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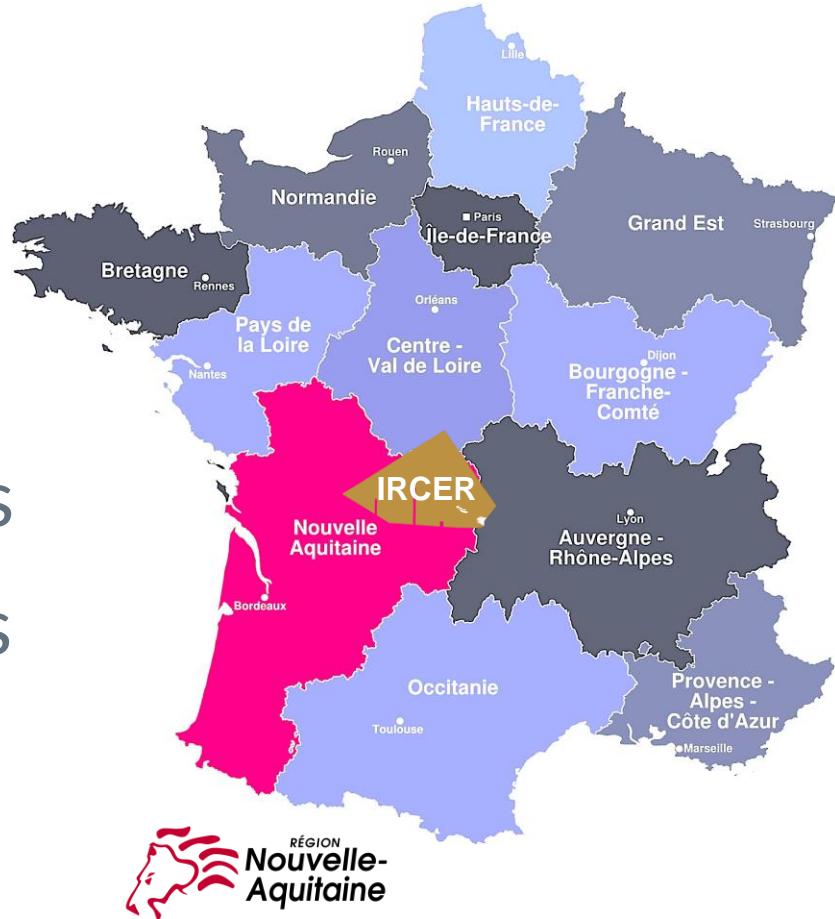
irCer
institut de recherche
sur les céramiques

Joint Research Unit



IRCER

1. Historical context
2. Key facts & figures
3. Four research axes
4. Major facilities*



* The "CARMALIM" platform
(CARactérisation des MAtériaux de LIMoges)

1. Historical context

More than 2 centuries of history

Progressive affirmation of a sector of excellence in technical ceramics

18th century to 1970s

The porcelain industry

1767: Kaolin Discovery

10,000 workers in 1891
at the apogee

Decline from early 1900
due to strong worldwide
competition

From early 1980s

Towards technical
ceramics

1979: Engineering college

1981: 1st lab dedicated to
technical ceramics

Technology transfer centers

1984: Ceramic processes

1998: Surface Treatments

From 2005

Sector of excellence

2005: Creation of the ceramic
competitiveness cluster (PEC)

2010: All research activities
in a single building (CEC)

2018: All research under a
single banner = **IRCER lab**

2. Key facts & figures

Largest academic lab in its field in EU

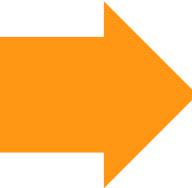


*Transformations of matter involved in
processes for bulk ceramics and
processes of surface treatment*



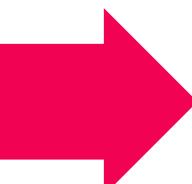
IRCER positioning

Materials Science & Process Engineering
Fundamental & Applied Researches



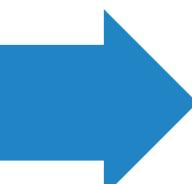
10,000 m²

Laboratory surface



200 members

Half being permanent staff



5,000,000 €

Annual budget without salaries

Applications & Societal Needs



Energy Transport

Intensification & Production

- Thermal barriers, wear / nuclear mat.
- SOFC/HTE, catalytic reactors (H_2/O_2)
- Solid-state batteries, capacitors...

Severe operating conditions (T, atm.)



Information & Communication Tech.

Mostly HF & laser devices

- 3D-printed HF devices, MEMS
- Vitro-ceramics for non linear optics
- Transparent ceramics, Pb-free mat.

Lab of Excellence Σ-LIM



Human Health

Diagnostic & Therapy

- Implants for Bone Tissue Eng.
- Peptidic ligation & drug delivery
- Bioreactors and cancer sensors

In-vitro, ex-ovo & in-vivo tests



Ecomaterials Environment

Sustainable development

- Biosourced additives
- Silicates, geopolymers
- Low carbon cements

LCA & recycling



Industry 4.0 Factory of the Future

Additive manufacturing

- Feedstocks (biosourced / responsive)
- Technologies (hardware / software, hybridization / multimaterials)

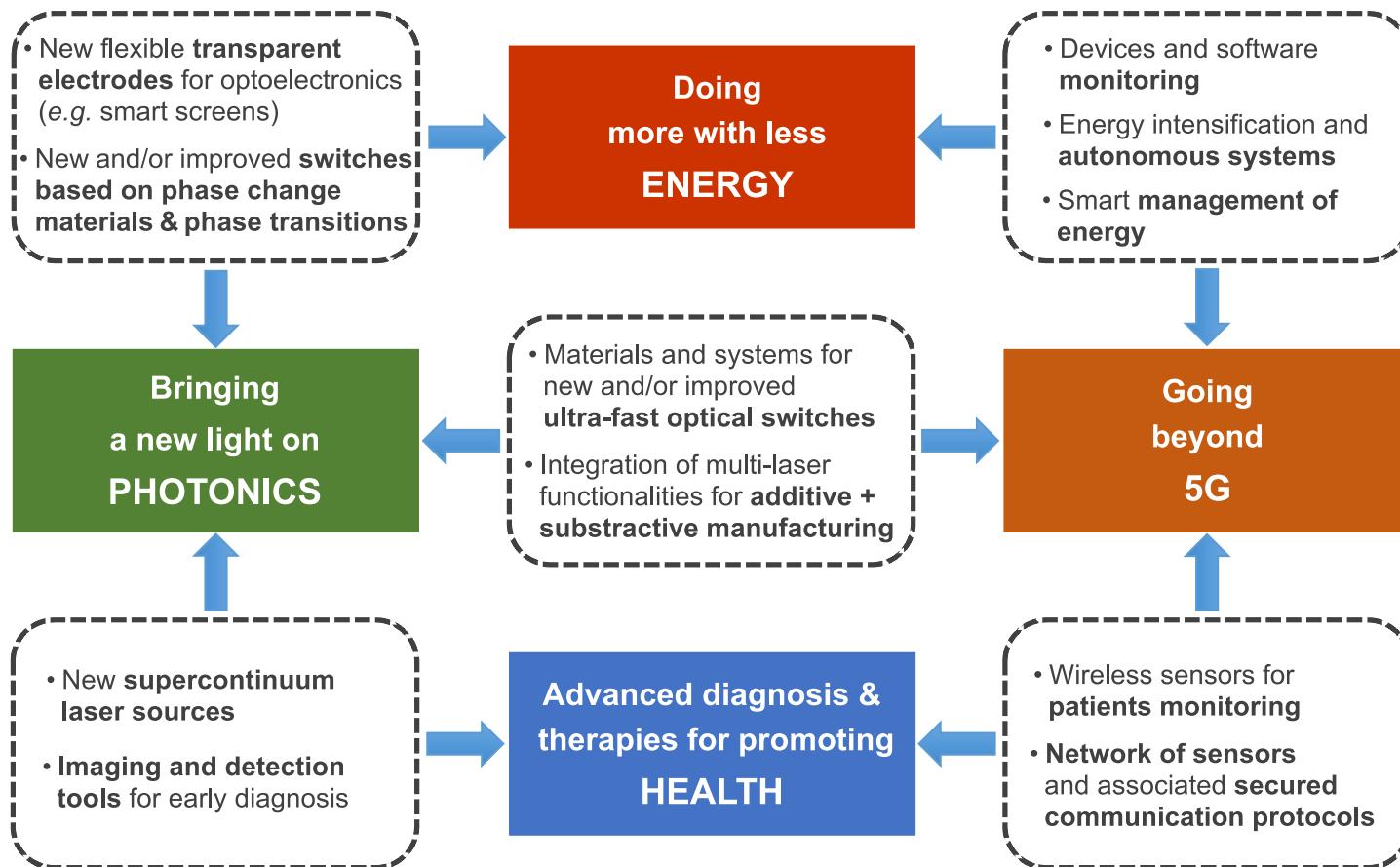
Optimization (topology / trajectory)



