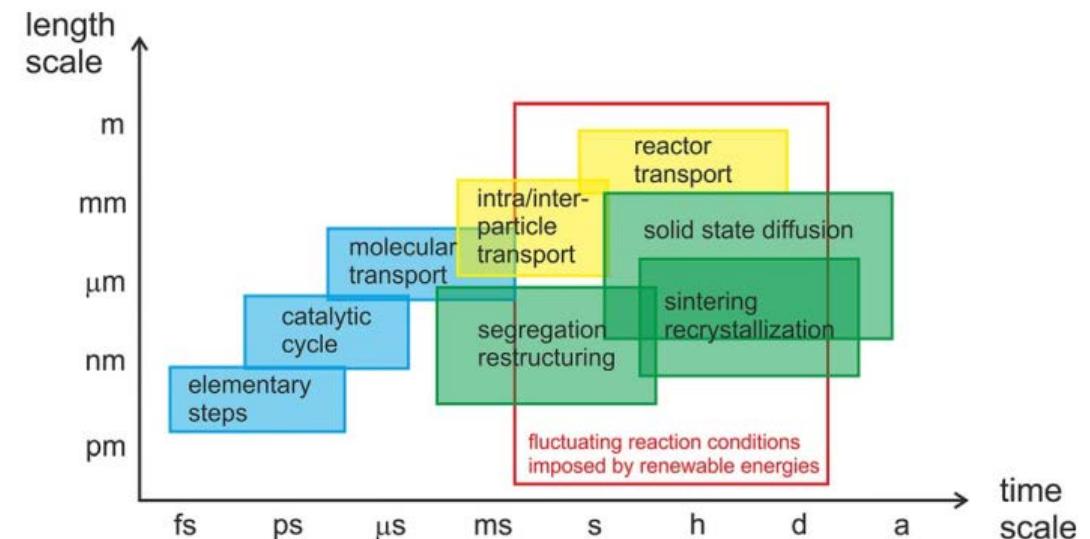


## Panorama de la caractérisation des matériaux

### Exemple matériaux pour la catalyse hétérogène

Du réacteur industriel à l'espèce active

### Changement d'échelle spatiale

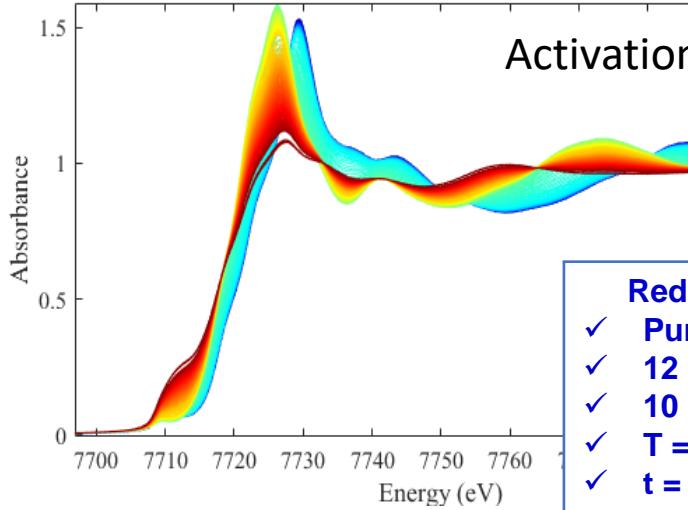


### Changement d'échelle temporelle

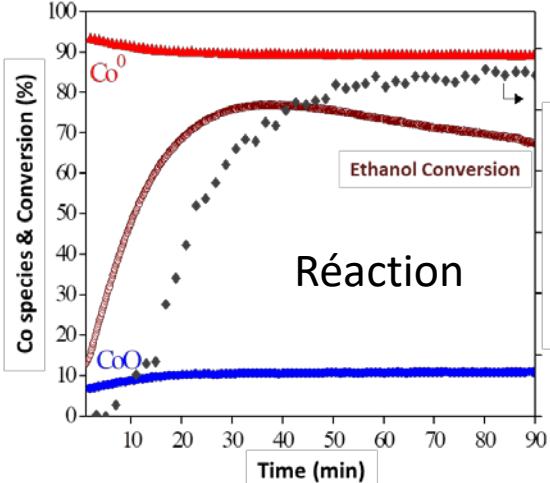
*ChemCatChem* 2017, 9, 17–29

## Description Macroscopique

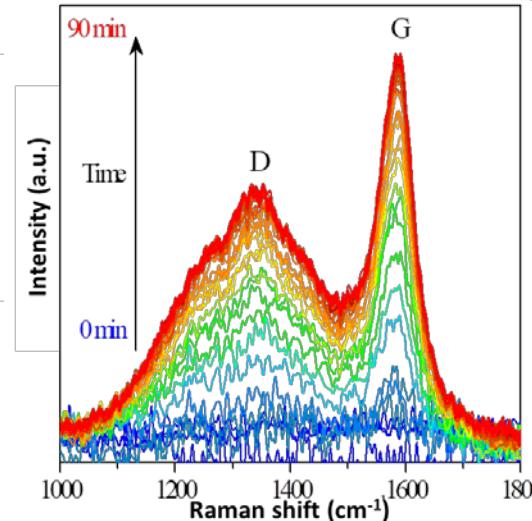
Co/Al<sub>2</sub>O<sub>3</sub> ESR



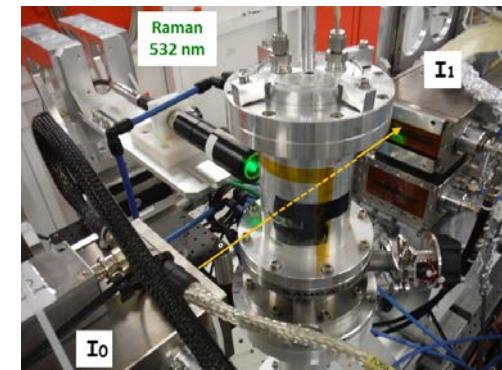
Spectroscopies / Mesure des produits



