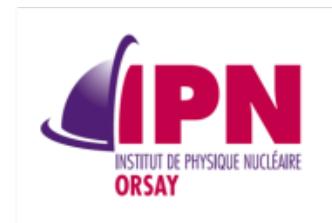


PANAMA

Plateforme d'ANalyse et de cAractérisation de
Matériaux pour les Accélérateurs

Technological Platform to Analyze and Characterize Materials for Accelerators

Sébastien Bousson, David Longuevergne



OUTLOOK

- ⇒ **History and Objectives**
- ⇒ **Scientific cases**
- ⇒ **Instruments selected**
- ⇒ **Future work**

HISTORY AND OBJECTIVES

Technological Platform to Analyze and Characterize Materials for Accelerators

- The idea of the PANAMA Platform is born from the general need within the P2IO laboratories of means to characterize material surfaces to address particle accelerators development issues:
 - ⇒ Thin films for accelerators: related projects: ECOMI (IPNO, IRFU)
 - ⇒ Surface treatment analysis: related project : PICASU (IPNO, IRFU)
 - ⇒ Target developments (stable and radioactive): IPNO
 - ⇒ Copper coating (power couplers): LAL, IRFU, IPNO
 - ⇒ NEG coating : LAL
 - ⇒ Anti-multipacting coating (power coupler): LAL, IRFU, IPNO
 - ⇒ SC magnets developments (IRFU)
 - ⇒ Calibration of coating equipment for thin films production: CSNSM

HISTORY AND OBJECTIVES

Technological Platform to Analyze and Characterize Materials for Accelerators

- **Creating a local network of outstanding instruments and expertise:**
 - ⇒ Integrate instruments already existing in the P2IO laboratories (mutualize)
 - ⇒ Acquire new instruments carefully chosen according to the following rules:
 - Relevant to address the P2IO labs needs
 - Technical performances
 - Easily accessible and ease of use
- Improve synergy among laboratories by creating a network of local experts
- Potentiality of use for radioactive sealed sources
- ... and obviously opening to other P2IO (and even larger) scientific applications

SCIENTIFIC CASES

Thin films for SRF Acceleration

R&D on superconducting materials for new RF acceleration scheme based on multilayer SC films for accelerating RF cavities

State of the art of bulk niobium: close to the theoretical limit

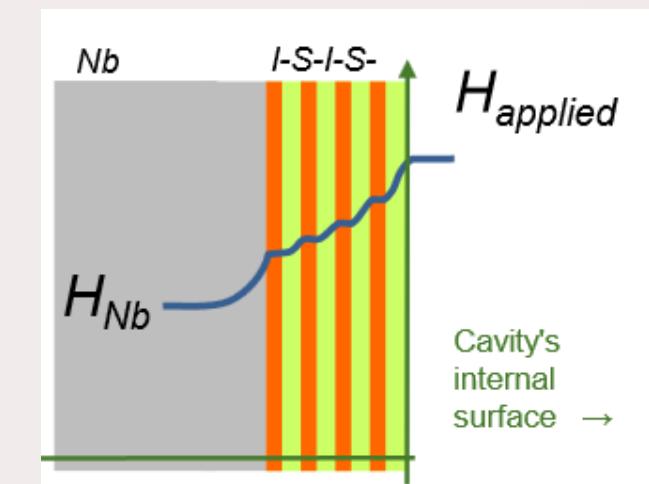
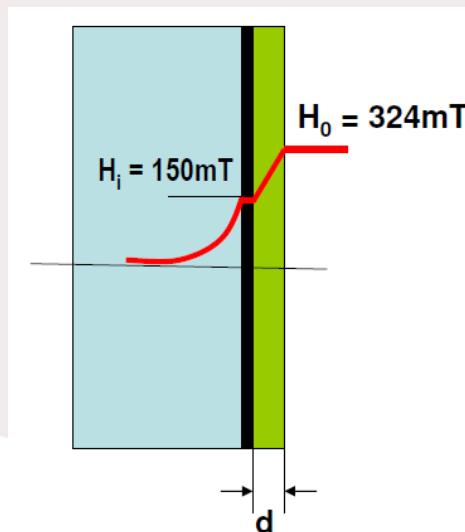


Potentiality of multilayer SC material:

- ⇒ Increase of the maximum magnetic field sustained by the bulk niobium in its SC state (screening effect)
- ⇒ Improvement of the surface resistance (decrease of RF losses)
- ⇒ Treatment of already existing cavities possible (coating)



ML2



Thin films for SRF Acceleration

R&D on superconducting materials for new RF acceleration scheme
based on the use of Nb₃N thin films for the superconducting cavity.

