

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 μm resolution at 7 W (5 μm at 5W)
- X9-X110 magnifications
- X-ray energy : 20-100 kV (63 kV, 55 μA , 3.5W)
- 39° fan beam aperture

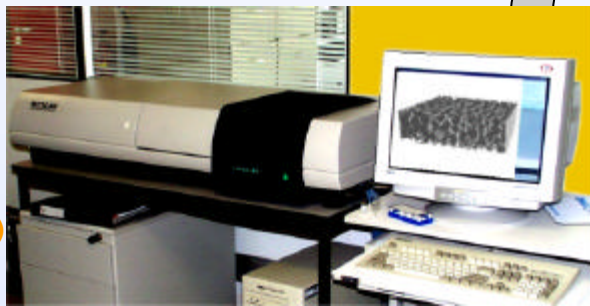


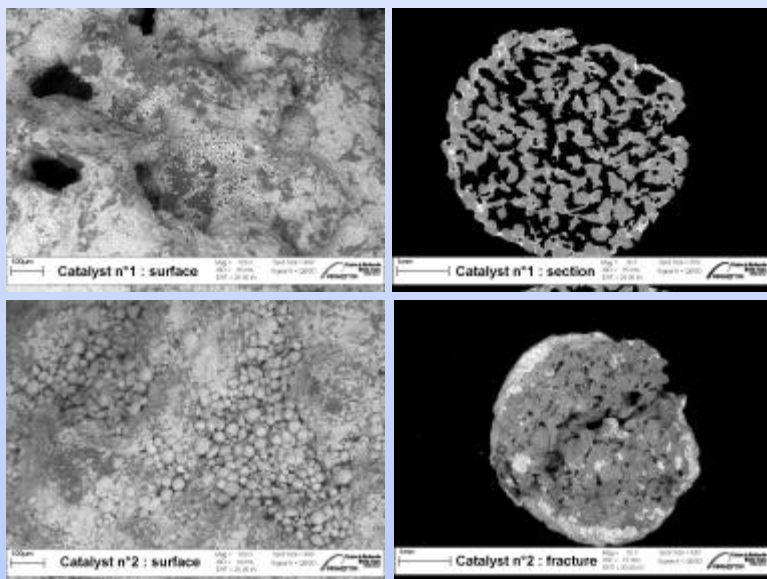
Image analysis software

- Visilog (Noesis)
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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 ⁽¹⁾	???	???
Size distribution spread		
Catalyst vol. / support vol.		
Support	SEM	CMT
Number of images	10	650
Porous mean diameter ⁽¹⁾	174 μm	174 μm
Porous distribution spread	3	3.9
Silico aluminate thickness ⁽²⁾	183 μm	170 μm
Thickness distribution spread	3	2.85
Porosity ⁽³⁾	37%	?
Porosity standard deviation	8%	?

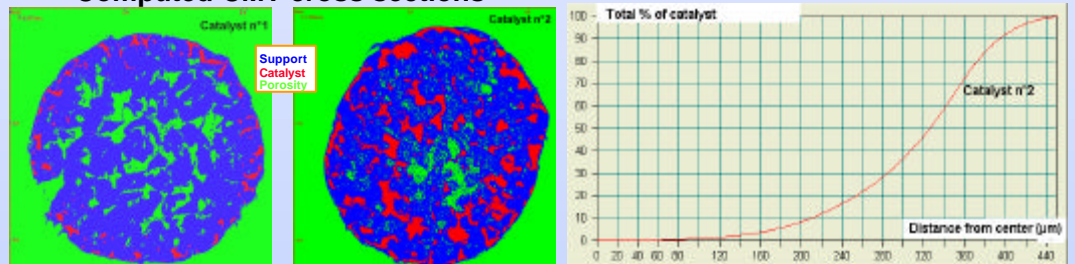
⁽¹⁾ Size measurement using iterative openings analysis

⁽²⁾ Thickness measurement using iterative openings analysis

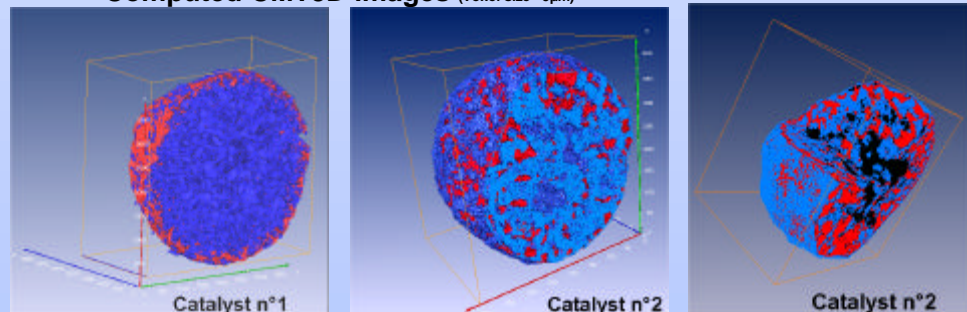
⁽³⁾ Pore surface / support surface

3D non destructive analysis

Computed CMT cross sections



Computed CMT3D images (Voxel size = 5 μm)



3D image analysis

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

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

⁽⁴⁾ pore volume / grain volume

Global analysis

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

⁽⁴⁾ Weight measurement before and after impregnation

⁽⁵⁾ 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