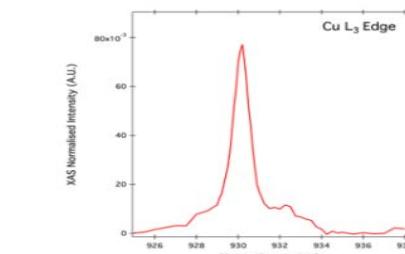
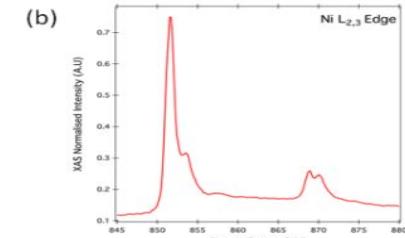
Désactivation



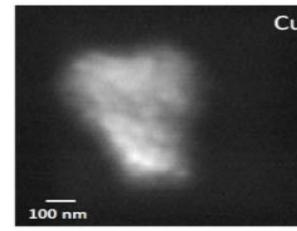
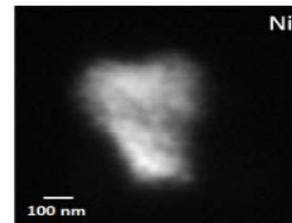
Activation  
Operando  
Désactivation  
Régénération



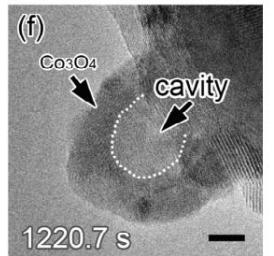
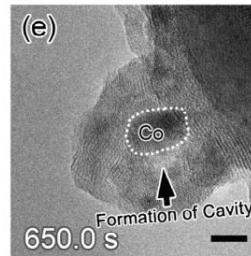
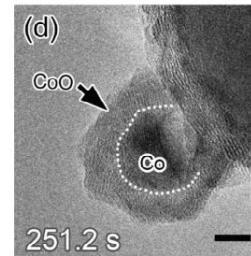
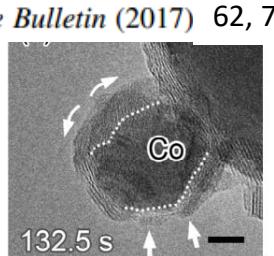
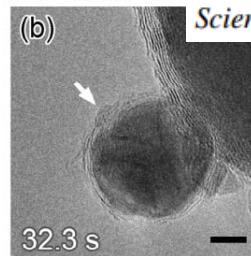
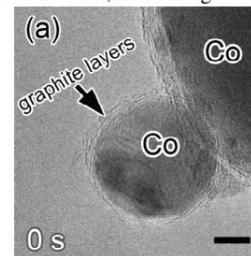
NiCu/Al<sub>2</sub>O<sub>3</sub> ESR



