

# 3D non destructive characterization of porous materials



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## Advantages of computed microtomography (CMT)

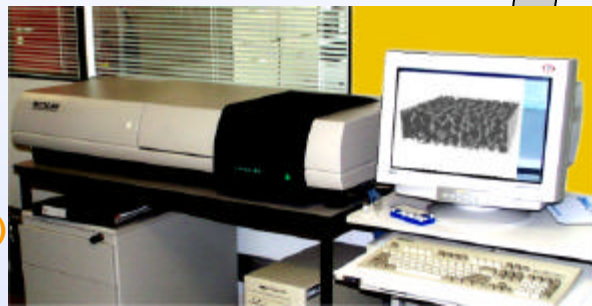
- miniaturization of medical X-ray tomography (desktop equipment)
- no preparation : quick and versatile technique for 3D analysis of micro samples
- well adapted to porous or multiphase material
- quantitative 3D information when coupled with image analysis software
- from global to local information : measure of heterogeneity level

## Catalyst

- silico aluminate support
- high atomic number catalytic phase
- low (catalyst 1) and high (catalyst 2) impregnations
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## Equipment

- Skyscan 1072
- 7  $\mu\text{m}$  resolution at 7 W (5  $\mu\text{m}$  at 5W)
- X9-X110 magnifications
- X-ray energy : 20-100 kV (63 kV, 55 $\mu\text{A}$ , 3.5W)
- 39° fan beam aperture



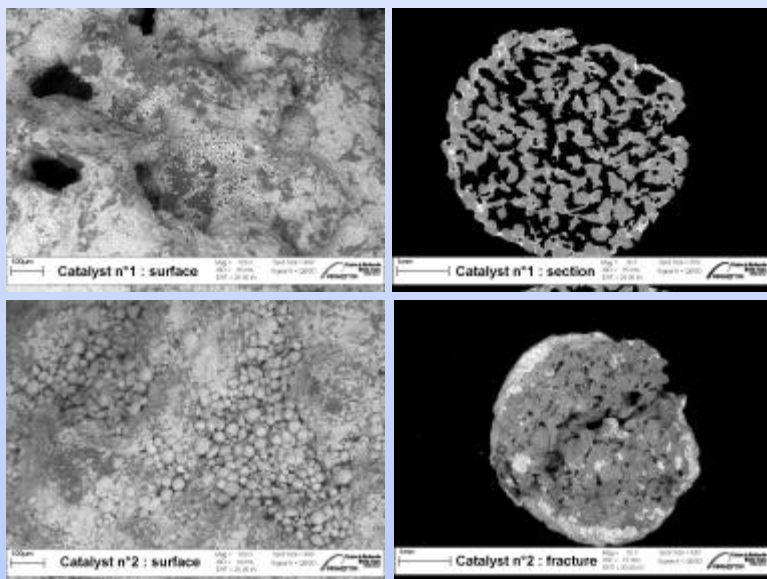
## Image analysis software

- Amiralimage (Noesis-TGS Mercury)
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## Application to porous and multiphase materials

### 2D local analysis

Scanning electron microscopy (SEM)



### 2D image analysis

Catalyst n°1	SEM	CMT
Size <sup>(1)</sup>	???	???
Size distribution spread		
Catalyst vol. / support vol.		
Support	SEM	CMT
Number of images	10	650
Porous mean diameter <sup>(1)</sup>	174 $\mu\text{m}$	174 $\mu\text{m}$
Porous distribution spread	3	3.9
Silico aluminate thickness <sup>(2)</sup>	183 $\mu\text{m}$	170 $\mu\text{m}$
Thickness distribution spread	3	2.85
Porosity <sup>(3)</sup>	37%	?
Porosity standard deviation	8%	?

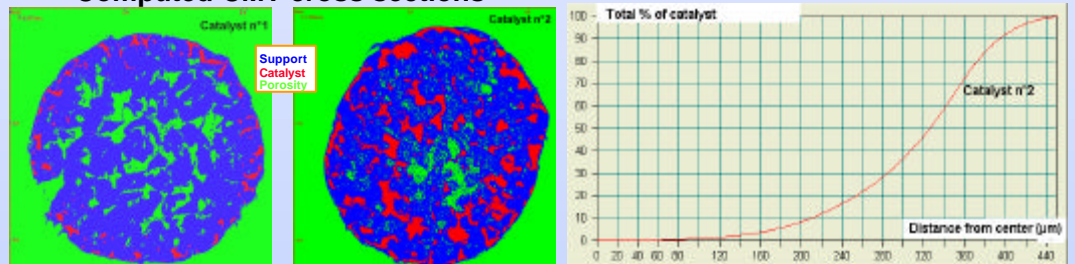
<sup>(1)</sup> Size measurement using iterative openings analysis

<sup>(2)</sup> Thickness measurement using iterative openings analysis

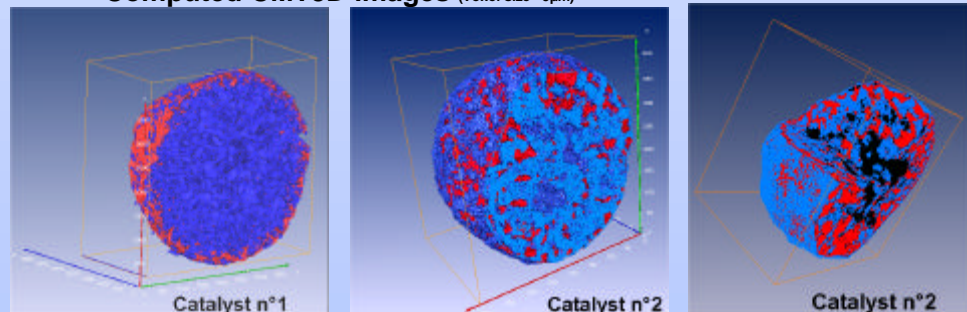
<sup>(3)</sup> Pore surface / support surface

### 3D non destructive analysis

Computed CMT cross sections



Computed CMT3D images (Voxel size = 5 $\mu\text{m}$ )



### 3D image analysis

Support	
Porous mean diameter <sup>(1)</sup>	???
Porous distribution spread	???
Silico aluminate thickness <sup>(2)</sup>	???
Thickness distribution spread	???
Porosity <sup>(4)</sup>	???
Porosity standard deviation	???

Catalyst	n°1	n°2
Size <sup>(1)</sup>	???	???
Size distribution spread		
Catal vol. / support vol.		

<sup>(4)</sup> pore volume / grain volume

### Global analysis

Catalyst	n°1	n°2
Mass impregnation <sup>(4)</sup>	2.5%	?? %
Volume impregnation <sup>(5)</sup>	?? %	?? %
Porosity		

<sup>(4)</sup> Weight measurement before and after impregnation

<sup>(5)</sup> Assuming a specific volume ratio catalyst/support of ???

## Perspectives

- ↪ A 3D characterisation technique for wide material research fields
- ↪ Decrease acquisition / reconstruction time (30 min/ 15 hours)
- ↪ In situ 3D non destructive analysis
  - ✓ Chemical reaction
  - ✓ Mechanical solicitation