Multiscale Numerical Simulations

Ato/nano/meso/micro/macro

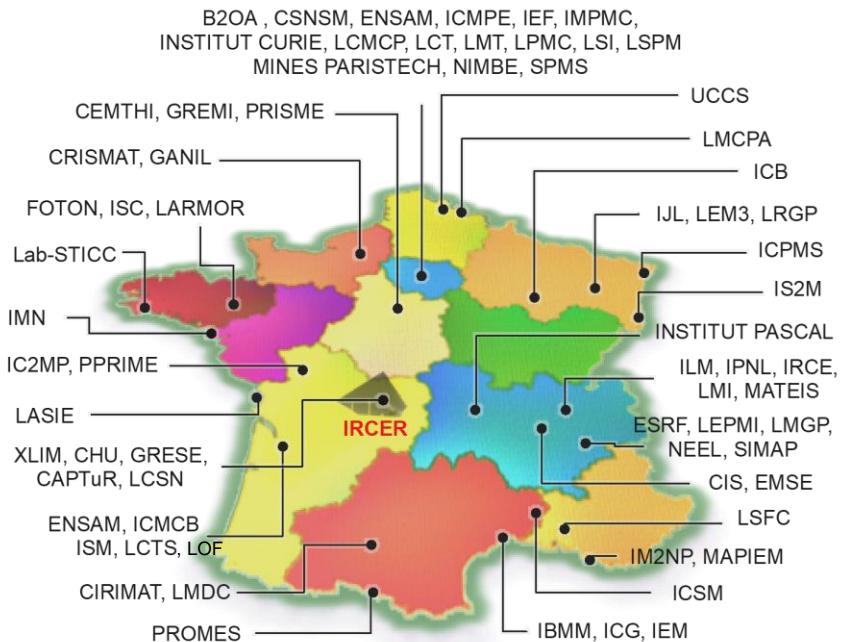
- *Ab-initio* DFT, Monte-Carlo
- BD, MD, MD-SRD
- Fluid dynamic, FEM / DEM
- GPUs calculation



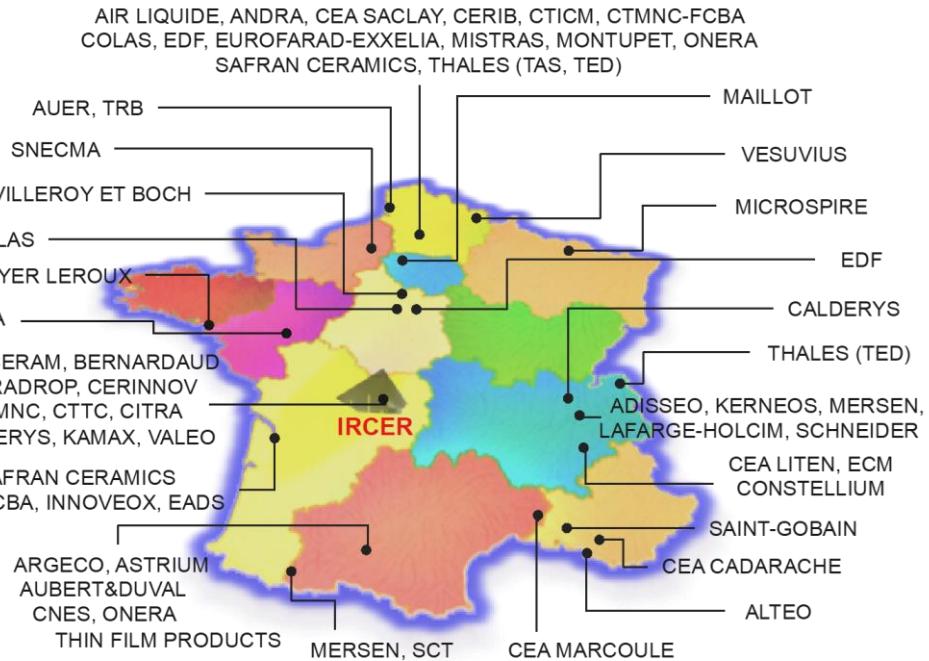
"From materials and specific ceramic components up to integrated, secure & intelligent communicating systems"



National collaborations



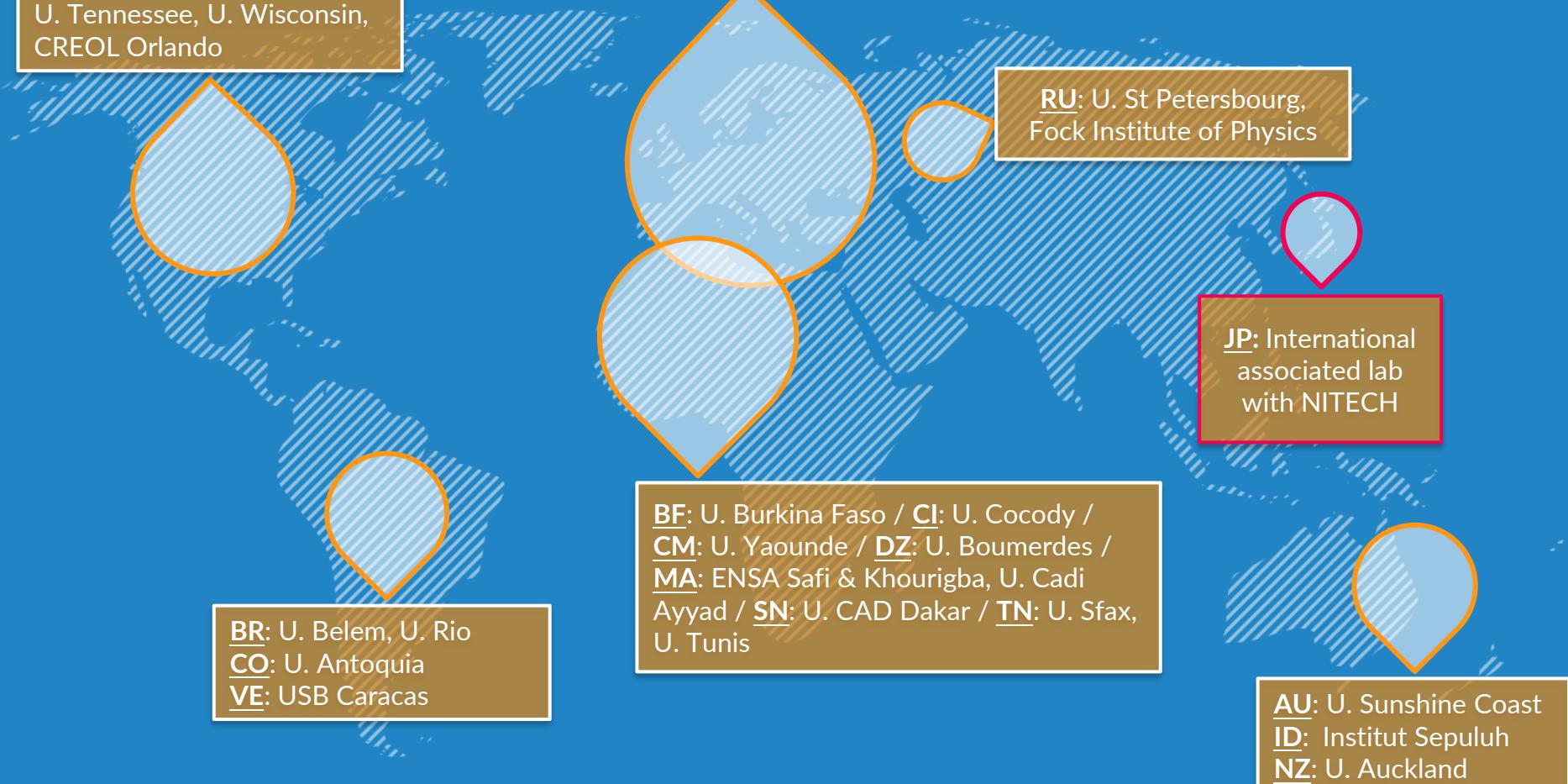
Academic



Industrial

CA: U. Québec 3 Rivères
US: U. Central Florida,
U. California, U. Columbia,
U. Tennessee, U. Wisconsin,
CREOL Orlando