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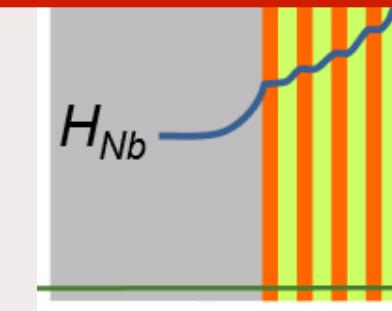
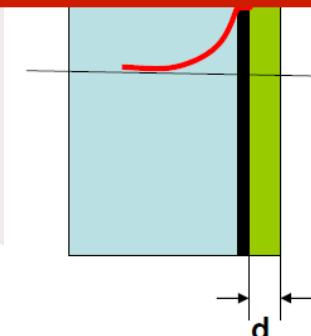
THE NEED:

Pote

- Analysis of surface roughness and quality -> confocal microscopy
- Thickness measurement (-> grazing incidence X-ray diffractometer/SIMS) et quality of deposited layers (-> SIMS).



ML2



Cavity's
internal
surface →

Mechanical etching of SC Cavities

Inovative method for SC cavity surface polishing

Potential benefit

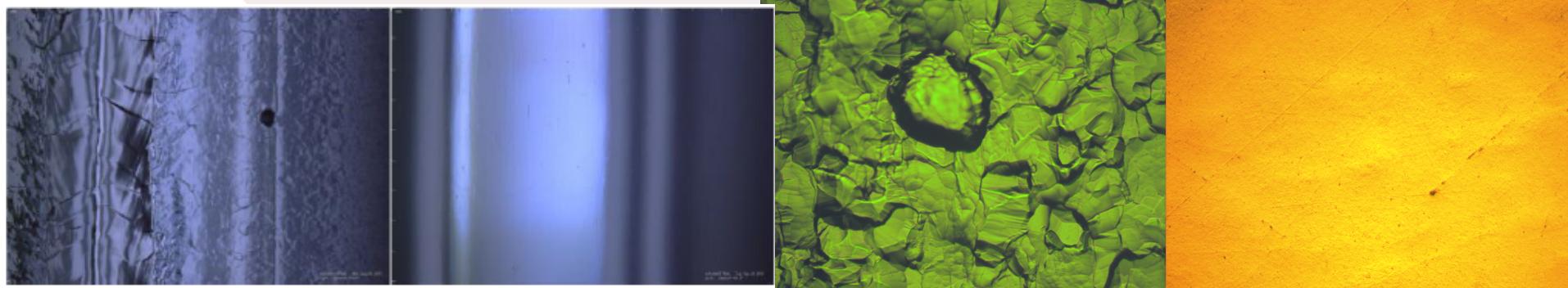
- + Improvement of surface state of SC material (as compared to chemical etching -> better SC behavior, improved cavity performances)
- + No more issues related to safety and recycling (eco-friendly) compared to chemical etching
- + Lower surface treatment costs
- Increase of treatment time, but full automation is possible and no monitoring required

* Study of crystallographic state / surface state link and its effect on superconductor properties



Chemically etched
Nb sample

Mechanically
etched Nb sample



Mechanical etching of SC Cavities

Inovative method for SC cavity surface polishing

Potential benefit

- + Improved surface quality
- + Reduced cost
- + Improved mechanical properties
- Increased time required for processing
- * Surface finish may vary depending on the material