Description Microscopique



Science Bulletin (2017) 62, 775



TEM/O<sub>2</sub> 300°C

Microscopie environnementale

# Multivariate Analysis of Coupled Operando EPR/XANES/EXAFS/UV-Vis/ATR-IR Spectroscopy: A New Dimension for Mechanistic Studies of Catalytic Gas-Liquid Phase Reactions

Jabor Rabeh,<sup>\*[a]</sup> Valérie Briois,<sup>\*[b]</sup> Sven Adomeit,<sup>[a]</sup> Camille La Fontaine,<sup>[b]</sup> Ursula Bentrup,<sup>[a]</sup> and Angelika Brückner<sup>\*[a]</sup>

*Chem. Eur. J.* 2020, 26, 7395 – 7404

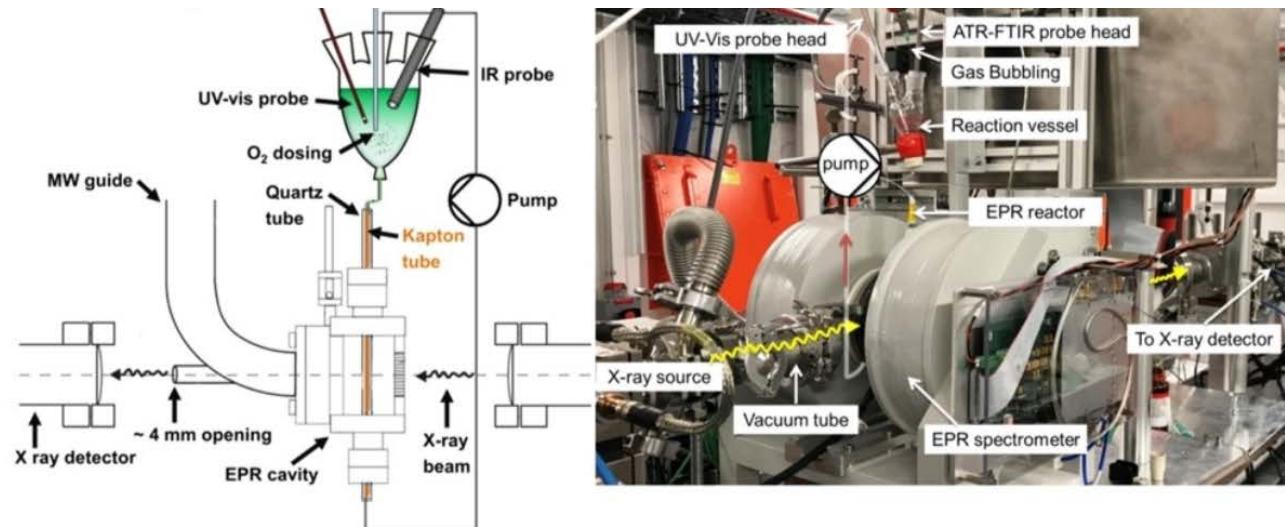
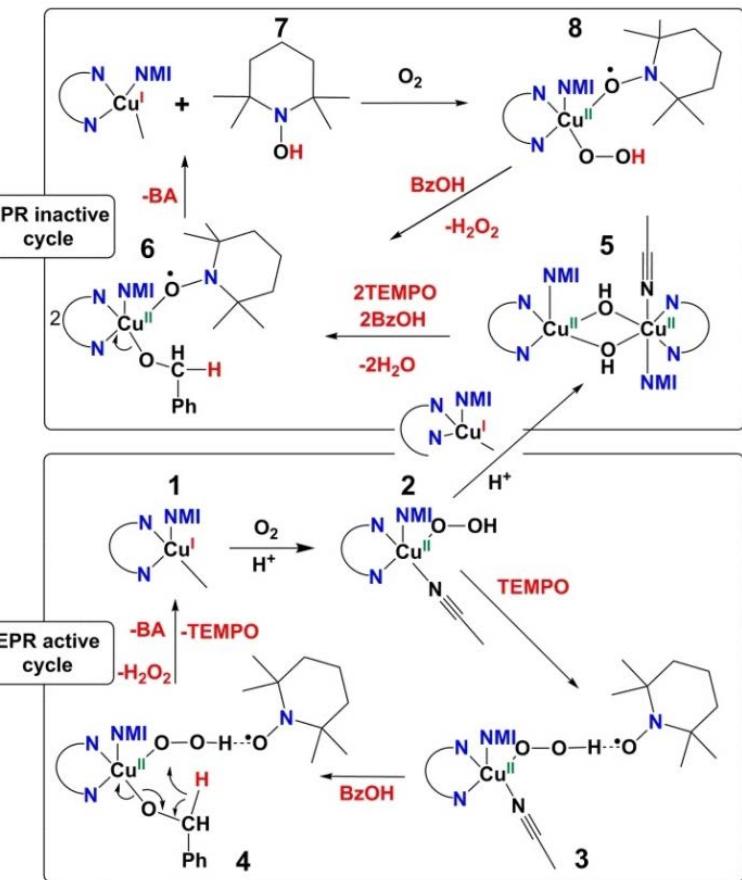
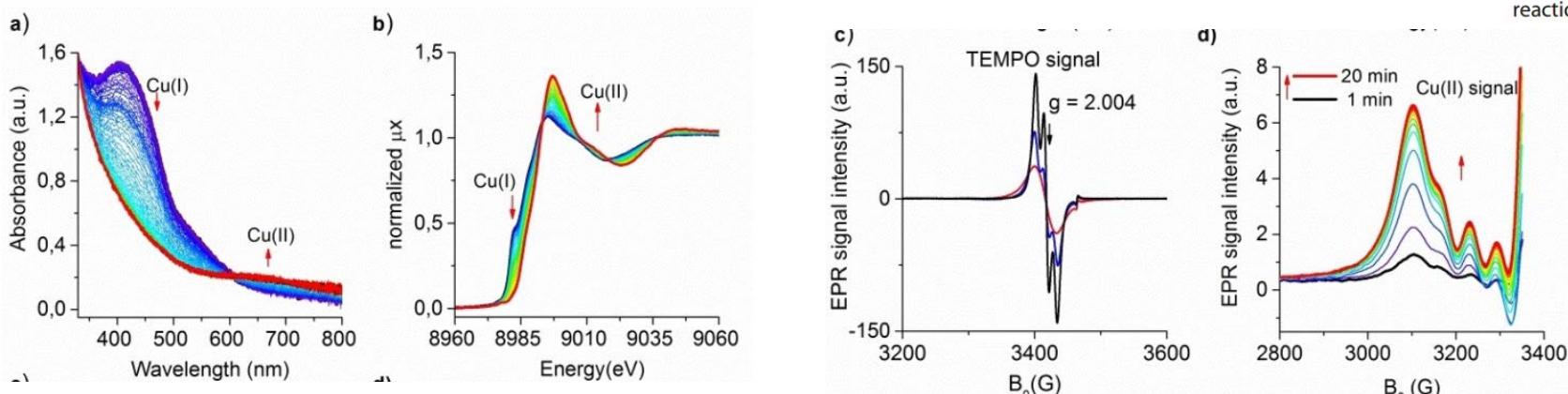