BG: UTBM Sofia / **CH**: ETHZ, Ansaldo / **CZ**: UTC Praha / **DE**: U. Aachen,
Alantum, U. Chemnitz, U. Dresden, KIT, U. Ruhr-Bochum / **ES**: U. Tarragona,
CSIC Madrid / **FI** : U. Aalto, U. Tampere / **GB**: U. Cambridge, Imperial College /
IE: U. Limerick / **IT**: U. Genova, U. Modena, U. Padova / **PL**: AGH, IISTR, U.
Varsaw, U. Wroclaw / **PT**: Aveiro Institute of Materials / **RO**: U. Cluj-Napoca /
SI: Jožef Stefan Institute / **SE**: U. Linköping



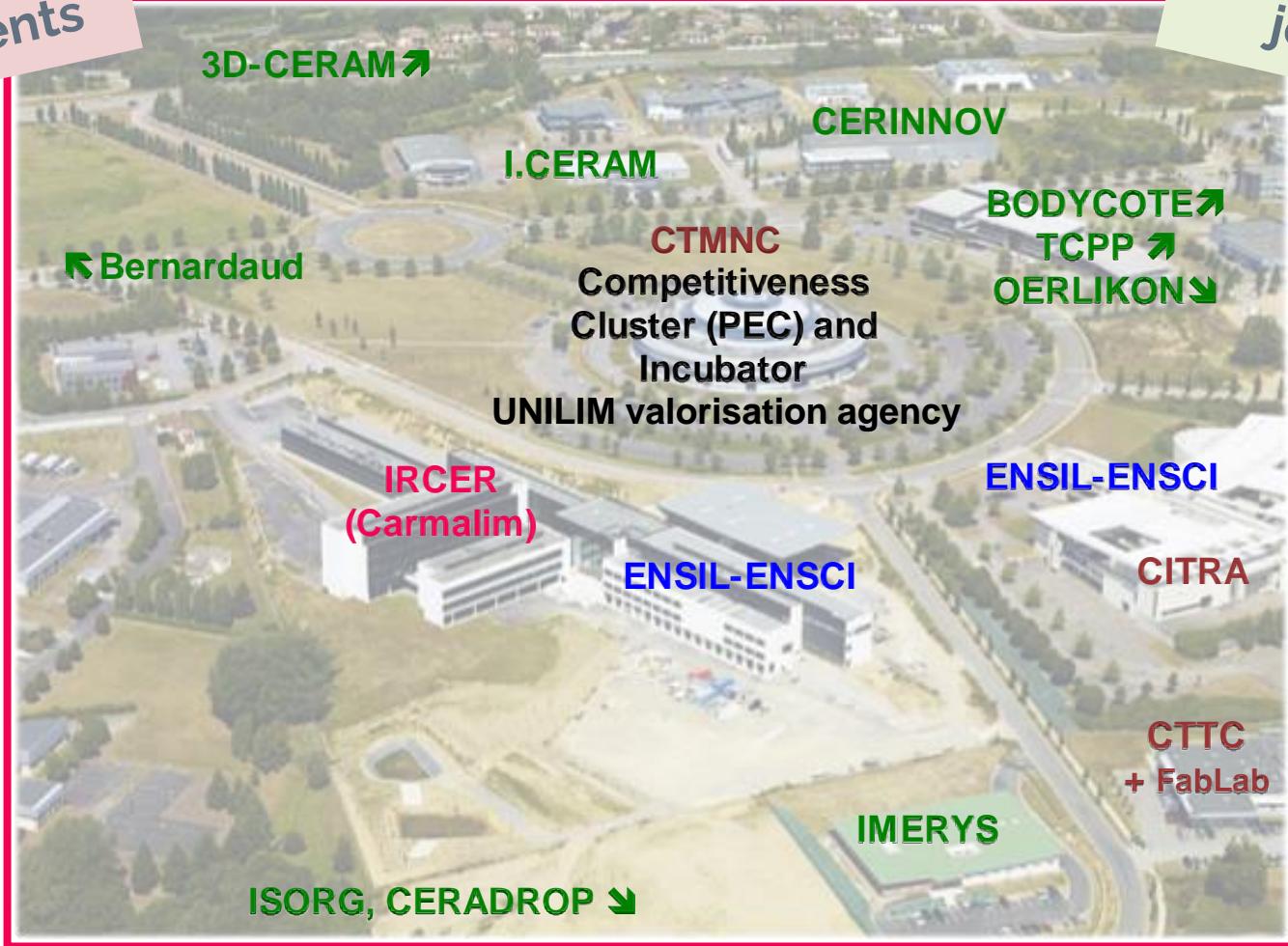
International collaborations

The Limoges technopole

A ceramic innovation "valley"

500
students

2000
jobs



Governing bodies

Engineering college

Research lab

Tech. transfer centers

Companies

3. Four research axes

Techno-push & market-pull approaches

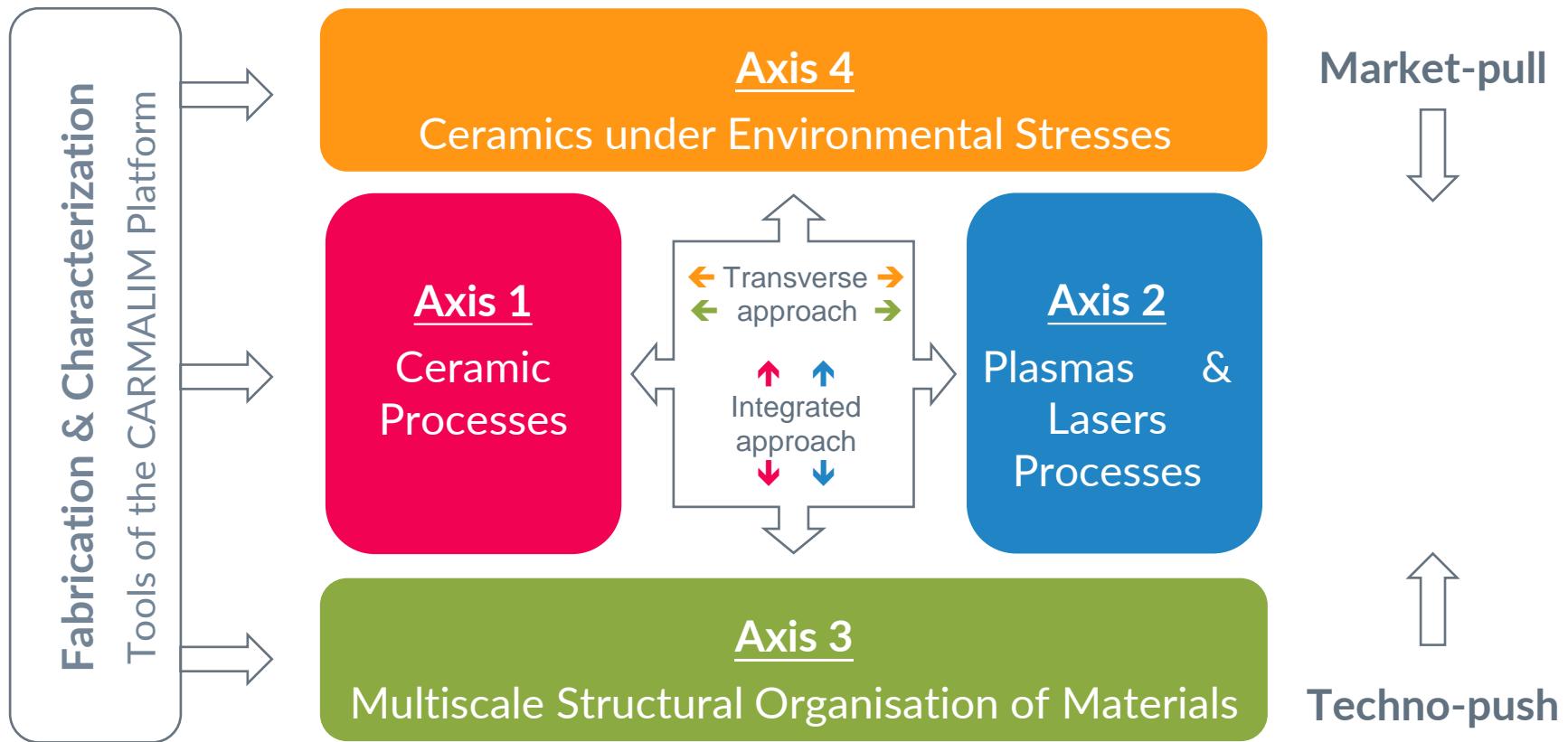


“

4 research axes with a matrix organisation:

