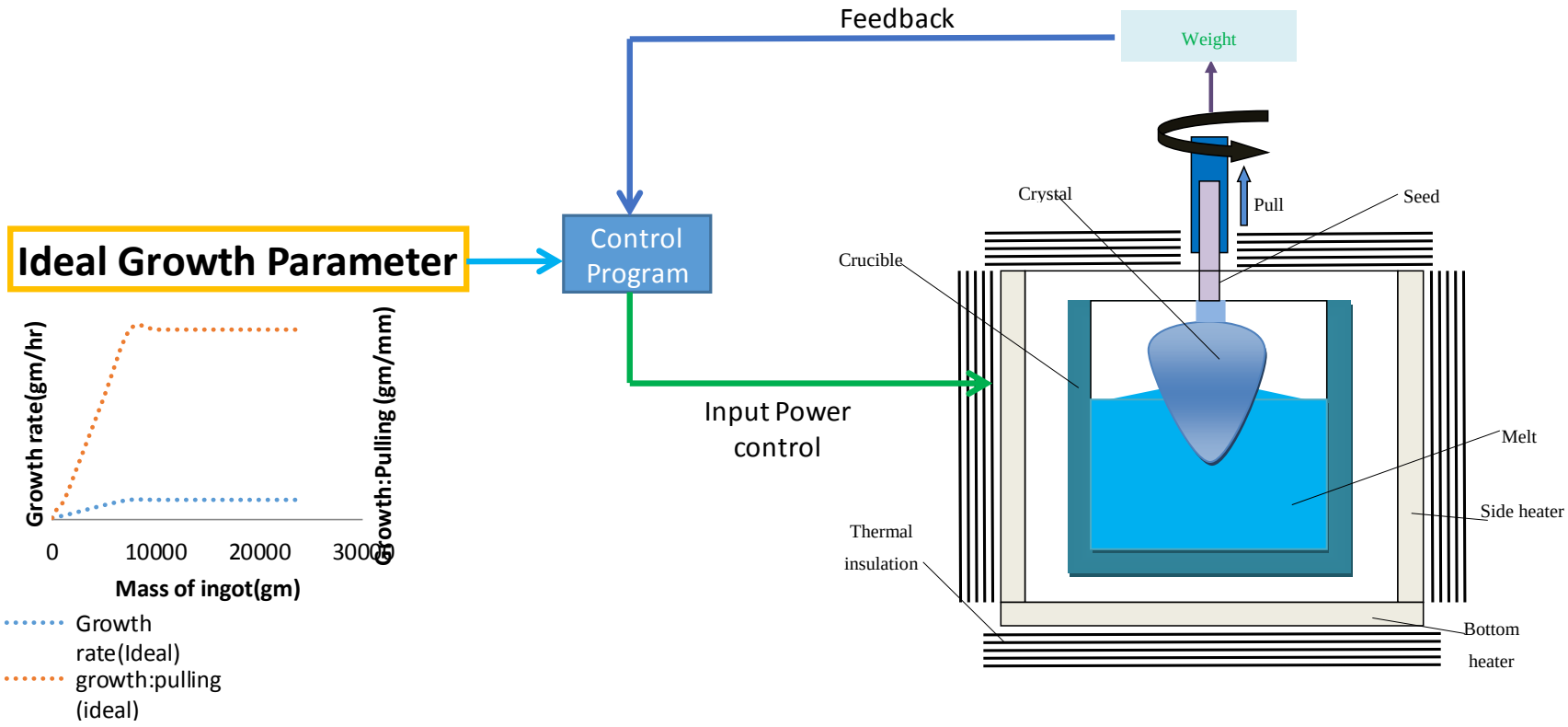
Marangoni



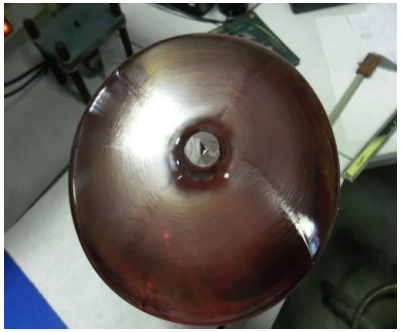
No Marangoni



Growth control system