Figure 1. a) Scheme of the operando EPR/XAS/UV-Vis/ATR-IR experimental setup; b) Picture of the setup at the ROCK beamline of the synchrotron facility SOLEIL (Paris).



Scheme 1. Pathway for the formation and cleavage of the Cu<sup>II</sup> dimer during reaction.

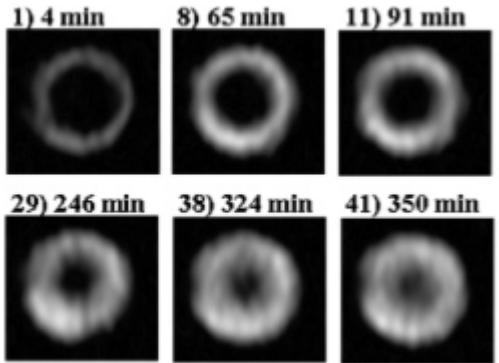
Combinaison de techniques  
Analyse Multivariée

**2005**

**$^{31}\text{P}$  MRI**

$172 \times 371 \mu\text{m}^2$

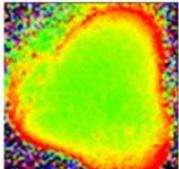
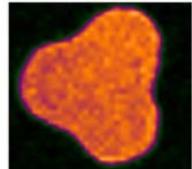
500 s/image



**Transport: Diffusion**

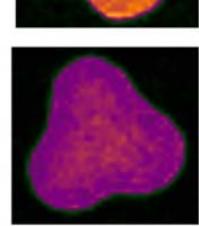
MRI –  $T_1$  contrast  
(Spatial resolution:  
 $39 \times 39 \mu\text{m}$ )