- *Experiments & modeling / simulations*
- *Integrated / transverse developments*

Matrix organisation: 4 research axes





Axis 1

Ceramic Processes

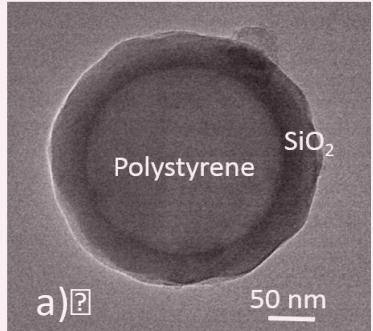


Prof. A. VIDEKOQ
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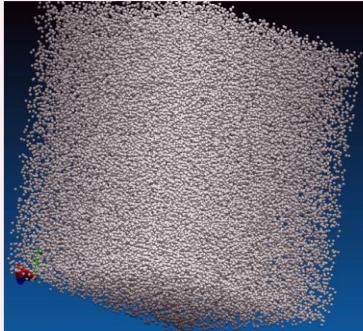
e.g. Ceramic processes

→ PROCESS CONTROL

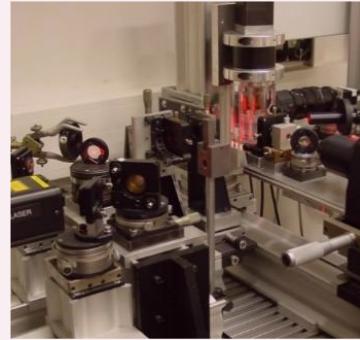
From specific powders to raw parts



Specific powders
(e.g. core-shell)



Suspension structure
(MD-SRD modeling)

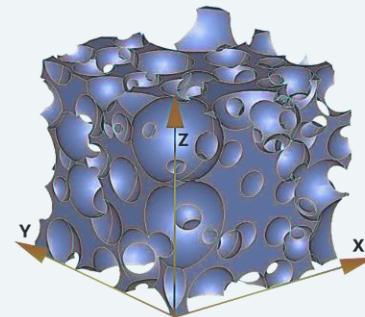
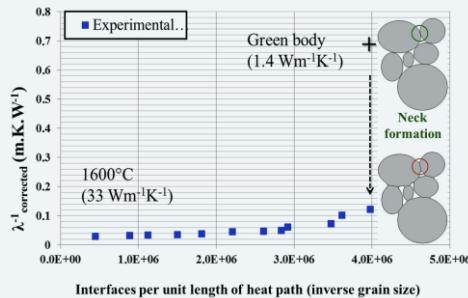
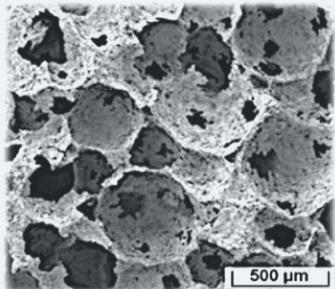


Rheology coupled
with DLS



Optimization of standard
shaping processes

Consolidation stage before sintering (debinding, drying...)

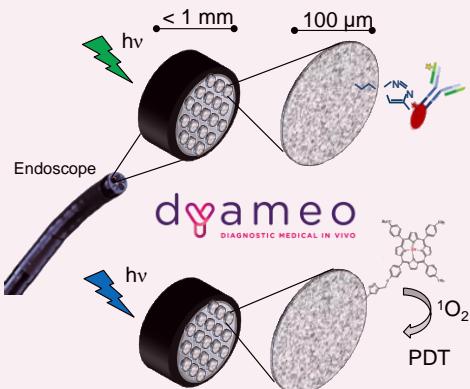


Thermal properties of porous networks: experiments and modeling (e.g. DEM)

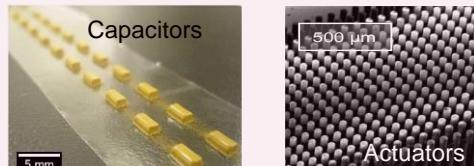
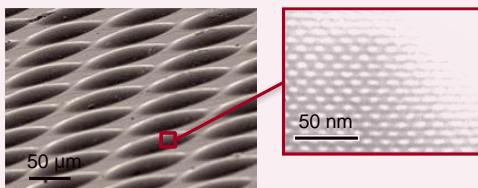
e.g. Ceramic processes

→ ADDITIVE MANUFACTURING since 2000

Inkjet printing



Smart endoscope for
cancer diagnostic & therapy



Microelectronic devices



Valorisation
Total of 40 employees



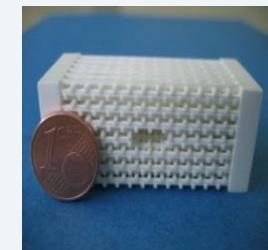
(micro) Stereolithography



3D implants



Thierry CHARTIER
CNRS Innovation Medal 2018

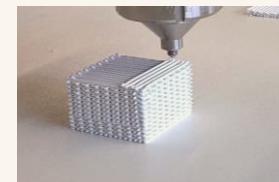


Filters and antennas

Robocasting & Direct Ink Writing



Biosourced feedstocks



Lattice structures

Current issues: Hybridization (multimaterials), topology optimization/increase complexity, mass production/large parts, trajectory control (5 axes), machine learning, artificial intelligence...



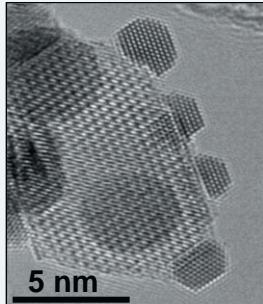
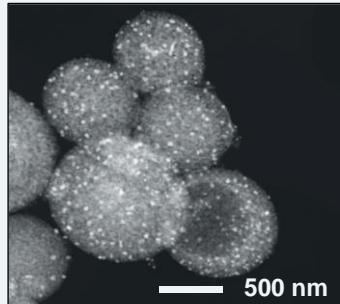
H2020 MSCA RISE
Coordination IRCKER

e.g. Ceramic processes

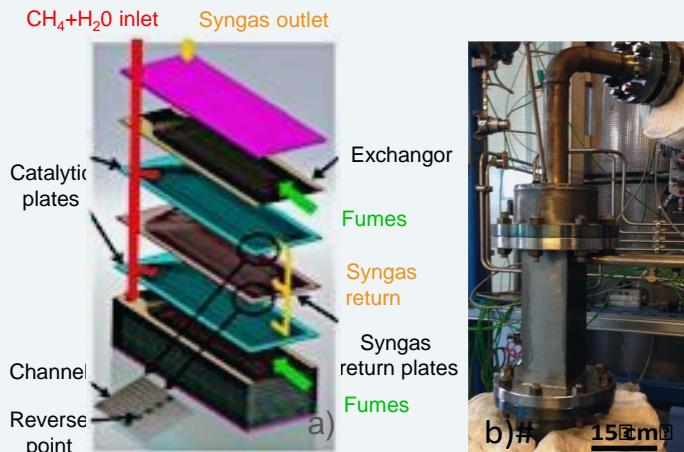


→ JOINT LAB with AIR LIQUIDE since 2005

Steam Methane Reforming



Processing of new catalysts and washcoats

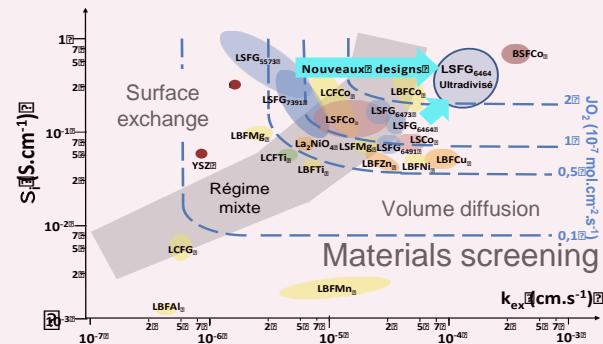


3D-printed millistructured reactor functionalized with new catalysts and tested on a pilot bench