Non-uniform shape of crystal



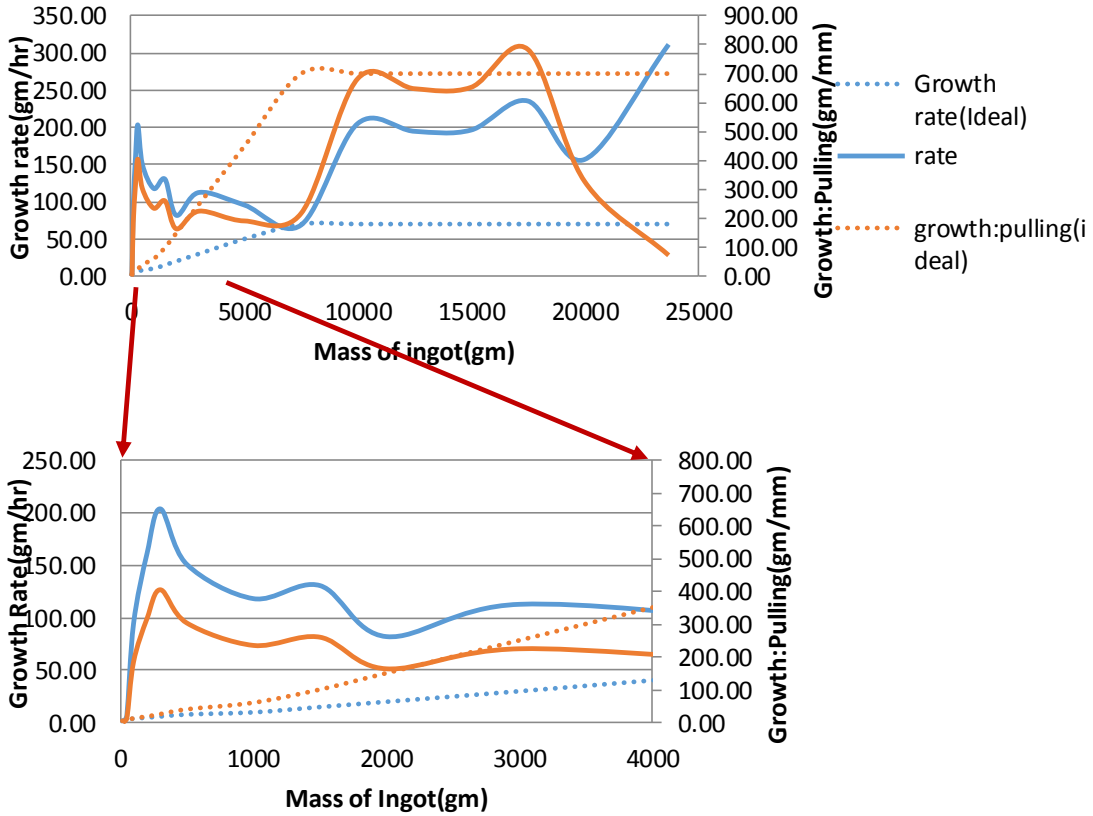
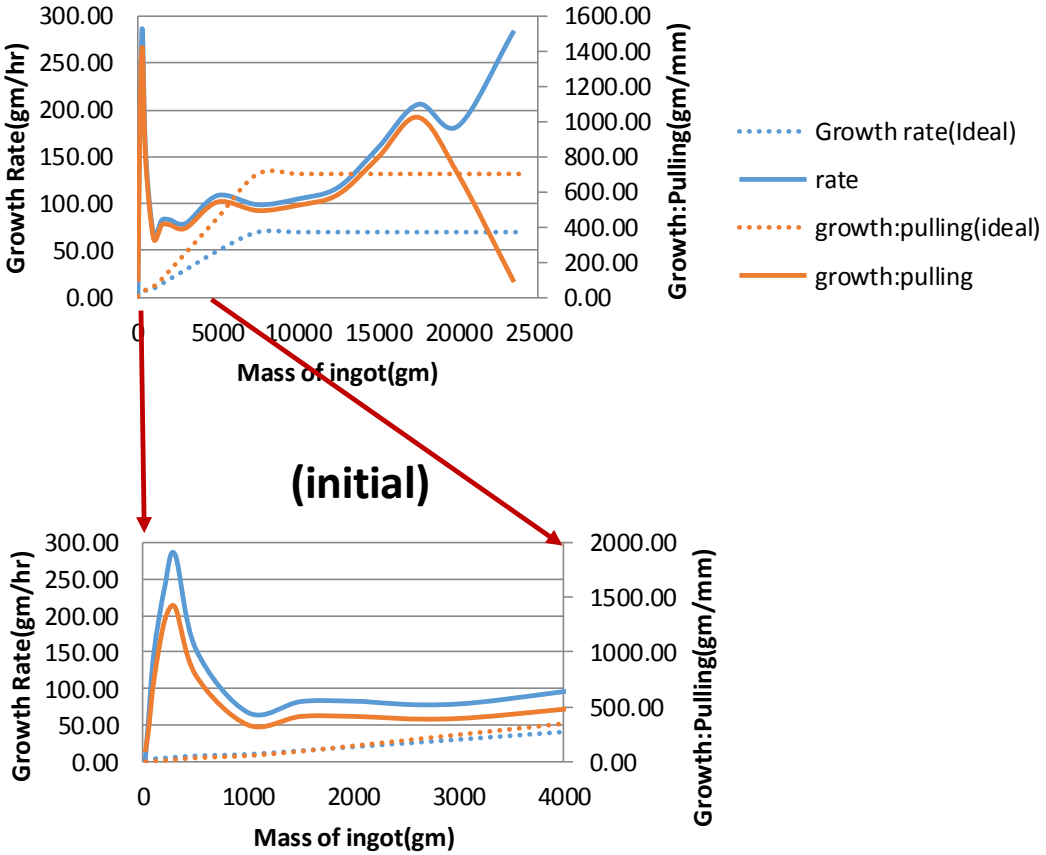
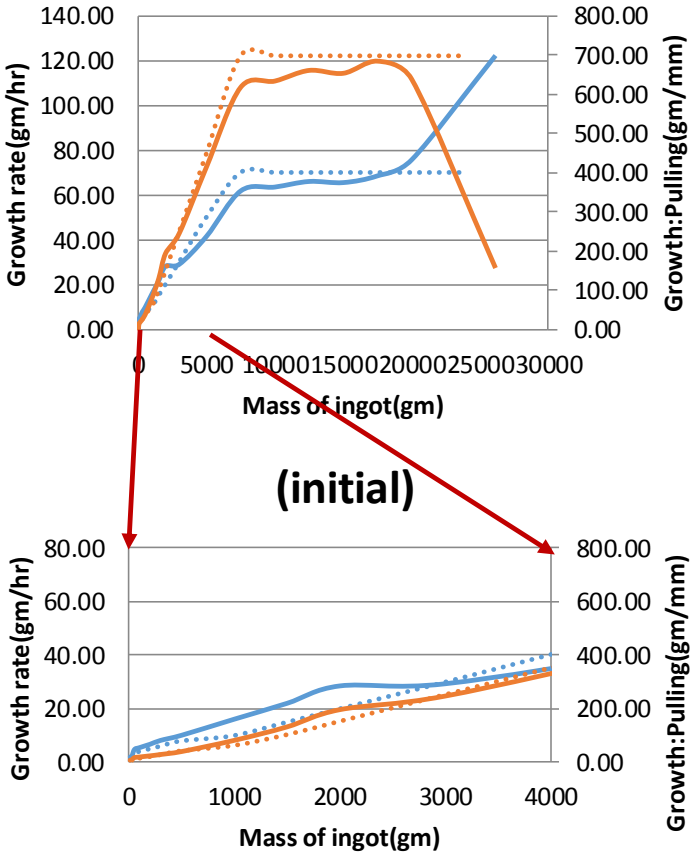