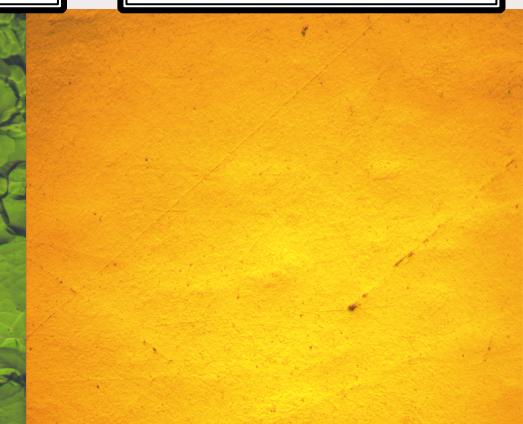
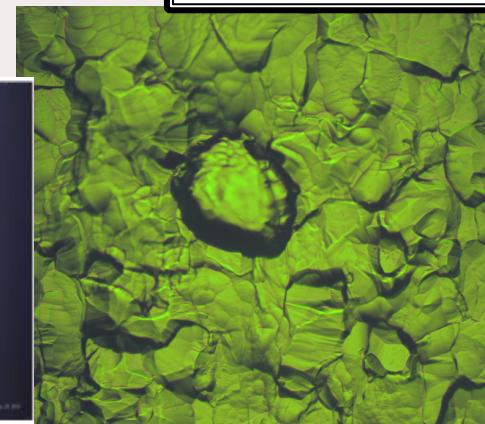
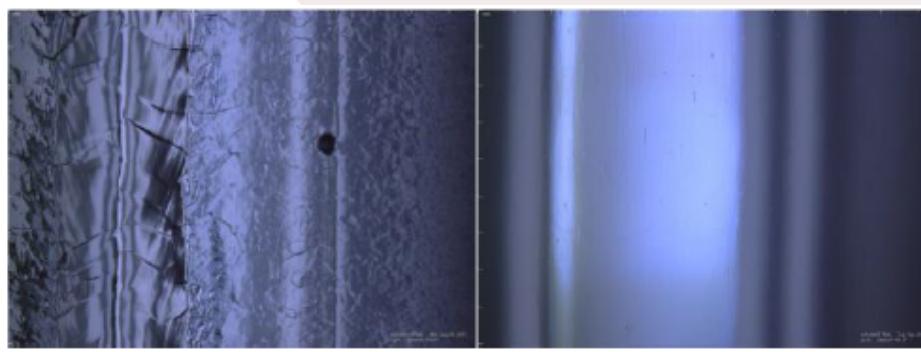
THE NEED:

- Topology and roughness analysis (-> confocal microscope)
- Identify inclusions ans surface pollution (-> SEM/XRD, SIMS)



Chemically etched
Nb sample

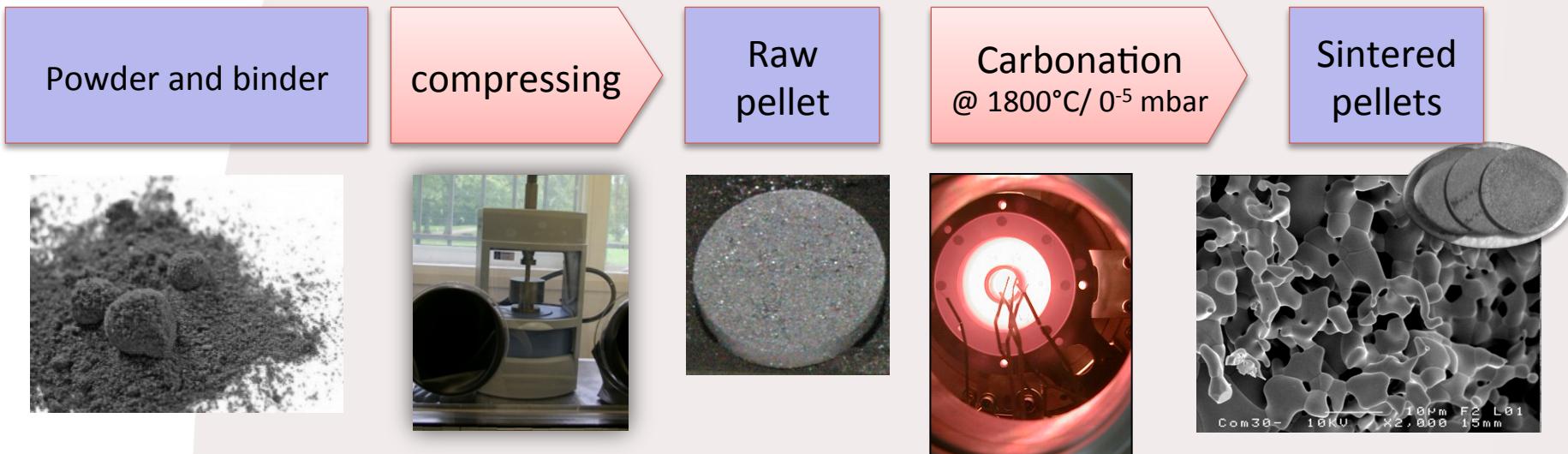
Mechanically
etched Nb sample



OPTIMIZING MICRO-STRUCTURE OF UCX TARGET

Pellet microstructure \Leftrightarrow fission product release \Rightarrow reproducibility and efficiency