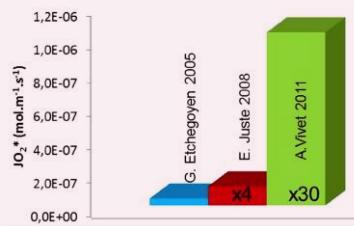
Catalytic Membrane Reactors



Flux and surface activity measurement

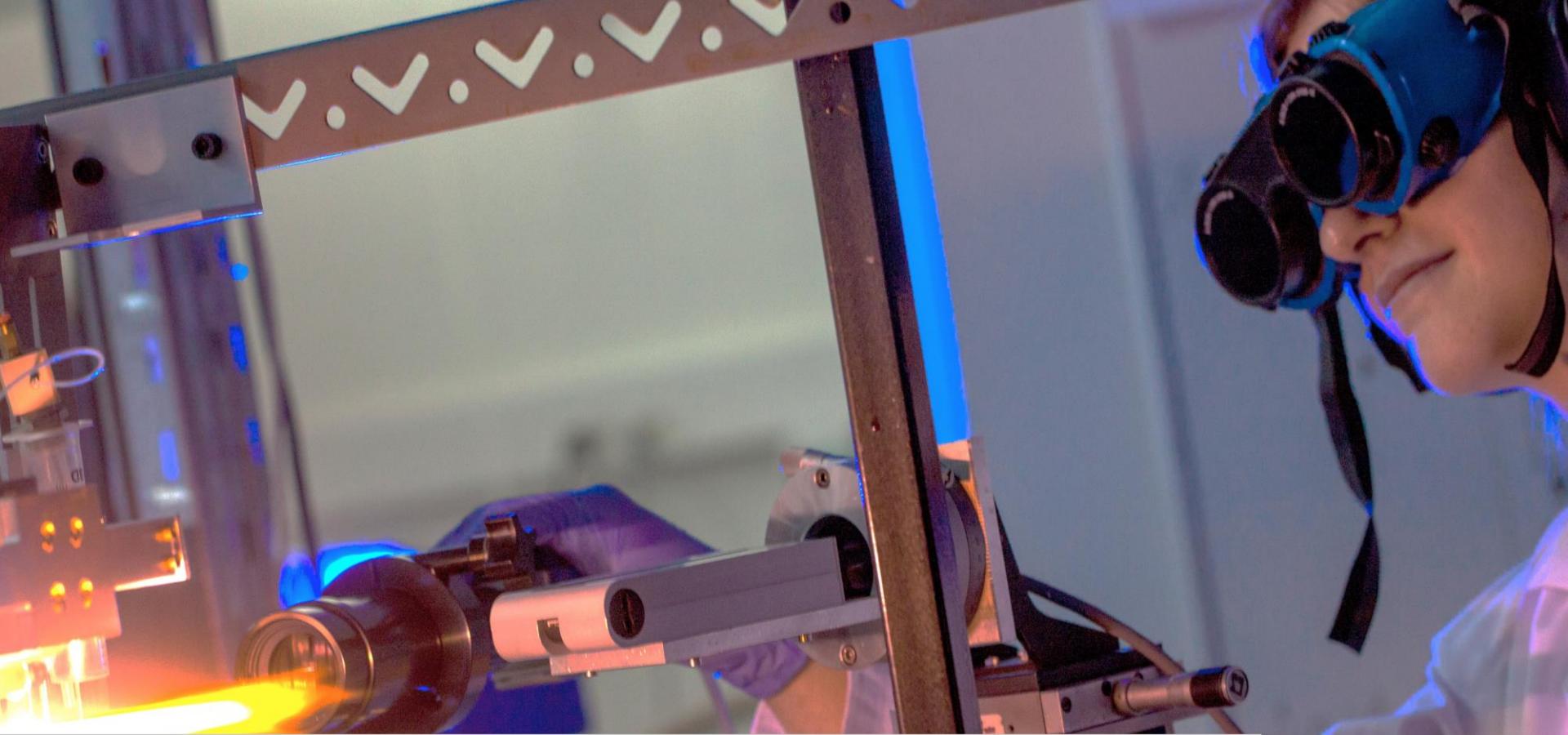


Performance increase



IKTS pilot (O_2)





Axis 2

Plasmas & Lasers Processes

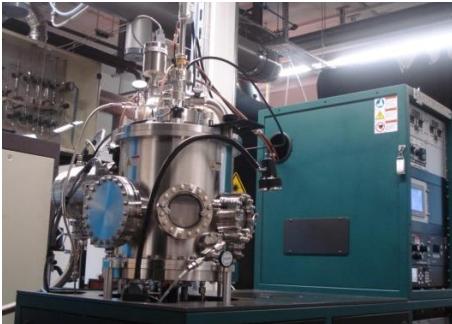


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Surface treatment processes at IRCER

1 nm - 1 μm



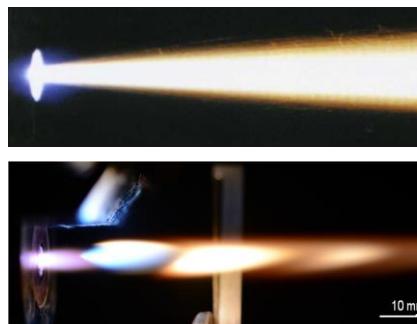
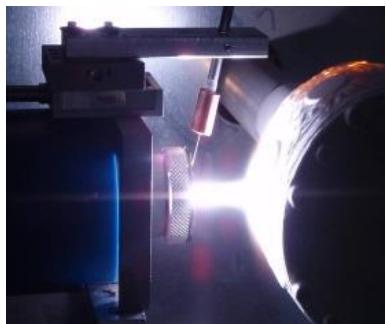
PLD → nanoparticles generators ← PVD

100 nm - 5 μm



PECVD

10 - 1000 μm



Plasma spraying
APS/SPS/SPPS

Arc spray



HVOF spraying

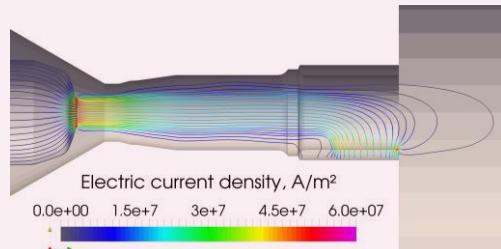


Cold spray
High Pressure

e.g. Plasmas & Lasers Processes

→ THICK FILMS

Plasma spraying



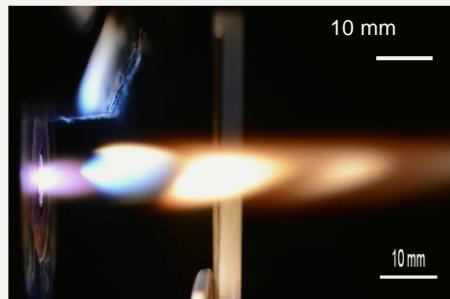
- Understand, optimize & develop sources
- Material treatment in flight
- Coatings and properties
-
- Instability control



Lech PAWLOWSKI
ASM Hall of Fame 2015

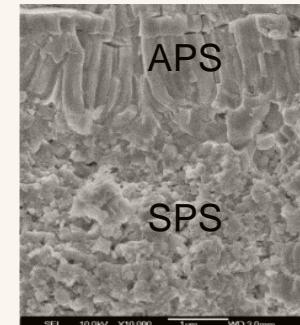


Armelle VARDELLE
ASM Hall of Fame 2016



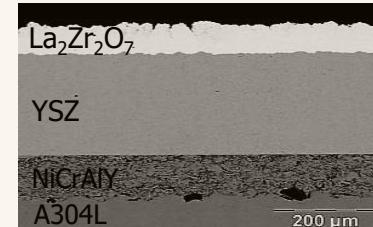
Pulsed arc plasmas

- Self-sustaining pulsed source
- Diagnostics and modeling
- Synchronous inkjet injection