Streamline Raman  
(Spatial resolution:  
 $16 \times 16 \mu\text{m}$ )



**2017**

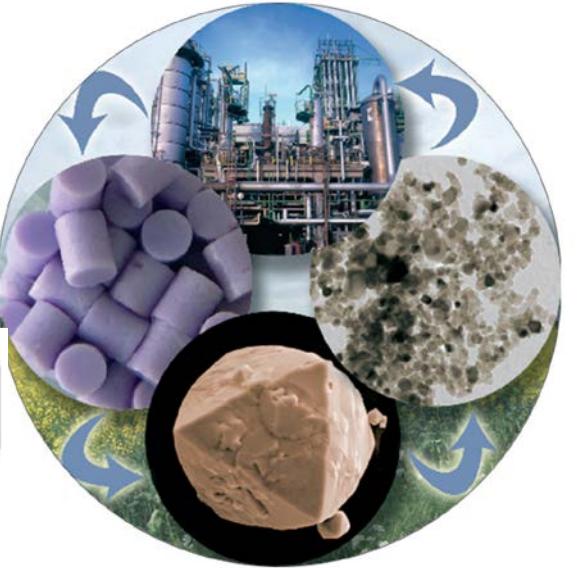
MoP/  
 $\gamma\text{-Al}_2\text{O}_3$



$^1\text{H}$  NMR  
signal

$\text{Mo}_7\text{O}_{24}^{6-}/\text{MoO}_4^{2-}$   
(Ratio of intensities 940/920  $\text{cm}^{-1}$ )

**Elaboration**



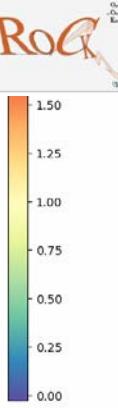
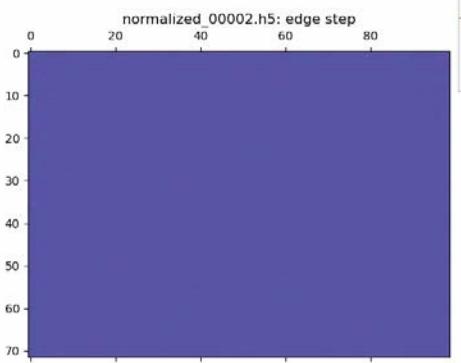
**Speciation**