Multi-parameter study: shape and size of powder grains, compressing and sintering method
 -> \Leftrightarrow impact on porosity – structure – micro-structure



DRX, He pycnometry, Hg porosimetry @ IPNO

SEM/XRD on radioactive samples @ labo. Sciences Chimiques de Rennes

SEM/XRD on stable samples (La_2O_3 ...) @ ICMMO

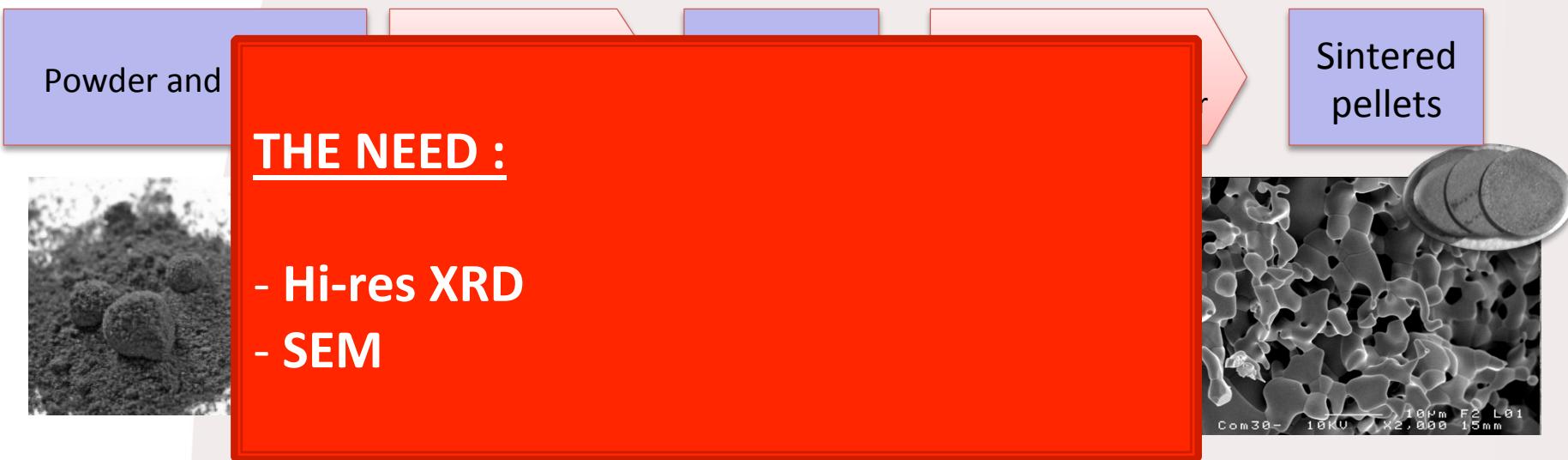
XRD and Hi-res XRF @ LISA

OPTIMIZING MICRO-STRUCTURE OF UCX TARGET

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Multi-parameter study: shape and size of powder grains, compressing and sintering method

-> \Leftrightarrow impact on porosity – structure – micro-structure



DRX, He pycnometry, Hg porosimetry @ IPNO

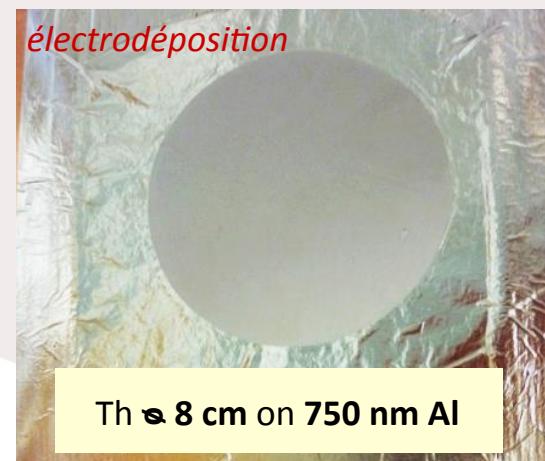
TEM/XRD on radioactive samples @ labo. Sciences Chimiques de Rennes