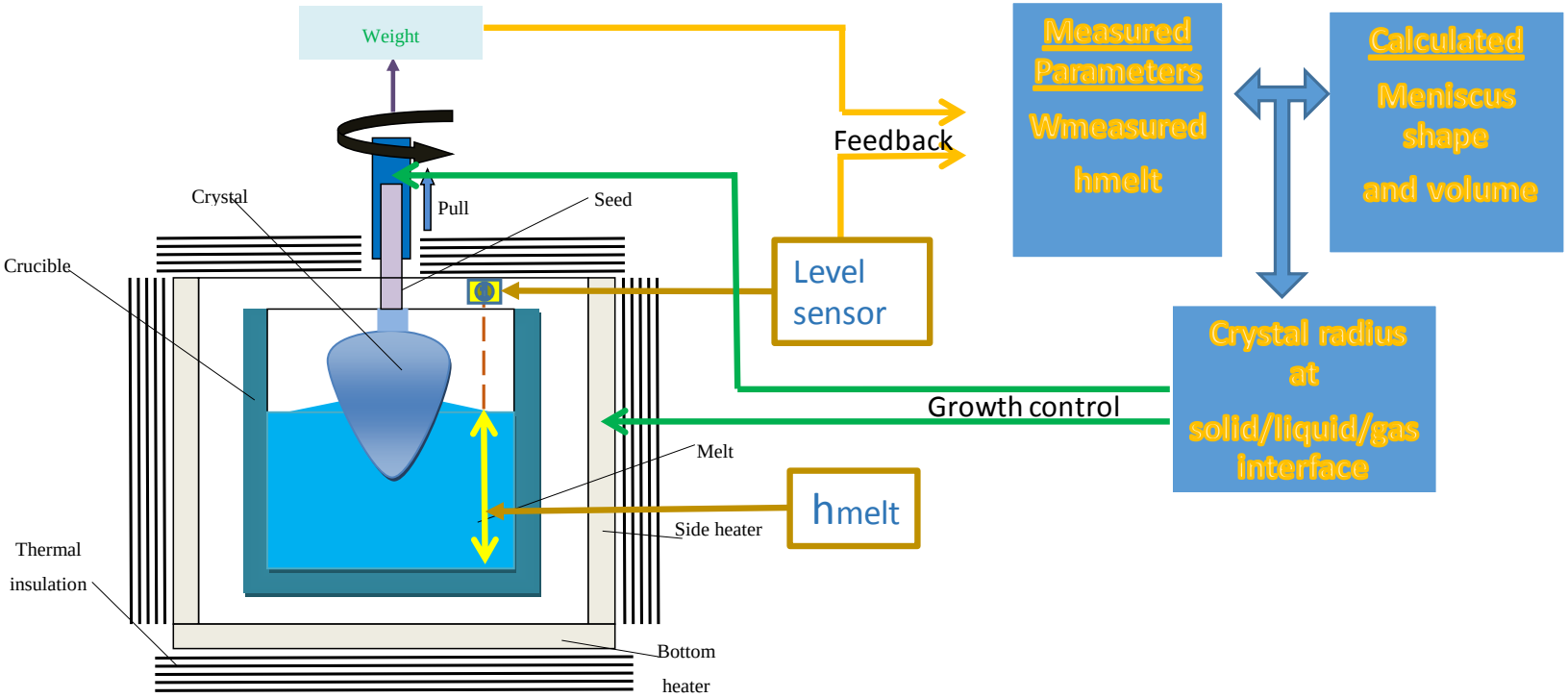
Plate formation in crystal



Good crystal morphology

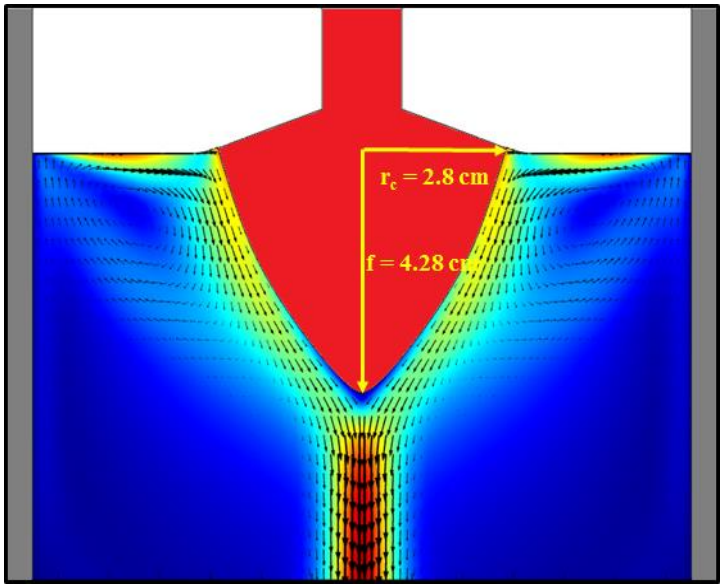


Automatic crystal radius control



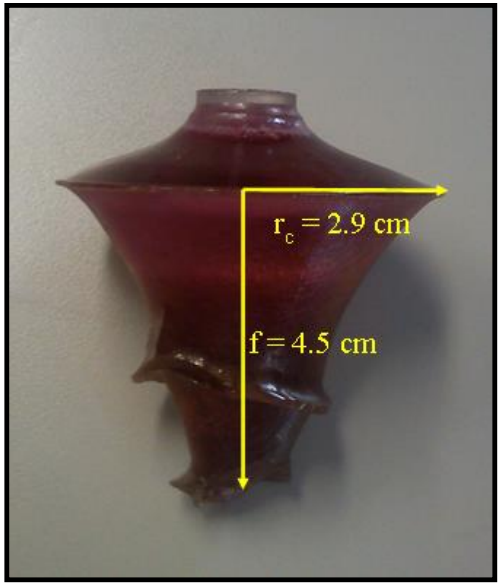
Numerical Modelling

Numerical modeling



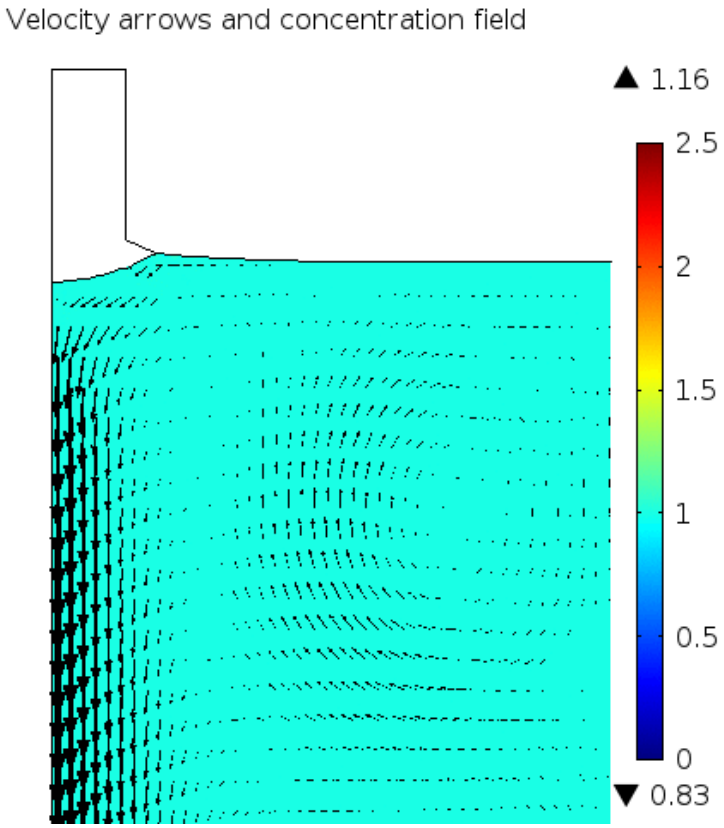
$$\tau = 4.28\text{cm} / 2.8\text{cm} = 1.53$$

Experimental visualization of the growth interface

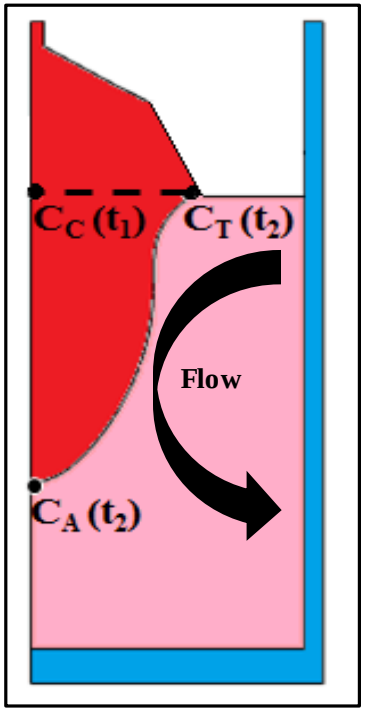


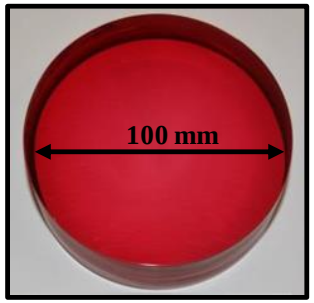
$$\tau = 4.5\text{cm} / 2.9\text{cm} = 1.55$$