**2021**

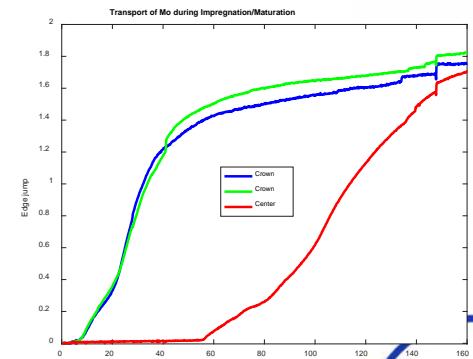
**Mo K**

$16 \times 16 \mu\text{m}^2$

11 s/image

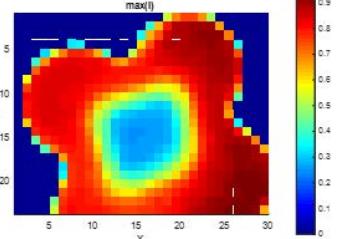


**Transport : Diffusion  
Adsorption/Desorption**

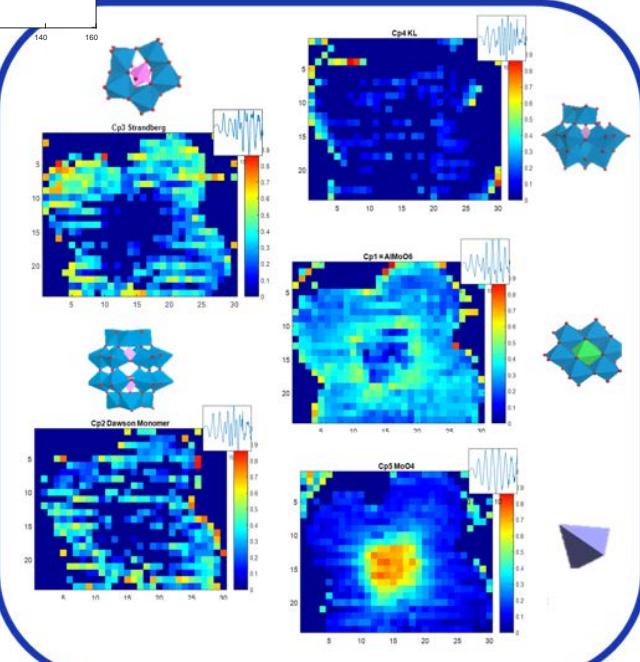


**Wet sample**

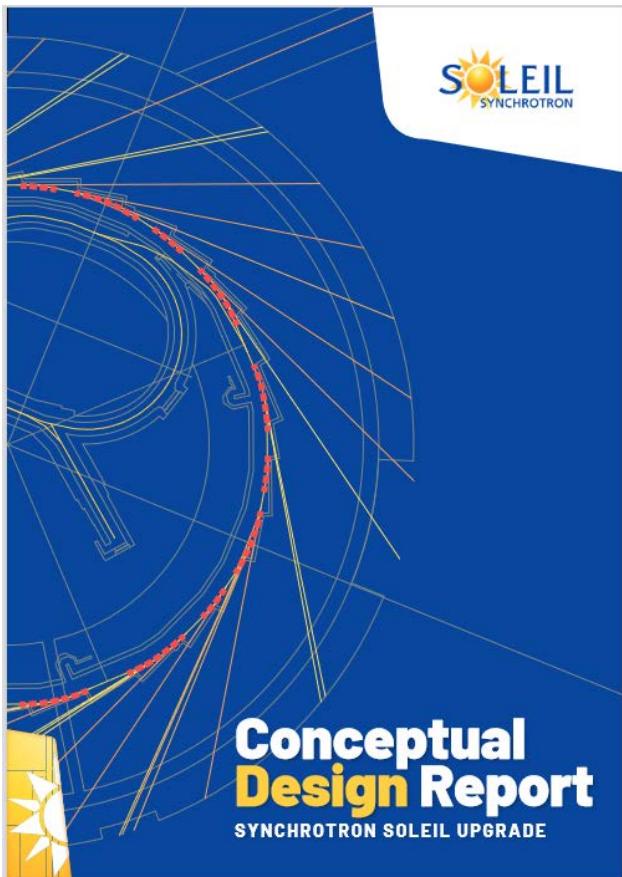
**Wet sample  
Absorption map**



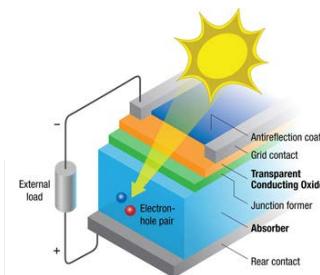
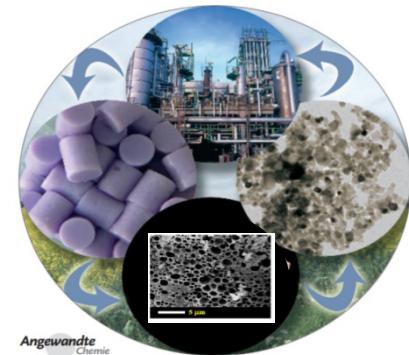
Spatial resolution:  $30 \times 30$  pixels  
 $50 \mu\text{m} \times 50 \mu\text{m}$



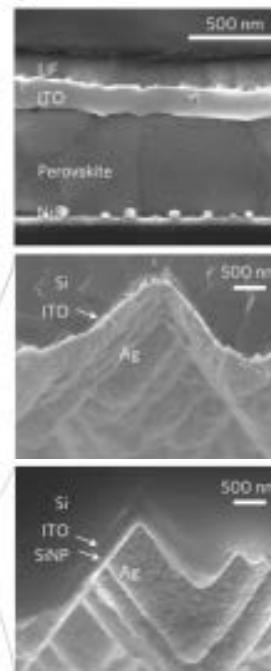
# Materials for Energy Production, Conversion or Storage: Complex Heterogeneous Materials where the Role of Interfaces is Crucial vs Applications