TEM/XRD on stable samples (La_2O_3 ...) @ ICMMO

XRD and Hi-res XRF @ LISA

➤ Development of radioactive thin films : experimental protocols developed with stable elements -> prototypes -> then transposed to radioactive isotopes

- Nature of stabilized phases ans structural crystallography : Grazing incidence XRD
- Composition of the layer : XRD , XPS, SIMS
- Chemical homogeneity : TEM/ XRD, XPS, SIMS
- Impurities analysis : SIMS
- Surface state characterization (thickness, roughness, homogeneity, grain size): SEM, confocal microscopy
- substrate bonding : microscopy



➤ Development of radioactive thin films : experimental protocols developed with stable elements -> prototypes -> then transposed to radioactive isotopes

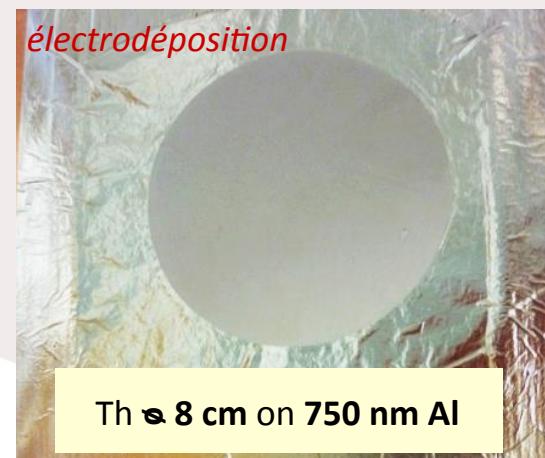
- Nature of samples
- Composition
- Chemical homogeneity
- Impurities
- Surface state
- confocal microscope
- substrate behaviour

THE NEEDS:

- Grazing incidence XRD
- SIMS or XPS
- SEM
- Confocal Microscope

ence XRD

ize): SEM,



Thin films for applications in particle accelerators

- Anti-multipacting layer (TiN) : nanometer-scale thickness measurement, layer stoichiometry, roughness, composition)
 - Reflectometry, XRD, XPS, SIMS
- Copper layer used for power coupler of SC cavities: thickness distribution (cylinder shape, bellows), roughness, composition and oxidation characterization
 - Confocal microscopy, XPS
- NEG coating: composition, thickness, roughness
 - XPS, SIMS, Confocal microscopy

Thin films for applications in particle accelerators

- Anti-multipacting layer (TiN) : nanometer-scale thickness measurement, layer stoichiometry, roughness, composition)
 - Reflectometry, XRD, XPS, SIMS
- Copper distribution and oxidation
 - THE NEED:**
 - Grazing incidence XRD, XPS or SIMS
 - Confocal Microscopy
- NEG coating: composition, thickness, roughness
 - XPS, SIMS, Confocal microscopy

Superconducting magnets

Measurements of mechanical and thermal properties of material between 300K and 4K to study and optimize new SC magnets (13 à 20 T) :

- Measurement of elasticity modulus, elastic limit, and SC compound material resilience
- Measurement of thermal conductivity and specific heat capacity

THE NEED:

- “Charpy sheep” @ cryo T°
- Dedicated cell test for thermal properties measurement at cryo T°

Calibration of coating equipment

At the CSNSM laboratory, several characterization instruments are already available : RBS, PIXE, ERDA, EDX et EELS.

One missing technique is the characterization of thin films of very low thickness (< 20 nm) : this is a frequently required measurement to calibrate thin films coating devices used for instance in the case of bolometer developments (like for EDELWEISS).

THE NEED:

- SIMS

INSTRUMENT NEED SUMMARY

- 5 instruments are frequently required in our scientific activity : confocal microscope, SIMS, XPS, SEM, grazing incidence XRD.
- Selected instruments being financed by P2IO (330 k€ awarded):
 - table-top SIMS
 - confocal microscope
 - “home made” cryogenic test cell for thermal characterization at cryo T°

SECONDARY ION MASS SPECTROMETRY

⇒ SIMS received in octobre 2015: Compact SIMS from Hiden Analytical

Beam:

Energie/courant: 1 to 5 keV / up to 400nA

Gaz: Oxygène ou Argon

Taille faisceau : < 50µm (imagerie) ,80 µm en profilométrie

Résolution : 2 nm

Modes :

- SIMS statique :