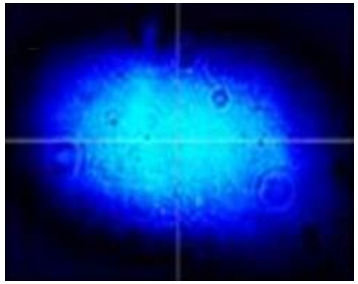
Numerical modelling
Ti distribution

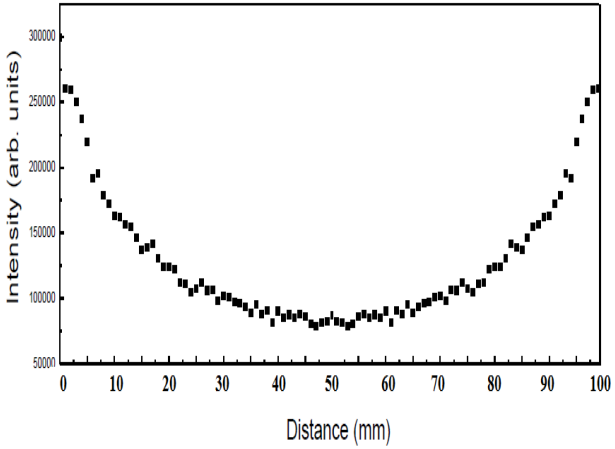


Radial distribution of titanium



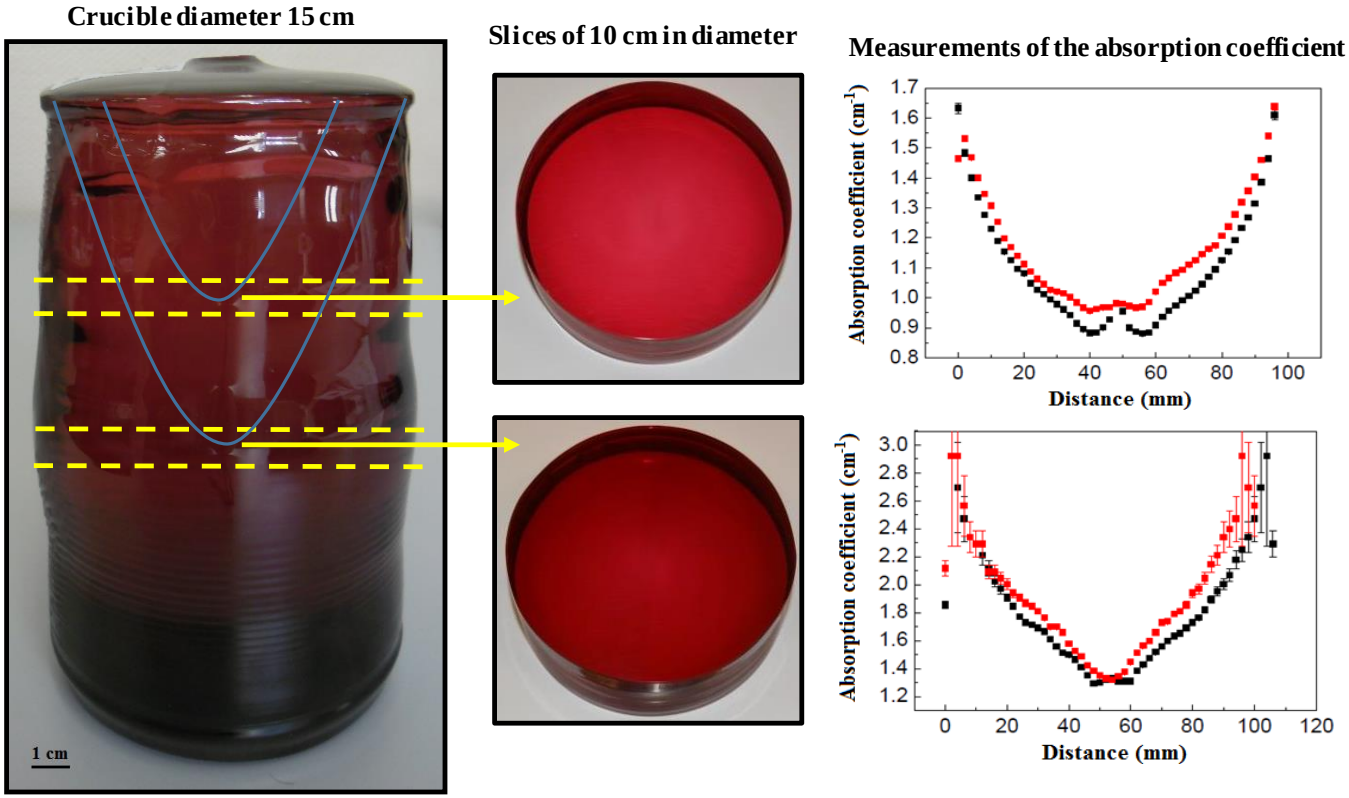






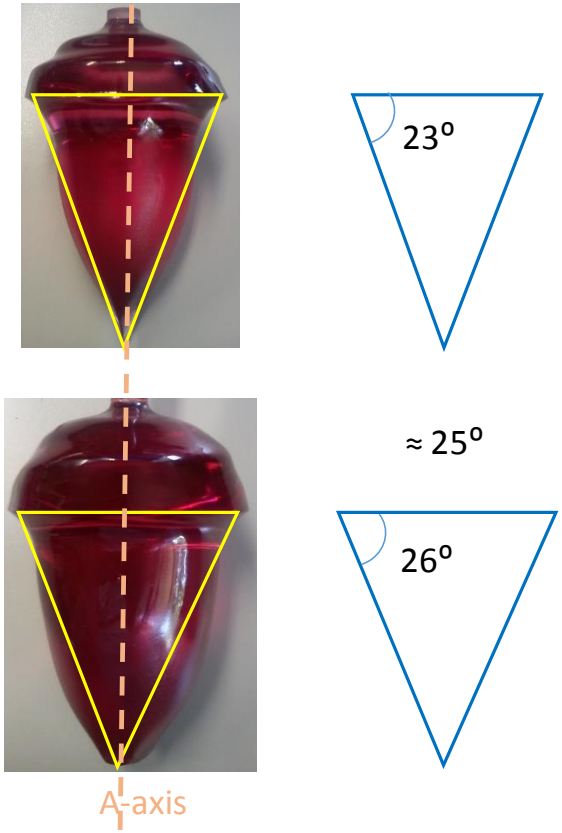
- Large curvatures of the crystal-melt interface
- Radial compositional non-homogeneity
- Variations in the intensity of the emitted laser beam