SPS-APS

Bilayer nanostructured thermal barrier for extreme conditions



SPS-SPPS



Joint lab since 2002

Thermal plasmas &
Properties of coatings

Specific bench test

Blown plasma in controlled atm. to
test materials for space applications



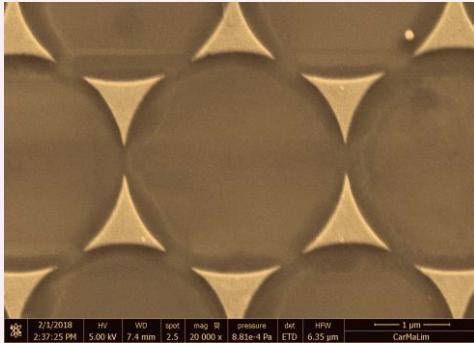
e.g. Plasmas & Lasers Processes

→ THIN FILMS

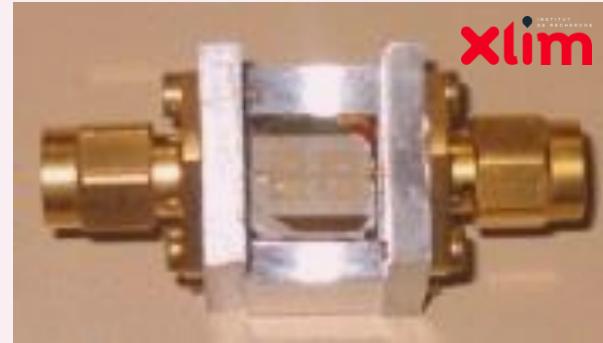
Laser ablation (from materials to components/devices)



Tunable properties, doping,
nanocomposites (VO₂, BST)

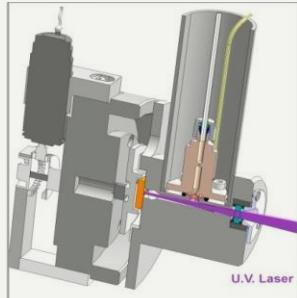


Surface
structuration

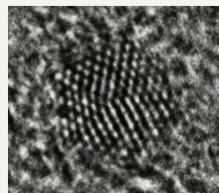


Integration in
RF components

Elaboration of nanoparticles in flight



Laser
sputtering



Magnetron
sputtering

DLC thin films



Industrial process coupling PVD & CVD

Axis 3

Multiscale Structural Organisation

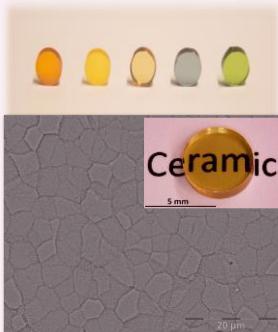
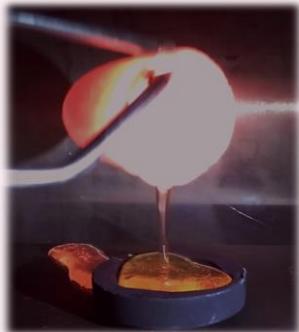


Dr. J. JOIN
jenny.join@unilim.fr

e.g. Multiscale Structural Organisation

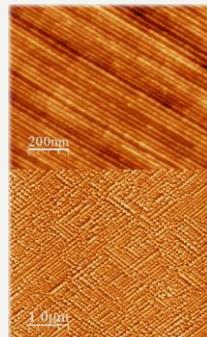
→ SYNTHESIS & ELABORATION OF OXIDE MATERIALS

Glasses & (vitro)ceramics

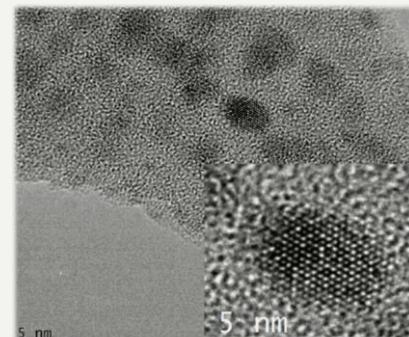


Synthesis of TeO_2 based glasses and ceramics by full glass crystallization

Nanostructures

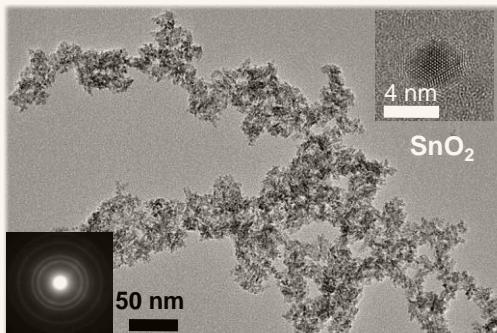


self-organization of vicinal surfaces



Sol-gel synthesis of nanocrystals in SiO_2

Nanocrystals of metallic oxides



Synthesis of ultrasmall SnO_2 nanocrystals by non hydrolytic sol gel route

Geopolymers



Polycondensation reaction from silicate solution clay

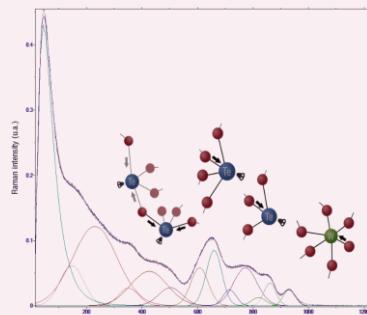
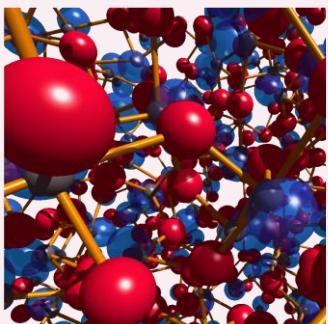


Additive manufacturing of geopolymers

e.g. Multiscale Structural Organisation

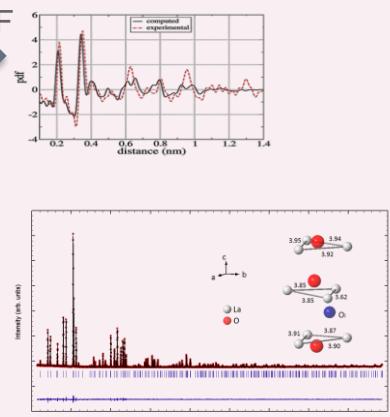
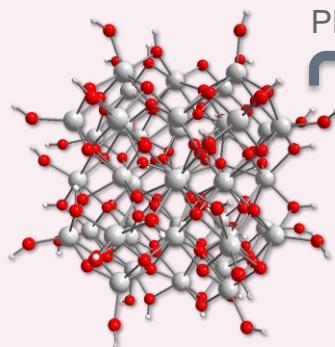
→ STRUCTURAL & NANOSTRUCTURAL STUDIES

Complex structures (deordered)