Spectre surface ($0 < m < 300$)

Image 2D

- SIMS dynamique

Profile : evolution des concentrations en fonction de la profondeur

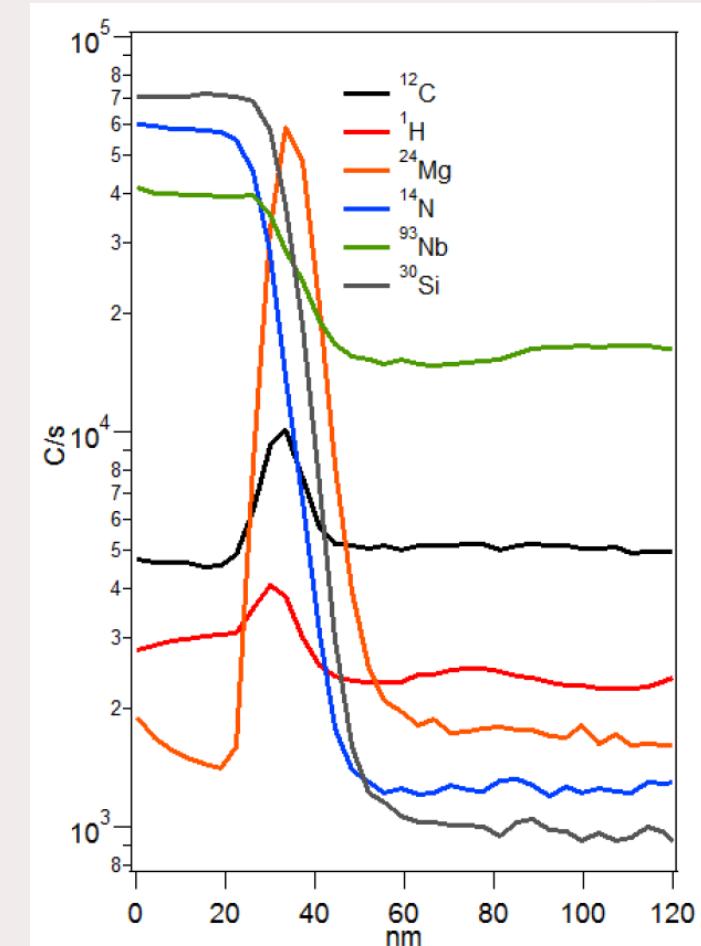
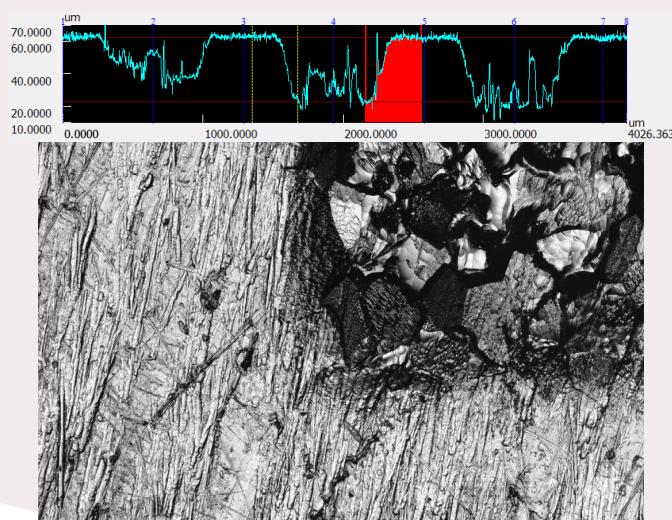
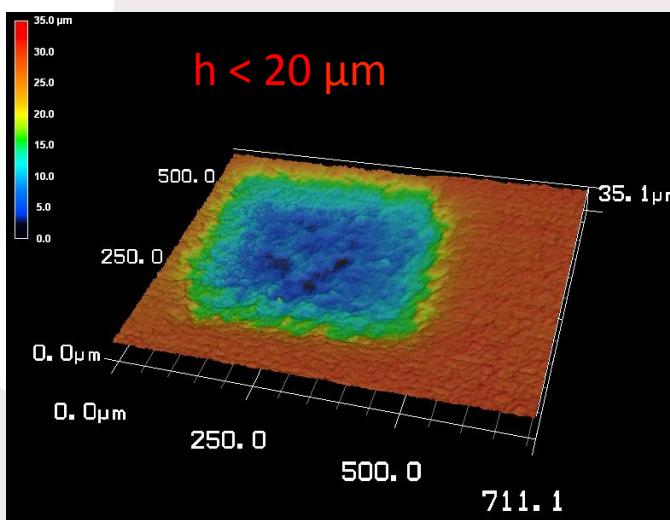
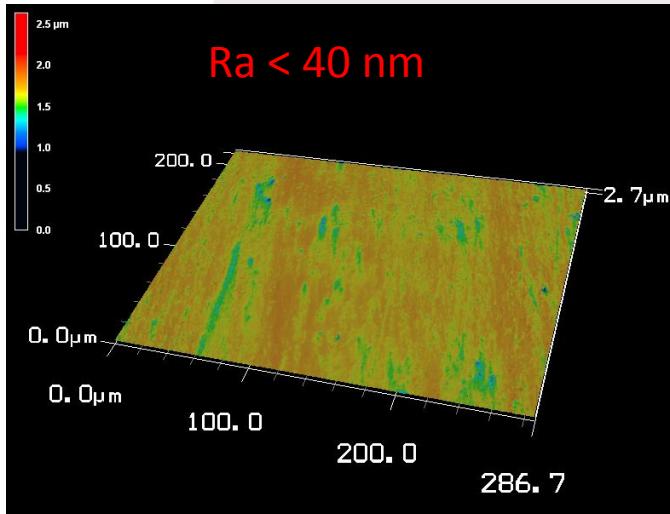