Radial distribution of titanium

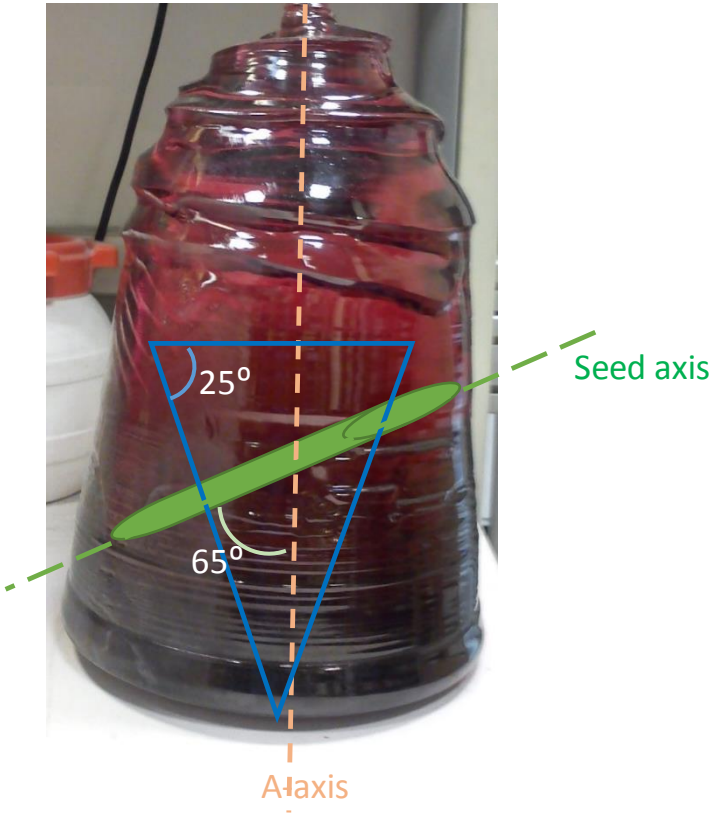


C. Stelian, G. Alombert-Goget, G. Sen, N. Barthalay, K. Lebbou, T. Duffar, Analysis of titanium distribution in sapphire crystals grown by the Kyropoulos method, article in preparation

New approach to preparation of seeds

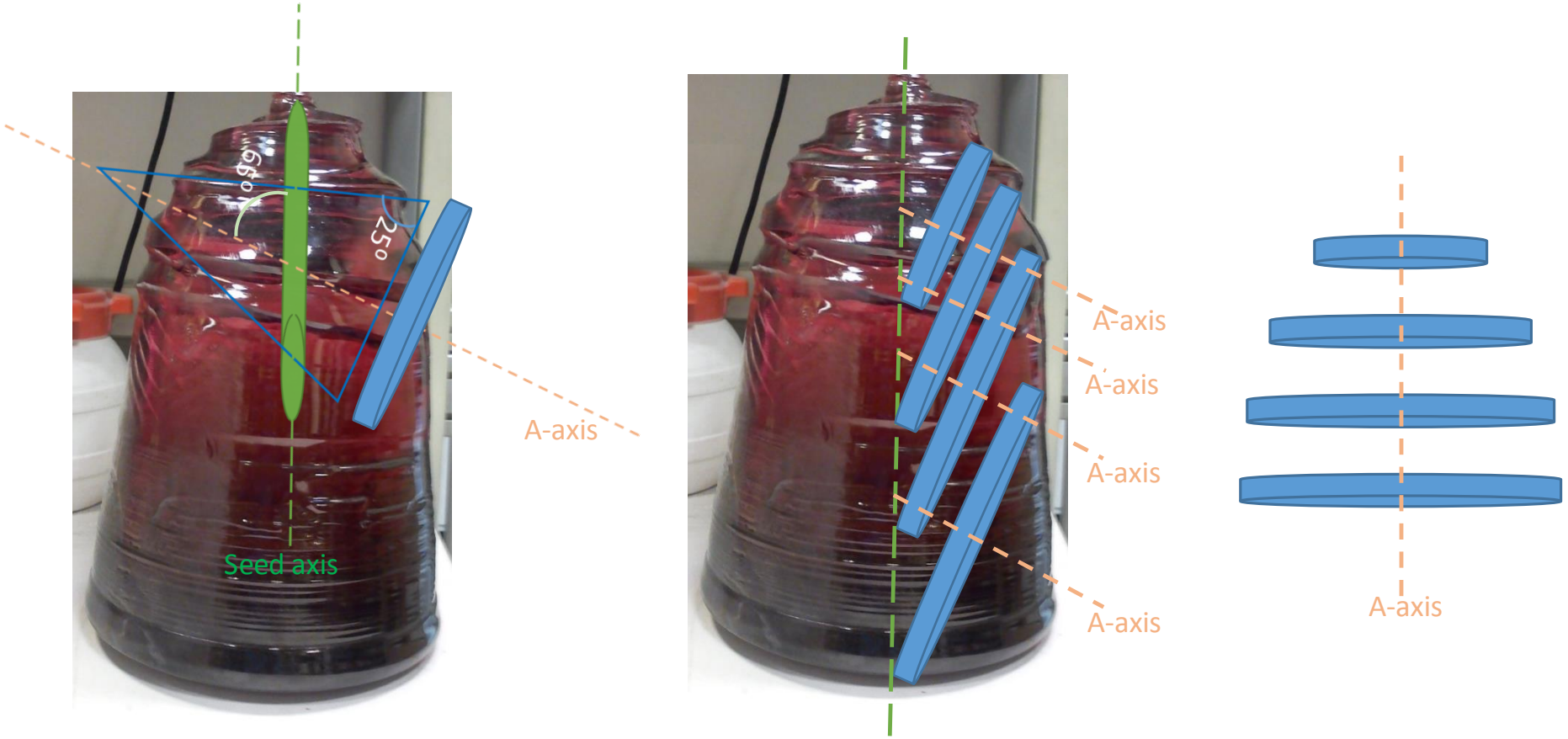


Solid-liquid crystal interface realised from previous pulling. (Interface angle: approx 25°)



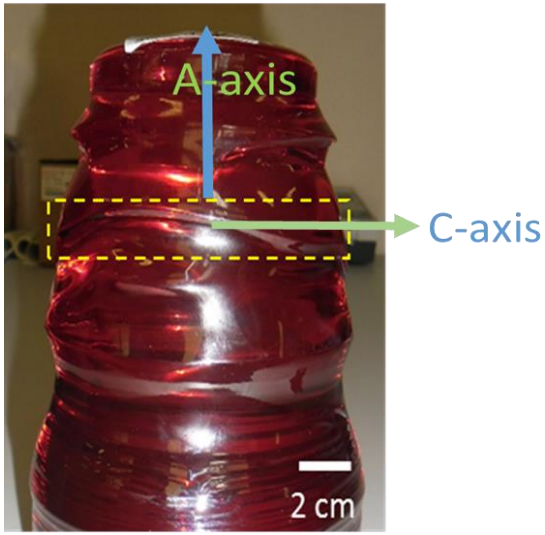
Prepare seeds from existing crystals with an axis offset of 65° with A-axis

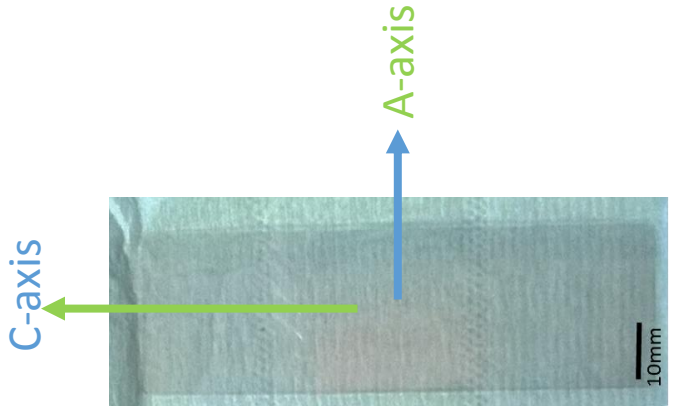
New approach to cutting Discs



Crystal grown with the offset seed axis will lead to crystals having the A-plane parallel to the solid-liquid growth interface. Thus A-Plane alligned discs can be cut out of various sizes parallel to the interface shape.