Structure of TeO_2 glass
MD simulations & PDF

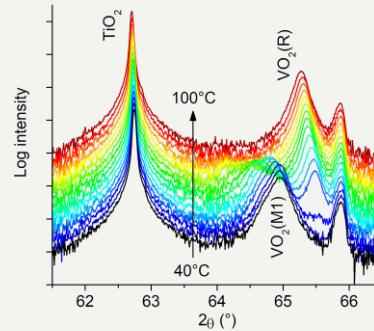
→ Short range order
Raman scattering



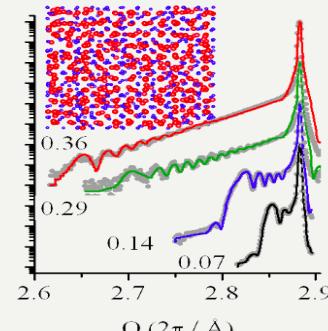
Structure models of
 ZrO_2 nanocrystals

Local structure of
lanthanum silicate

Defects in epitaxial films

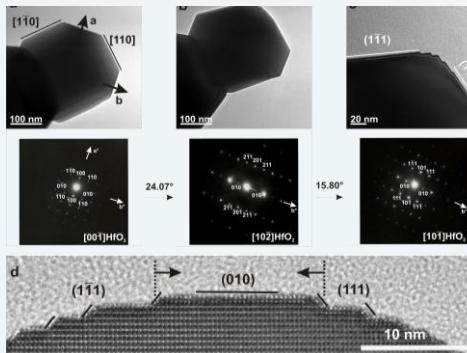


XRD characterisation of
 VO_2 epitaxial film



Amorphisation of SiC
under irradiation

Surface / Interfaces

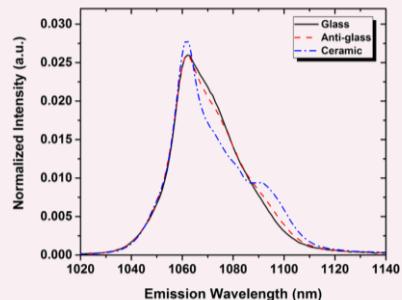


Growth mechanism of HfO_2
during a carbothermal reaction

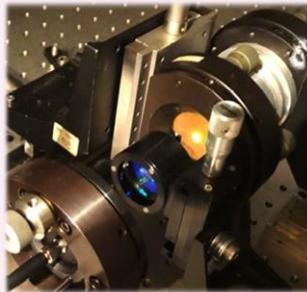
e.g. Multiscale Structural Organisation

→ PHYSICAL PROPERTIES

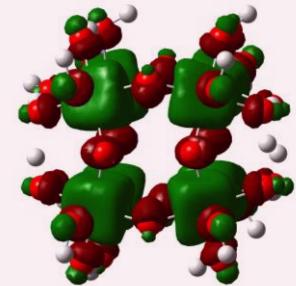
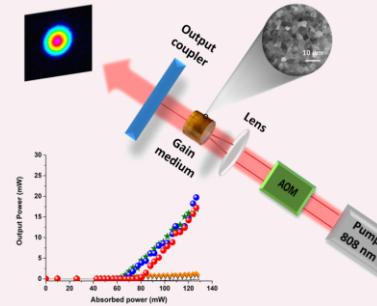
Optical properties: light emission and non-linearities



Photoluminescence spectra of TeO_2 based glasses & ceramics

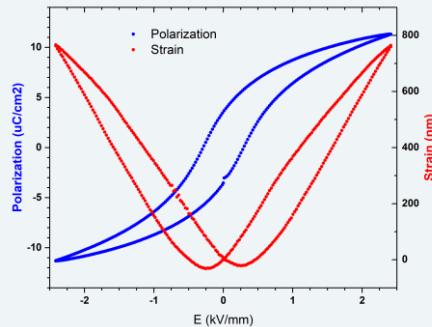
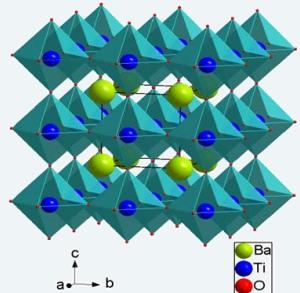


Continuous laser emission in bulk tellurite ceramics



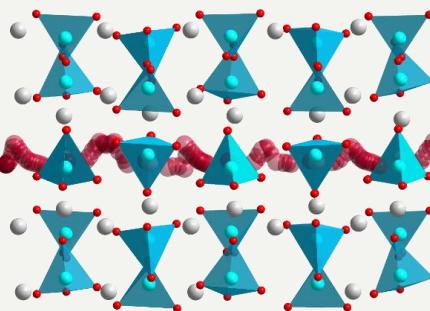
Calculation of NLO properties using DFT

Ferroelectric/piezoelectric properties



Polarisation and strain loop from BaTiO_3 ceramics

Ionic conductivity

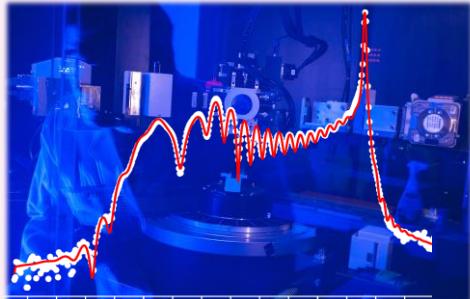


Oxide ions conduction pathway in lanthanum silicate compounds computed by DFT

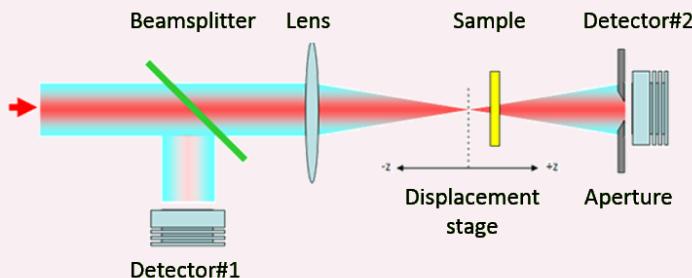
e.g. Multiscale Structural Organisation

→ ADDITIONAL ACTIVITIES

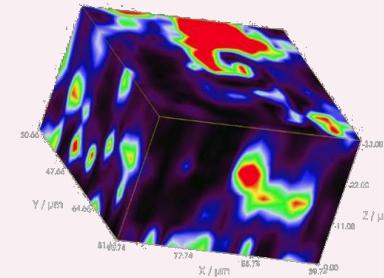
Advanced analyses



Advanced X-ray
crystallography



Measurement of optical nonlinear properties
(Z-scan, CARS)



Chemiometry for
3D Raman imaging

Computing

A code editor window showing Python and C++ code. The Python code defines a Gaussian fit function and processes a matrix. The C++ code includes icons for C++, Python, and other languages.

Scientific computing &
software development

Access to large scale facilities



ESRF, SOLEIL , ILL ...

Axis 4

Ceramics under Environmental Stresses



Prof. S. FOUCAUD
sylvie.foucaud@unilim.fr

e.g. Ceramics under Environmental Stresses

→ RELATIONSHIP ELABORATION/MICROSTRUCTURE/PROPERTIES

Preceramic polymers

Synthesis / rheology / pyrolysis in controlled atm.

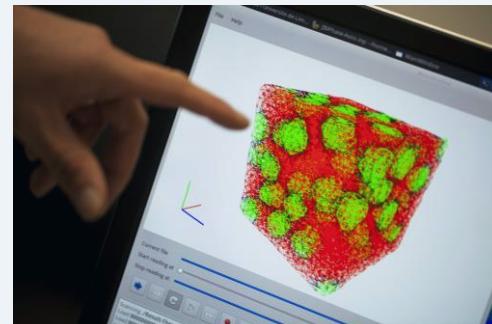
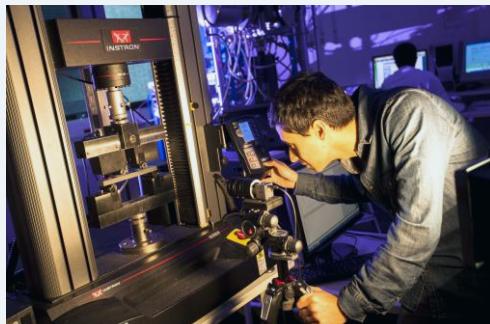
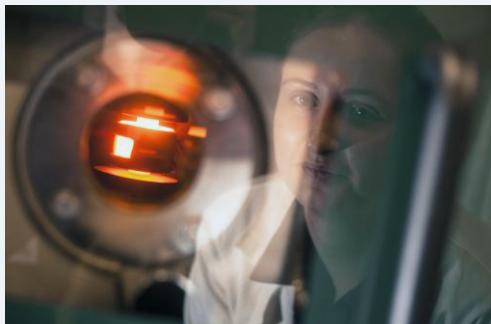
Fibres (electrospinning), porous materials (templating), nanocomposites



Multiscale approach of sintering

Thermomechanical tests

Discrete Element Modelling



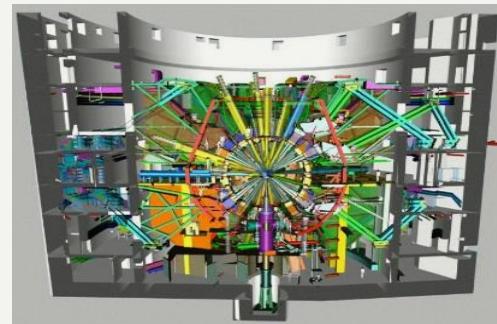
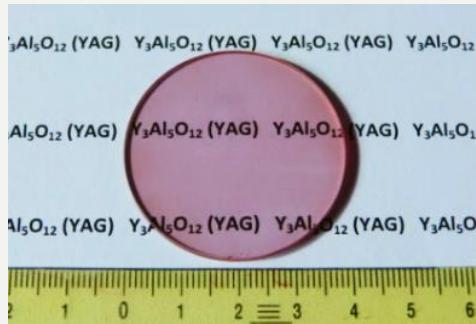
e.g. Ceramics under Environmental Stresses