Figure 2: Detail of near surface region of oxygen primary ion depth profile.

CONFOCAL MICROSCOPE

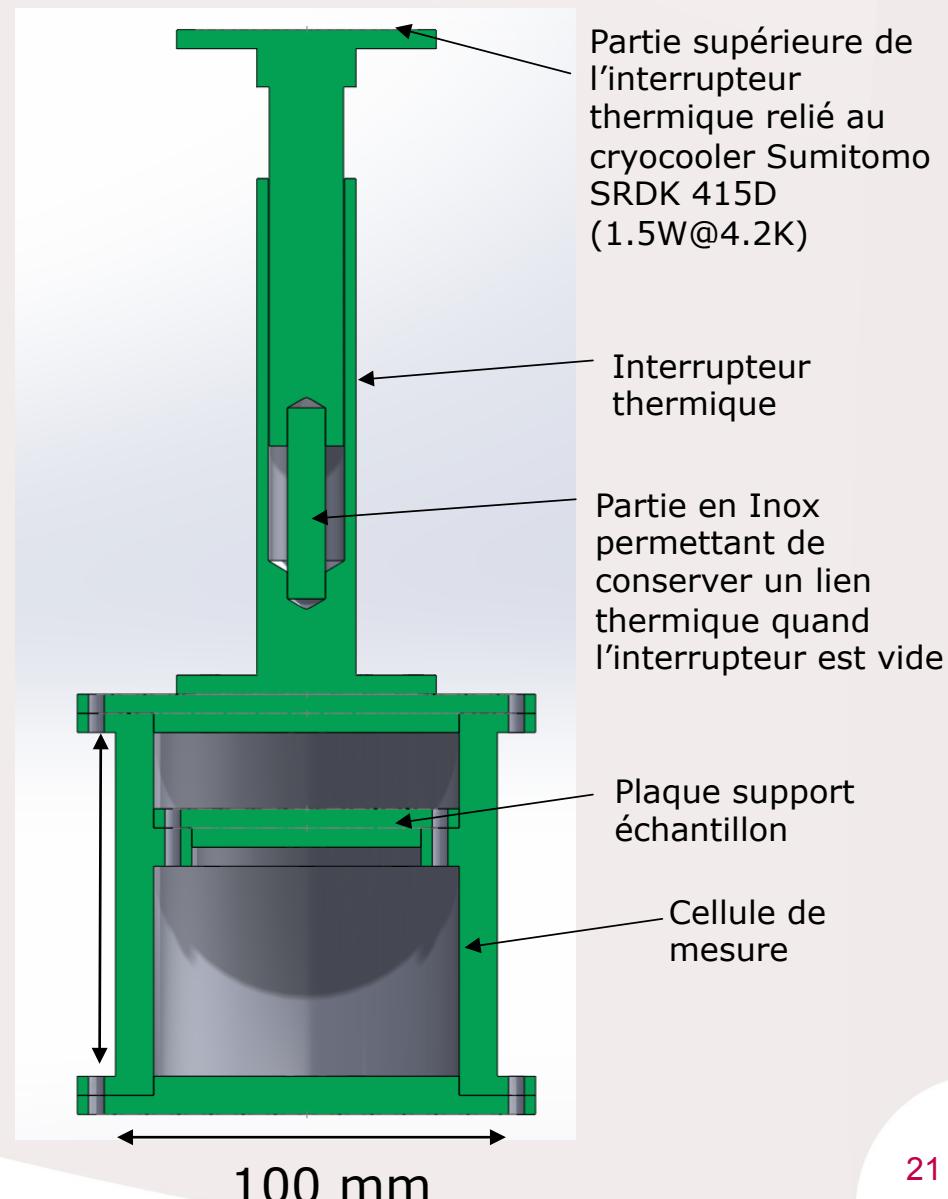
- Confocal microscope delivered in march 2015 : VKX200 de KEYENCE
 - Microscope optique et laser haute résolution (objectifs 10, 20, 50 et 150 fois)
 - Mesure de rugosité sans contact (10 microns > Ra > qq nm)
 - Mesure de profil (après analyse SIMS, évaluation épaisseur, ...)