Sapphire crystal & sample





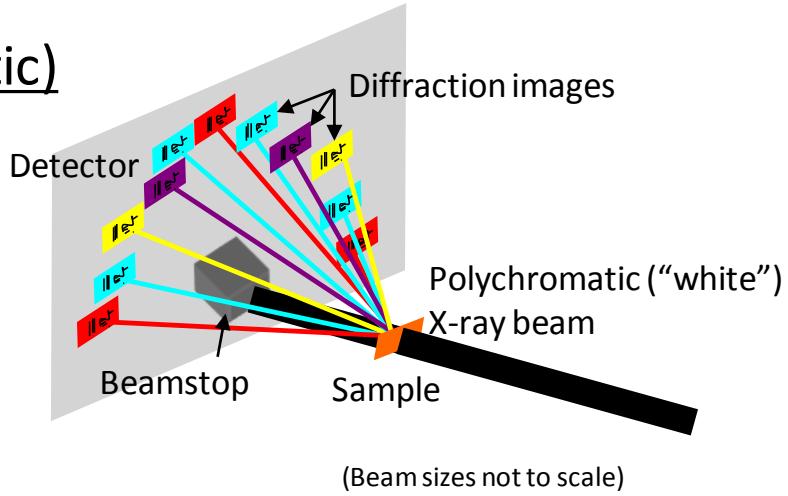
- 6 kg in weight
- Diameter – 10 cm
- Height – 16 cm
- Duration of growth – 6 days

X-ray diffraction imaging (x-ray topography)

- Bragg diffraction
- Image defects in the crystal lattice via the deformation around them ➔ diffracted intensity/direction altered ➔ *contrast*
  - White beam (polychromatic)
  - Section topography

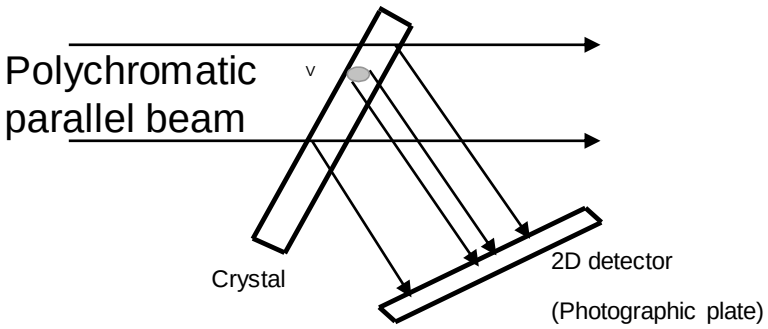
Beamline: BM – 05

ESRF
The European Synchrotron

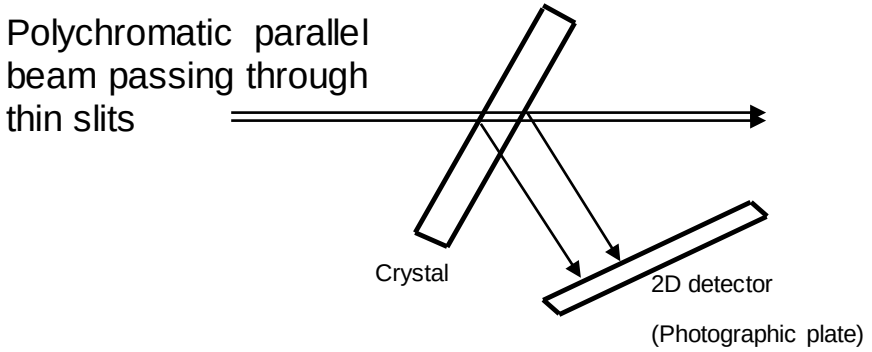


(Beam sizes not to scale)

Projection & Section topography



Projection

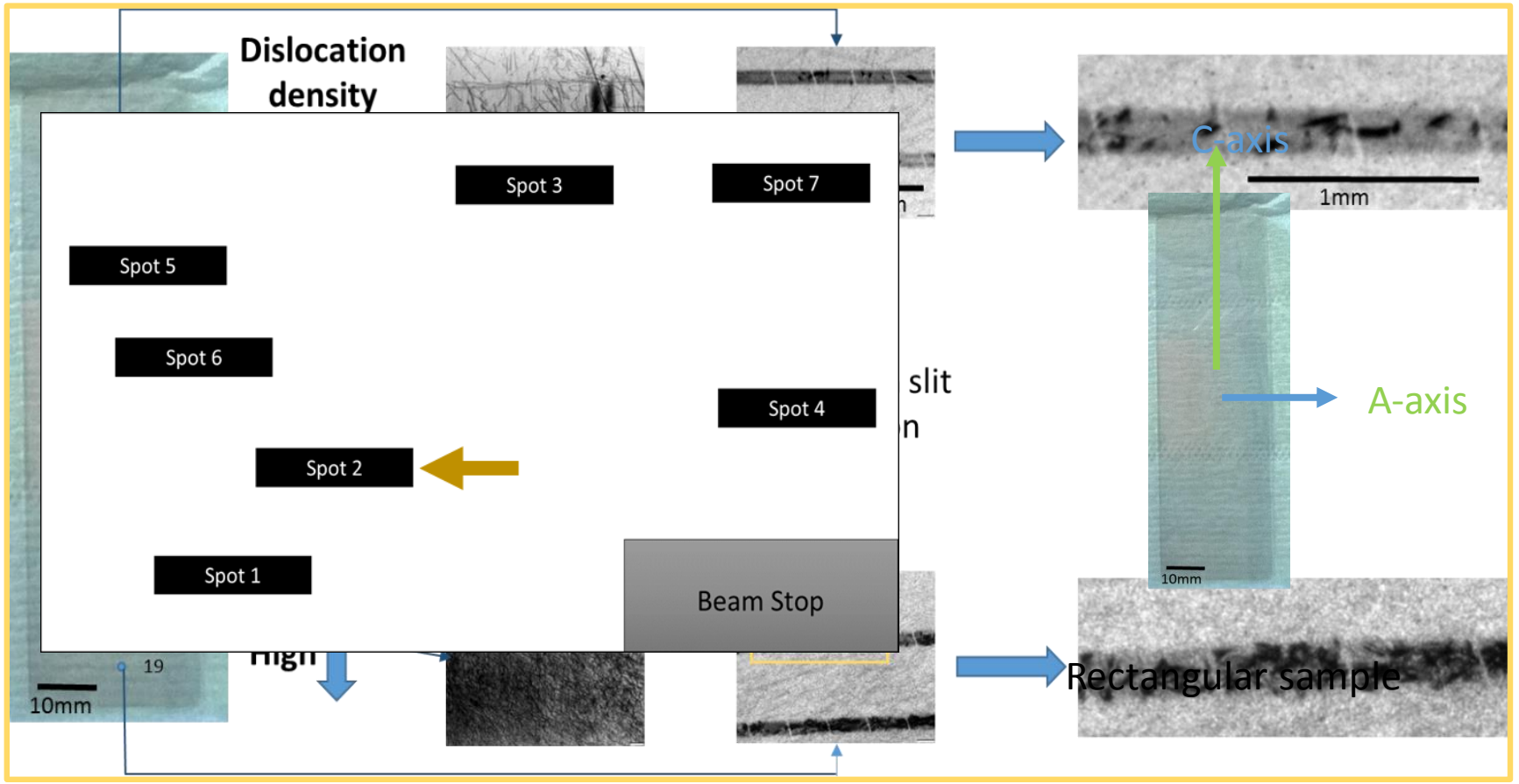


Section (slit configuration)