→ HIGH POWER LASERS & BONE TISSUE ENGINEERING

Transparent ceramics

Doping - Functionally graded materials

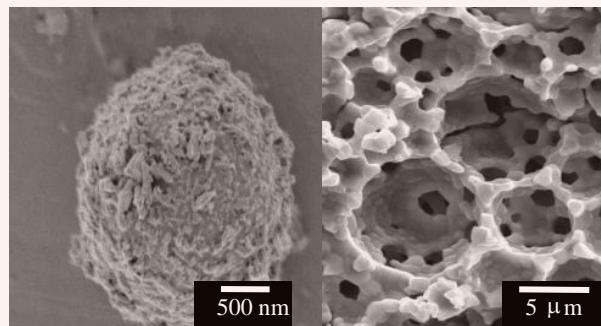
Megajoule Laser (LMJ) for nuclear fusion simulation



Bioceramics

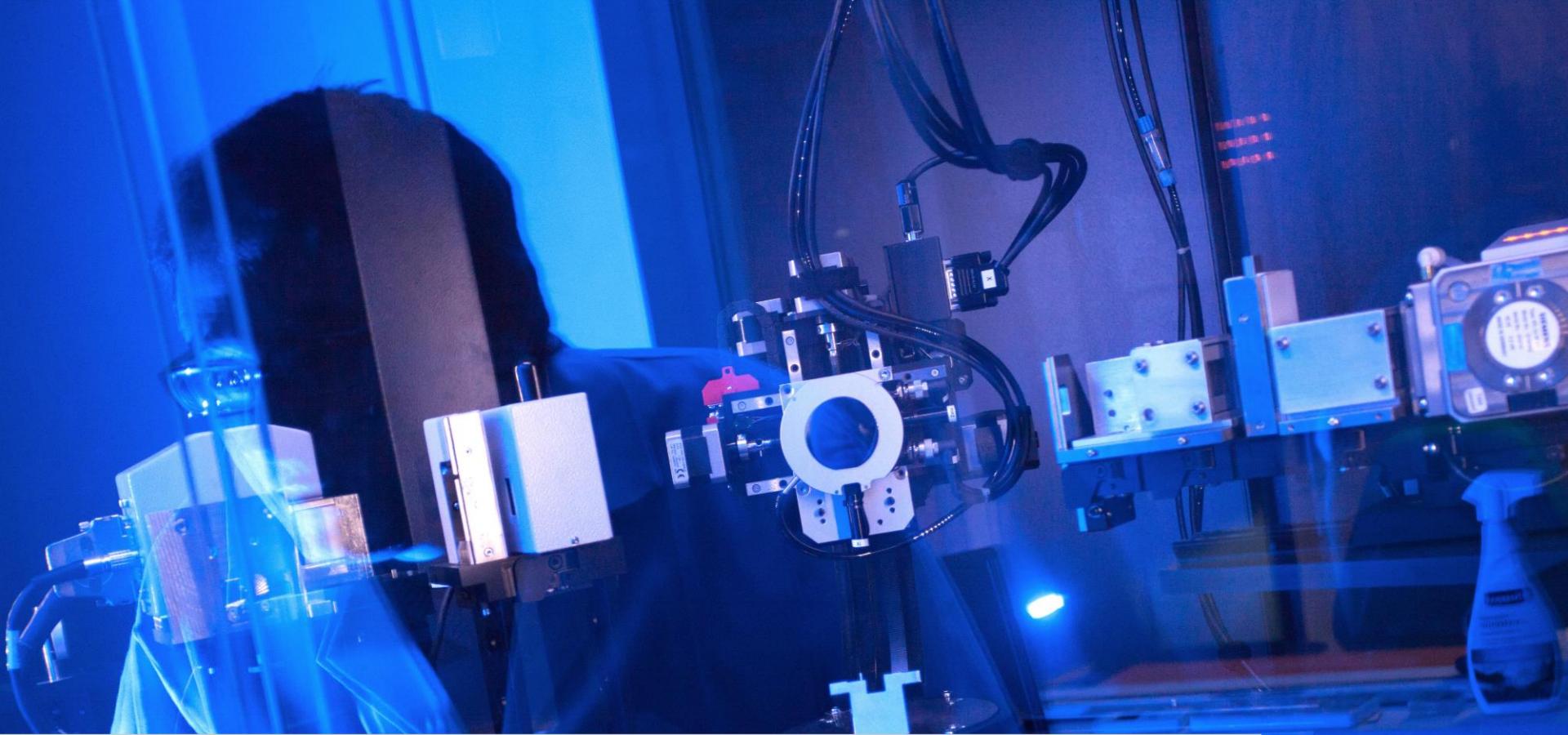
Functionalisation

Drug delivery, 3D implants



4. Major facilities

Advanced fabrication & characterization tools



CARMALIM (carmalim@unilim.fr)

Characterization of Materials of Limoges



Eng. E. LABORDE
etienne.laborde@unilim.fr

CARMALIM: 13 technical staff / 6 poles

MICROSCOPY

- 1 FIB-SEM (EDX-3D, EBSD-3D, litho.)
- 1 ESEM (in-situ mecha & thermal treatment)
- 2 FEG-SEM (EDX, cryo-stage)
- 1 SEM (standard - EDX)
- 1 HR-TEM (200 kV, EDX, HAADF-STEM)
- 3 AFM (including 1 fast scan)
- 2 Interferometers (optical)
+ sputtering, ion etching, high pressure freezing, polishing...

X-RAYS

- 2 Standard diffractometers
Both equiped with a sample holder and a furnace up to 1200°C
Specific set-up:
 - 1 XRD on monocrystals (4 cercles)
 - 1 HR-XRD on epitaxial layers
 - 1 Total scattering equipment
 - 1 X-Ray fluorescence

THERMAL, CHEMICAL and MORPHOLOGICAL ANALYSES

- 3 TMA / Dilatometers (vertical, horizontal, optical / various atm.)
- 2 thermal diffusimeters (up to 1500°C)
- TGA/DTA/DSC (up to 2400°C, various atm. including H₂O, coupled with quadrupolar MS)
- 2 thermal diffusimeters (up to 1500°C)
- 1 ICP-OES, 1 He pycnometer
- CHNO elemental analysers
- 1 Laser granulometer
- 2 BET/BJH, chemisorption

SPECTROSCOPY and OPTICS

- 1 FT-IR (solids or liquids, 50-4000 cm⁻¹)
- 1 Spectrophotometer UV-near IR
- 1 Spectrofluorometer UV-near IR (-150°C to 550°C)
- 3 Raman (5-4000 cm⁻¹, up to 800°C, mapping, 1 portable)
- 1 Ellipsometer (UV-near IR)
- 1 XPS (-150°C to 600°C)

THERMAL TREATMENT MECHANICAL PROPERTIES

- Furnaces for natural sintering (various atm., vacuum, up to 2200°C & large samples)
- 1 HP (1800°C/4.5 tons), 1 HIP (2000°C/2000 bars with in-situ dilato.), 1 SPS (φ 100mm, high pressure cell), 1 arc furnace
- 3 Traction/compression equipment (3/4 points bending, up to 1800°C, image correlation)
- 2 Indentors (micro/nano, room T°C, friction)
- Ultrasound characterizations (high T°C elastic modulus, echography, defect mapping)

SUSPENSIONS RHEOLOGY

- 1 Turbidity meter
- 1 Zetasizer (DLS)
- 1 Cordouan (DLS, suspensions up to 40%)
- 1 Acoustophorometer
- 3 Rheometers (plan/plan, cone/plan, couette)
- 1 Capillary rheometer
- 1 Rheometer (up to 600°C)

HR-TEM



ATOMIC PROBE

HR-NMR



HEAVY IONS



NEUTRONS



CLSM

MICROPROBE



X-RAY TOMO

SYNCHROTRON



Thanks!

Any questions?

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