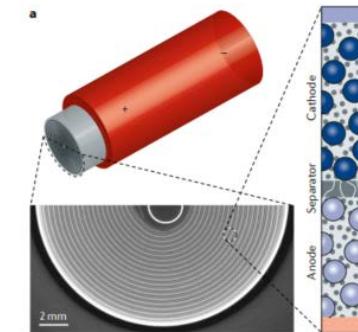
## Heterogeneous Catalysts



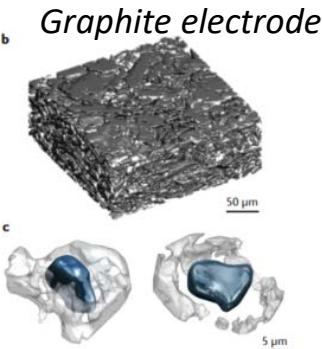
## Photovoltaic Devices



## *Commercial cell*



## Li-ion Batteries



## To tackle the Challenges of Sustainable Energy Technologies:

- ❖ Multi length Scales Imaging from nm to μm - large FoV - elemental composition
  - ❖ Time resolved *Operando* Characterization
  - ❖ Various Characteristic Times from ps to s, min or .... more
- Integrative Approach with Leading Research Groups to develop Cutting-Edge Research

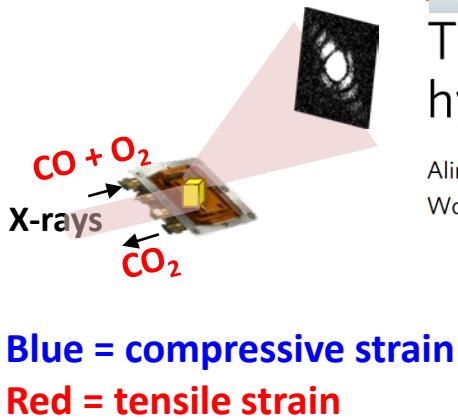
Au/TiO<sub>2</sub>

## Bragg Coherent Diffraction Imaging



Three-dimensional strain dynamics govern the hysteresis in heterogeneous catalysis

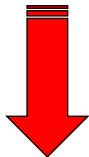
Aline R. Passos<sup>1✉</sup>, Amélie Rochet<sup>1✉</sup>, Luiza M. Manente<sup>1</sup>, Ana F. Suzana<sup>1,2</sup>, Ross Harder<sup>3</sup>, Wonsuk Cha<sup>3</sup> & Florian Meneau<sup>1</sup>



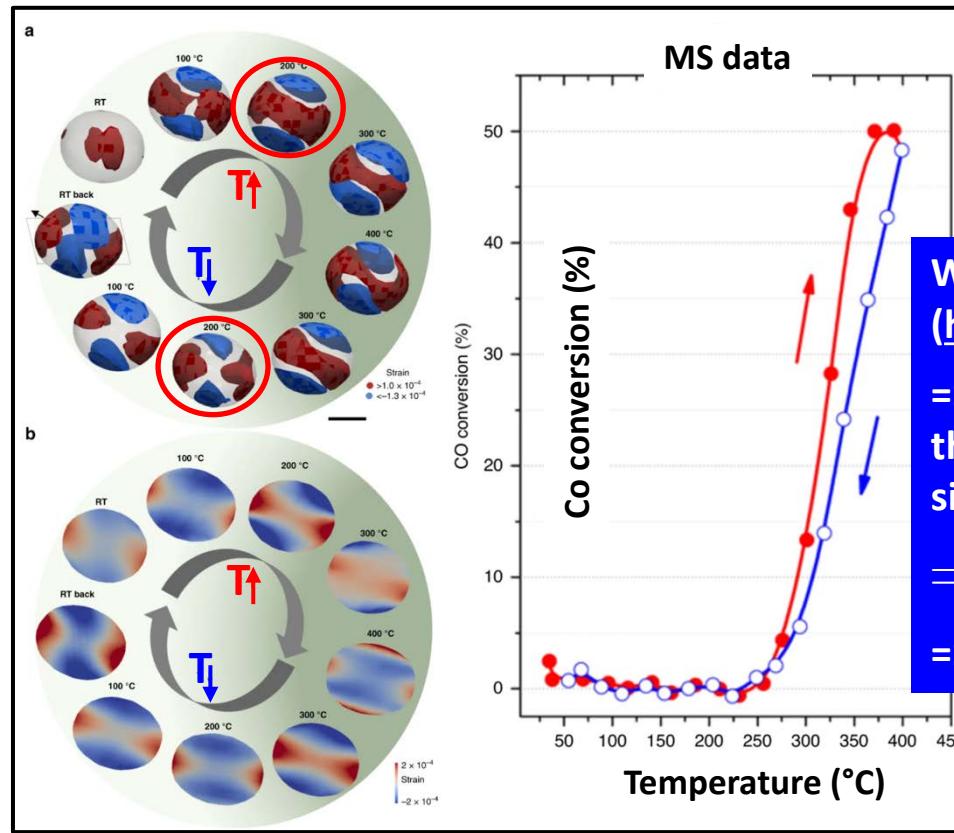
Blue = compressive strain  
Red = tensile strain