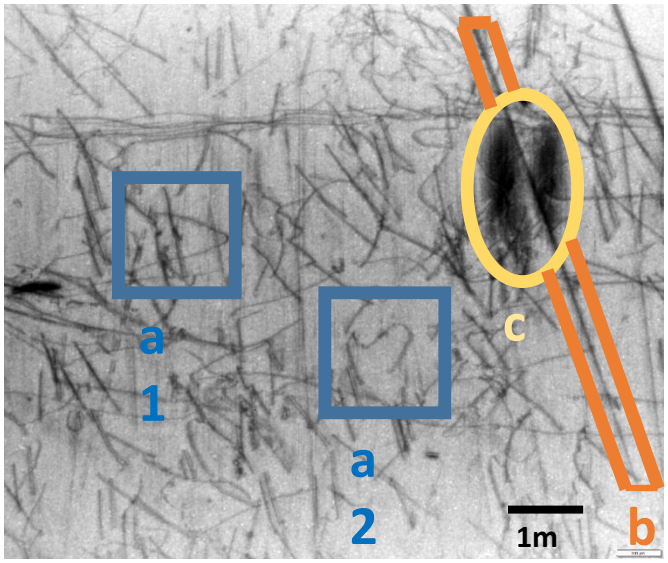
Good lateral spatial resolution, but poor depth resolution

Reveals the features within the depth of the sample

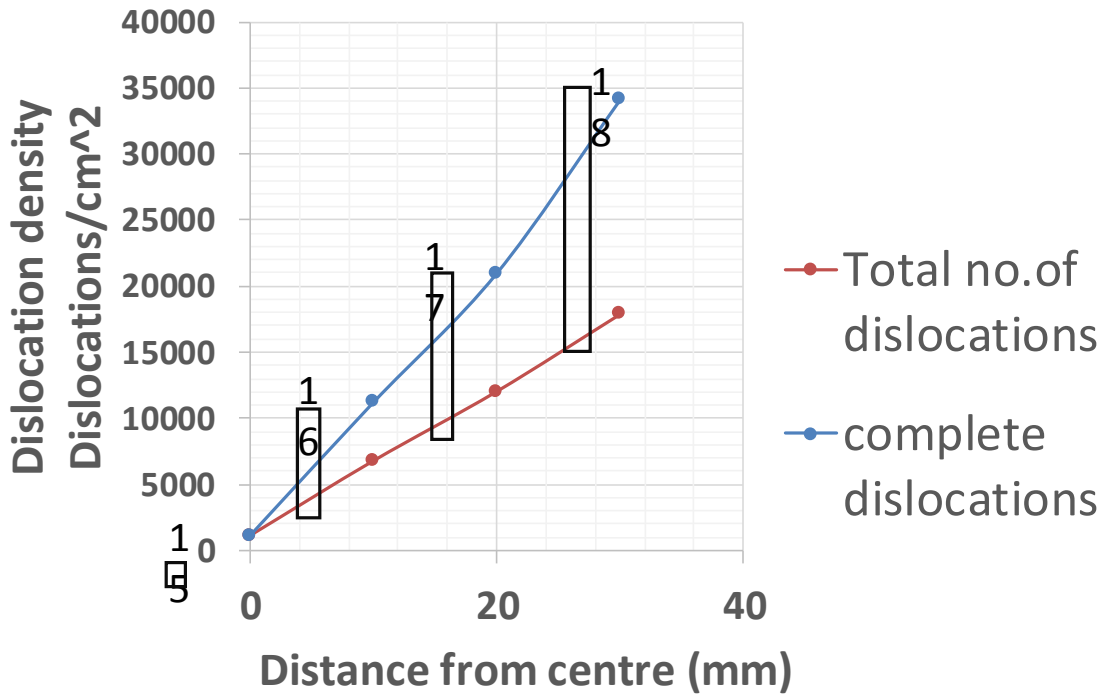
Diffraction spots



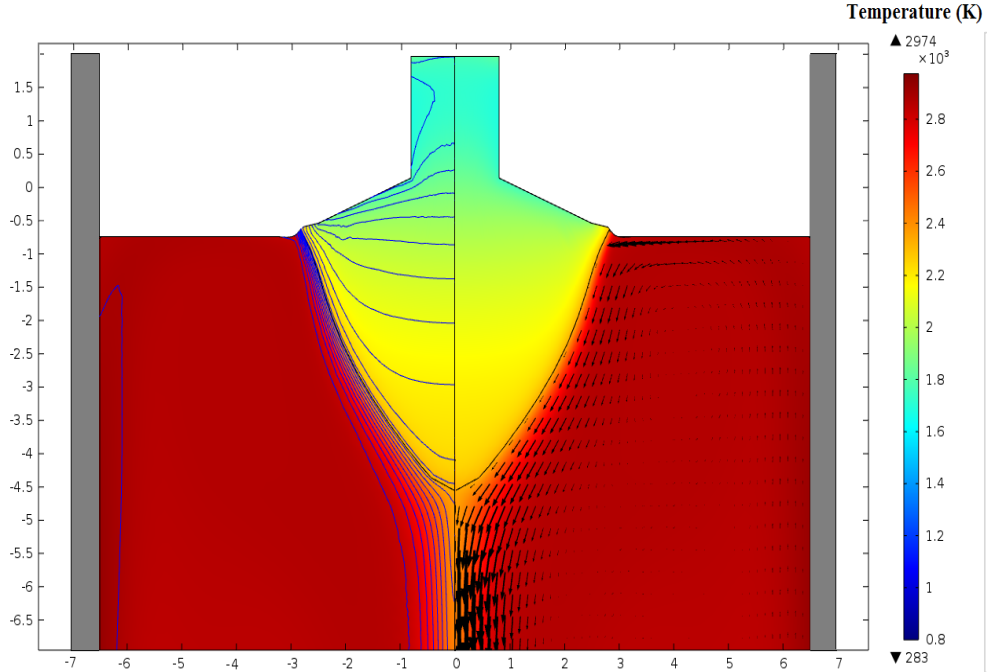
Dislocation density (Quantitative estimation)