MEASUREMENT OF THERMAL CONDUCTIVITY & DIFFUSIVITY

Mesure de conductivité thermique et de diffusivité
afin de déterminer le Cp de l'échantillon: Méthode la plus adaptée pour des échantillon de forte masse (100g max)

- Tous les plans sont réalisés
- cellule sera opérationnelle fin 2016 au CEA



GRAZING INCIDENCE XRD RE-INSTALLATION AND UPGRADE

–Ré-installation du diffractomètre à rayon X en configuration rasante en 2016 :
modèle de PANALYTICAL , bâtiment 200, LAL

–Formation effectuée en mars 2016 par un expert du CSNSM (Mr Gael Sattonnay)

–DRX configuration θ , 2θ : analyse bulk, poudres

–DRX rasant : analyse couches minces, reflectométrie

–Mesure des contraintes, textures, ...



SUMMARY AND FUTURE WORK

➤ Instruments :

- Confocal microscope : installed and operational
- SIMS installed and operational
- Grazing angle DRX: software upgrade
- Specific heat measurement : under development

➤ New instruments also foreseen: SEM-EDX, XRD

➤ A network of local experts is already existing among the P2IO laboratories

➤ Location :

In the coming 2 years, a dedicated space to integrate all these instruments (except cryogenic characterization) will be available

Thanks for your attention



Additionnal slides

Technique destructive d'analyse de surface. Mesure la concentration élémentaire de la surface. Elle permet d'obtenir des détections limite d'éléments trace entre 10^{12} et 10^{16} atomes/cm³. Du fait de la pulvérisation de la surface de l'échantillon, la technique permet aussi la reconstitution de "profils en profondeur" jusqu'à une profondeur de 10 µm.

■ Interactions en jeu :

- Excitation : bombardement avec un flux d'ions (Ar, O, Xe, Ga, ...)
- Réponse : électrons, photons, atomes et molécules neutres, atomes et molécules excités, ions.

■ Informations obtenues :

- Analyse élémentaire : tous les éléments sont détectables (H compris)
- Analyse quantitative des éléments présents
 - absolue : impossible sans un étalonnage préalable (effets de matrice)
 - comparative : possibilité de comparer les intensités entre plusieurs échantillons de même matrice

■ Profondeur analysée :

Profondeur d'analyse variable de 1 nm à 20 µm
↳ méthode d'analyse de surface

Les rayons X sont diffractés de façon différente par le matériau, selon qu'il soit cristallisé ou non. Technique non destructive.

■ Informations obtenues :

- **identification des phases en présence, nature cristallographique**
- une analyse de la **déformation** de réseau : contraintes = déformations *et* texture = orientation préférentielle...

Dans le cas d'un système polyphasé :

- **analyse quantitative** des phases présentes possible mais pas triviale (méthode de Rietveld), déduction possible de la quantité de phase amorphe présente

■ Profondeur analysée : Profondeur d'analyse variable selon la nature chimique, du micromètre au millimètre → méthode d'analyse de volume

Nature de l'élément	Profondeur de pénétration (μm)
Pb	8,6
Fe	9,4
Ti	26,1
Al	175
Mg	347