APS – Spatial Resolution 15 nm  
– 35 min / pattern

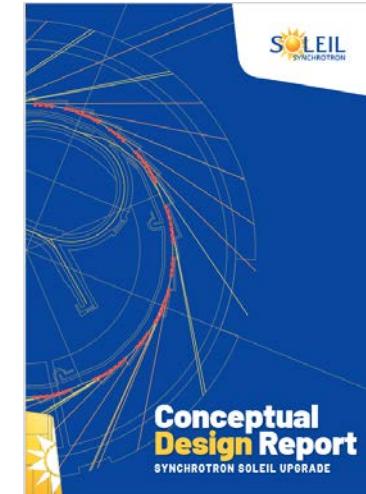
68 nm NP size



SOLEIL-U – Spatial Resolution 1-2 nm –  
30 sec or less/ pattern  
5 to 10 nm NP size



With the design of catalytic reactors  
(high pressure and high temperature)  
=> *Operando* characterization rather  
than *in situ* measurements of relevant in  
size catalysts  
=> Statistical evaluation of a lot of NPs  
=> Experimental data for DFT simulation



9h00	Microscopie X au Synchrotron pour l'étude de matériaux complexes et expériences operando Rachid Belkhou (Synchrotron SOLEIL, France)	<u>Techniques:</u>
9h30	Explorer le nanomonde avec la microscopie électronique in-situ avancée Christian Ricolleau (Université de Paris, CNRS, Laboratoire des Matériaux et Phénomènes Quantiques, France)	<i>Résolution spatiale</i>
10h00	Microscopie électronique en temps réel et corrélative pour l'étude de la matière : naissance, vie et transformation des nanomatériaux Ovidiu Ersen (Université de Strasbourg, CNRS, Institut de Physique et de Chimie des Matériaux de Strasbourg, France)	<i>Résolution Temporelle</i>
10h30	Quelques perspectives dans l'étude expérimentale du transport colloïdal en milieu poreux complexe Pierre Levitz (Sorbonne Université, CNRS, Physicochimie des Electrolytes et Nanosystèmes Interfaçaux, France)	<i>Sensibilité</i>
11h00	Pause-café	
11h30	Apport de la RMN du solide à la caractérisation de matériaux fonctionnels Franck Fayon (Université d'Orléans, CNRS, Laboratoire Conditions Extrêmes et Matériaux : Haute Température et Irradiation, France)	<i>Analyse de Données Et le BiG Data</i>
12h00	Avancées récentes en RMN hyperpolarisée et RMN paramagnétique du solide pour l'investigation de matériaux fonctionnels Anne Lesage (Université Claude Bernard Lyon 1, CNRS, Centre RMN à très Haut Champ, France) et Andrew Pell (Université Claude Bernard Lyon 1, ENS de Lyon, Centre RMN à très Haut Champ, France)	<i>Domaines d'Applications</i>
14h00	Apports de la RMN à l'étude des transferts d'eau dans les piles à combustible à membrane échangeuse de protons Jean-Christophe Perrin (Laboratoire LEMTA, CNRS, Université de Lorraine, France)	<i>Limitations</i>
14h30	Avancées récentes en spectroscopie RPE pour la caractérisation des matériaux Hervé Vezin (Université de Lille, CNRS, LASIRE - Laboratoire Avancé de Spectroscopie pour les Interactions, la Réactivité et l'Environnement, France)	<u>Modélisation:</u>
15h00	Etat des lieux et les perspectives de la spectroscopie vibrationnelle pour les matériaux fonctionnels Arnaud Travert (Normandie Univ, ENSICAEN, UNICAEN, CNRS, LCS, France)	<i>Domaines d'Applications</i>
15h30	Pause-café	
16h00	La chimie théorique au service de la caractérisation des catalyseurs hétérogènes Céline Chizallet (IFPEN, France)	<i>Limitations</i>
16h30	Statistical Physics approaches to bridge length and time scales in the modeling of complex porous materials Roland Pellenq (CNRS, George Washington University, EpiDaPo Lab, France)	<i>Résultats de Rêve</i>