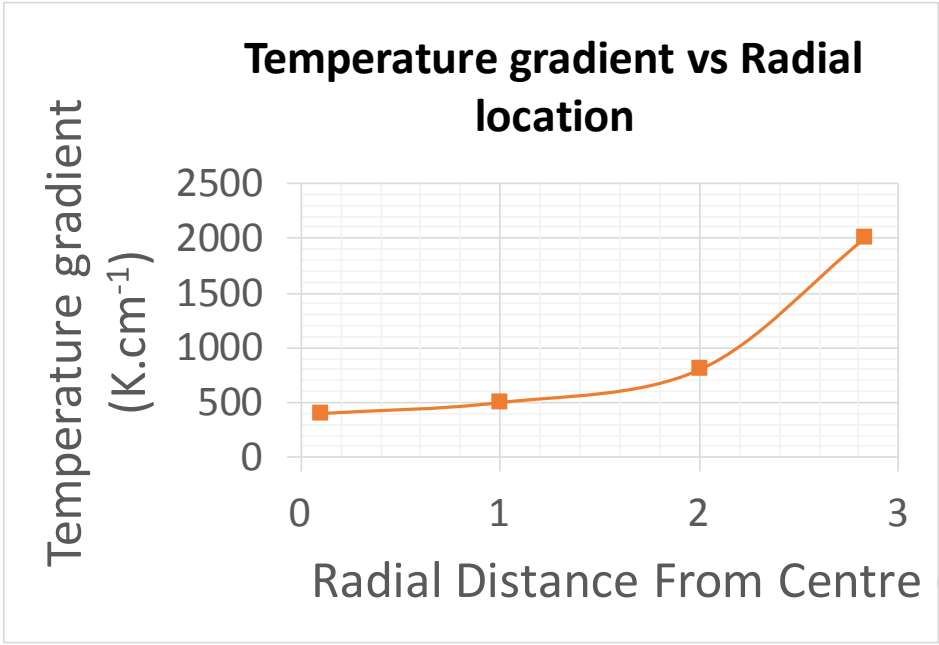


a1 and a2 – dislocations,
b – scratch marks,
c – precipitate



Numerical modeling (COMSOL)





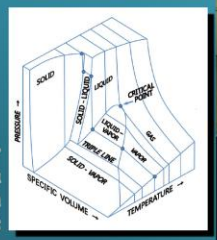
Conclusions

- Growth of 20cm diameter Al_2O_3:Ti crystals by Kyropoulos technique
- Top plate problem solved
- Full shape control under progress
- Radial Ti segregation solution
- Dislocations are the main type of defects,. 10^3-10^4 cm^{-2}
- good quality which is suitable for optical applications

Réseau National de la MI-CNRS
Cristaux, Micro-nanostructures
et Dispositifs pour l'Optique

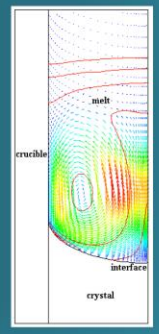
ACTION NATIONALE DE FORMATION

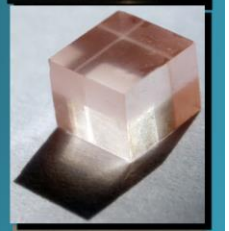
"Thermodynamique (d'équilibre et des processus irréversibles) appliquée à la cristallogenèse par solidification à haute température"



08-09 Décembre 2016
ICMCB PESSAC (Aquitaine)

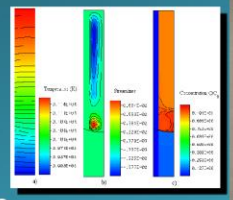
Formation théorique et pratique en thermodynamique pour la cristallogenèse : description thermodynamique de la phase, diagrammes d'équilibre de phases (avec extension au cas légèrement hors équilibre), méthodes calorimétriques/spectrométriques, utilisation des tables thermodynamiques, méthode Calphad (exemples d'application), diagrammes de Wulff et thermodynamique des défauts ponctuels, modélisation et simulation de procédé (démonstrations sur machine)





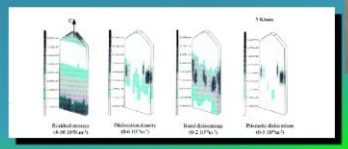
Organisation

- *2 journées de cours et travaux dirigés/pratiques*
- *Visite des services d'analyse thermique et de cristallogenèse de l'ICMCB*



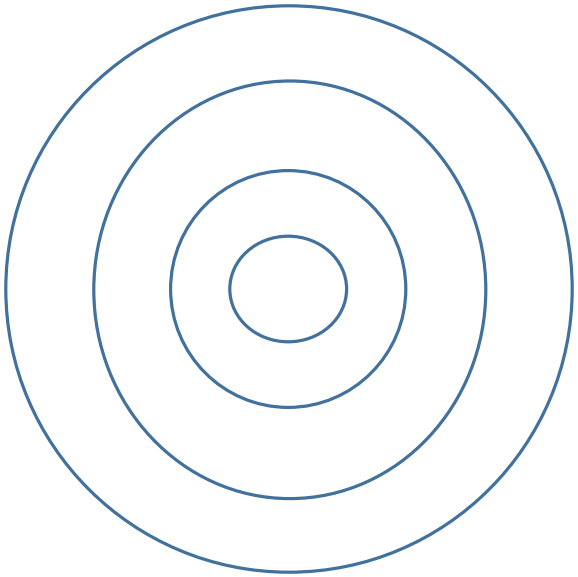
Tarifs

- *Gratuit pour tous les agents pris en charge par la formation continue du CNRS (dans la limite des places disponibles)*
- *125 € pour les privés et les agents non CNRS*



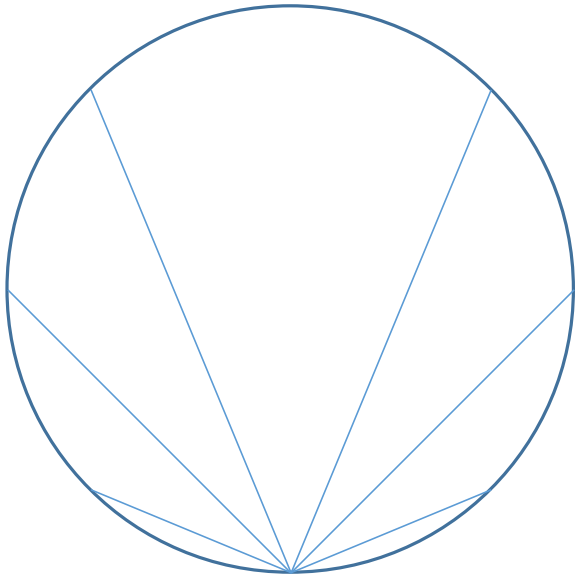
RENSEIGNEMENTS ADMINISTRATIFS : Marie-Noëlle Besson, formation-permanente@dr15.cnrs.fr, Tél. : 05 57 35 58 29
INSCRIPTIONS : https://services.aquitaine.cnrs.fr/limesurvey/index.php/415557/lang-fr
Les demandes d'inscription dûment remplies et signées sont à retourner à l'adresse suivante :
CNRS - Délégation Aquitaine – Formation Permanente, Esplanade des arts et métiers – 33400 Talence ou par mail à formation-permanente@dr15.cnrs.fr
PLAN D'ACCÈS À L'ICMCB : http://www.icmcb-bordeaux.cnrs.fr/spip.php?article25

Change in Ti segregation pattern



Current Segregation profile

We have a concentric profile of segregation owing to the conical interface shape. The concentric profile is very detrimental for the optical properties.



Improved Segregation profile

Though we will still have segregation the gradient will be much reduced. Also we get rid of the concentric gradient profile and hence the centre is not distorted.

Diffraction spots (Circular Sample)