





characterization of silica, focus on statistical size distributions

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Introduction

Materials

Caracterization tools : Usual & AETomography Raw silica results for usual & AET

Aged silica, 2D

Conclusion



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Introduction, material and product



Silica **Particles** within Nanostructured silica (schematic view)

Silica Agglomerates (3D – AETomography) A composite, Optimized packed Vaccum (X tomography)

A SIM

Board or Sealed panel

MATe'B

Particle & Pore imbricated Networks





Introduction, mechanisms

Create a Nanostructure or Confine



Introduction, mechanisms



Bulk density and granular Compacity always matter

Antagonism in WANTED properties











Highly porous Architecture

on at least 3 scales

1. Nano particles.....diameter 15nm

2. Agglomerates......200nm

3. Microscopic GrainS.....10µm

→..COMPOSITE Super Insulation System





Precipitated Sample

Fumed Sample

Same nature but ≠ Si surfaces

Mass production available

Used in low Vf in many Applications (transportation, health...)

Barely used as DURABILITY & EFFICIENCY Leader

Candidates for SI due to their x Scale nanoporosity

Ageing should be considered

WANTED : Pore and Particle Size Distributions







MATER Microstructure analysis



E. Maire, J Adrien, C. Petit CR physique 2014







MATER Microstructure analysis, GLOBAL

Global methods

Sorption (He, Ar, N, H2O) or Intrusion (Hg, water,..) + Modeling



Preparation STEPs :

Pressure, Drying, Dispersion....

Mechanisms are overlapping :

Shrinkage and surface measurement for BET

Mechanical collapse and Intrusion for poro. Hg

High specific surfaces imply artefacts

Efficiency CONFIRMED,

For PORE SD if methods are coupled







MATE Microstructure analysis, 3D

CI YM

ETEM + EDS. EELS

RCELYDN

Recently developed :

High image resolution (nm and µm)

Numerical treatment of 3D volume

No pretreatment required :

Raw and aged could be qualify

To be confirmed:

Ability to image SilicaS (Beam sensitive)





Ag Particles on silicalite surface Wall 328 images 3'54'' Tilt angle -78° et 38.5°





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Raw silica, global measurements

Hg pressure : mechanical test + intrusion



Raw silica, global measurements

Hg pressure : mechanical test then intrusion







Silica 81images 1'10 2048x2048 pixel Tilt angle -71° et 71°





AET tomography, projections Prec. Sample Fumed Sample



Silica particles are viewed in black

50 nm

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A. Perret ,MATeB thesis

XY projections issued from a reconstructed volume,

enough contrast no artefact for both Silica

Particles and pores are observed



AET tomography, projections Prec. Sample Fumed Sample



Silica particles are viewed in black



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XY projections issued from a reconstructed volume,

enough contrast no artefact for both Silica

Each projection has its own structure



A. Perret ,MATeB thesis



Reconstructed volume, Silica particles (Green) only are viewed

A few transverse pore are observed + free branch







AET tomography, volumes



Reconstructed volume, Silica particles (Green) only are viewed

Network features : Size, morphology, connection ...

3D "Particles" and Pore characterized







AET tomography, distribution



AET tomography, distribution





Introduction

Materials Caracterization tools : Usual & AETomography Raw silica results for usual & AET Aged silica 48°C , results for usual + 2D

Conclusion







Aged silica, global measurements

Hg pressure : mechanical test + intrusion



Aged silica, fast 2D imaging

48°C 65% RH





No beam damage, AET feasible Transverse pores, particles, free arms observed





Aged silica, fast 2D imaging





No beam damage, AET feasible

Transverse pores size

Particles size



, free arms not observed









50°C 90% RH

Conclusion and perspectives

✓ Electronic tomography

DES SCIENCES APPLIQUÉES

- Advanced protocol developped,
- 3D pores + particule Volume observed
- Data within a fair computation time
- 2 samples evaluated : Precipitated + Fume
- ✓ Experimental SD towards Thermal modelling Pore SD were used to compute
 Particle SD should be used to
- ✓ Electronic tomography versus Global measurements
 Complementary tools, towards new synthesis

IVIS, Nanjing 09-2015, G. Foray

✓ Perspectives : Aged AET Silica, new products...





Thanks for attention...any Question ?



Acknowledgement : ARC ENERGY Cluster, ADEME, METSA, CLYM







AET tomography, projections **Prec. Sample Fumed Sample** 50 nm 50 nm Silica particles are viewed in black XY projections issued from a reconstructed volume, enough contrast no artefact for both Silica Particles and pores are observed A. Perret .MATeB thesis IVIS, Nanjing 09-2015, G. Foray MATe'B

Raw silica, global measurements Hg test versus N adsorption







AET tomography, distribution MATe'B



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Aged silica, global measurements

Hg pressure : mechanical test + intrusion







Given an industrial process, many parameters :

Reactant

pН

temperature

Pressure...

Given an application, many requirements

Thermal conductivity, Mechanical ppties,

Durability, Aesthetic, Fire, Confort,

Tremendous synthesis possibilities, how to **EVALUATE** ?

Microstructure Versus Functionnal properties,





