Emerging technologies with intense electromagnetic fields and plasma

National Institute for Laser, Plasma and Radiation Physics

for energy, life sciences, environment, communications and security

http://www.inflpr.ro

http://tomography.inflpr.ro

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Motivation

X-ray tomography is an imaging technique for non-invasive volumetric characterization of materials and processes

It can be used in optimization of processes of waste valorization as:
- recycling & resource recovery (ex. rare earths, tungsten);
- pelletization of coal ash or fly ash resulted from solid waste incinerators;
- production of composites from waste recycled armor materials and natural matrix (ex. volcanic ash, mortar);
- characterization of waste recycled glass/textile fibers to be used in composites;
- production of ultra-light composites used as building materials;
- characterization of wood-plastic composites;
- advanced characterization and modeling of porous materials (ex. charcoal pellets) ...

It could provide a unique access channel for a fully non-invasive inspection and quantitative analysis of some hazardous waste.

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Applications of X-ray microtomography in microstructural analysis of materials resulting from waste processing

Outline

• Tomography equipment
• Porosity analysis & fluid transport in porous media
• Passive treatment to remediate contaminated water from acid mine drainage
• Tomography analysis of fly ash pelletization process
• Volumetric analysis of composite materials based on waste of metal or wood processing
• Geological CO2 storage

http://tomography.inflpr.ro
Tomography Equipment

Submicron resolution

High penetration power microfocus @ 320 kV
### Technical data of various XCTs

<table>
<thead>
<tr>
<th>Type</th>
<th>X-ray source</th>
<th>Voxel size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Medical XCT-systems</td>
<td>Med-XCT</td>
<td>&gt; (0.3 mm)$^3$</td>
</tr>
<tr>
<td>Cone beam XCT:</td>
<td>μXCT</td>
<td>&gt; (2 μm)$^3$</td>
</tr>
<tr>
<td>Rayscan 250E or v</td>
<td>tome</td>
<td>x s 240</td>
</tr>
<tr>
<td>Cone beam XCT:</td>
<td>Sub-μXCT</td>
<td>&gt; (0.4 μm)$^3$</td>
</tr>
<tr>
<td>nanotom 180</td>
<td></td>
<td></td>
</tr>
<tr>
<td>INFLPR NanoCT</td>
<td>Sub-μXCT</td>
<td>&gt; (0.5 μm)$^3$</td>
</tr>
<tr>
<td>INFLPR XCT</td>
<td>μXCT</td>
<td>&gt; (2 μm)$^3$</td>
</tr>
<tr>
<td>Synchrotron XCT:</td>
<td>sXCT</td>
<td>&gt; (10 μm)$^3$</td>
</tr>
<tr>
<td>Grenoble, ESRF-ID19</td>
<td></td>
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</tbody>
</table>

X-ray microtomography

Equipment for X-Ray microtomography analysis and compositional mapping

Four versatile tomography units designed and constructed in INFLPR with energies from 50 to 320 keV and sub-micron feature recognition. Wide variety of applications with samples sizes from 5 m down to 100 µm.

Carbon Fiber Composite

3D targets for high power laser interaction

X-Ray Microtomography analysis of superconductor strand & cables

Medical devices
Berea sandstone multi-resolution analysis

Φ = 38 mm

Φ = 5 mm

mini-core
Berea sandstone $\Phi = 5 \text{ mm}$ - pore analysis

Porosity classification by volume, area, shape, connectivity etc.
Berea sandstone $\Phi = 5 \text{ mm}$ – inclusions analysis

Inclusion analysis:
- volume, area, shape,
- density, composition etc.

**Material**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material volume [mm$^3$]</td>
<td>22.7257</td>
</tr>
<tr>
<td>Defect volume [mm$^3$]</td>
<td>0.2432</td>
</tr>
<tr>
<td>Defect volume ratio [%]</td>
<td>1.06</td>
</tr>
</tbody>
</table>
Berea sandstone 2 mm - submicron pore analysis

3D representation of all pores from reconstructed volume

ROI – magnified inner pores in 3D
Capillary pressure simulation $\Phi=6\ mm$ – wetting phase

2D visualisation of trapped wetting volume

3D visualisation of wetting phase trapped in all volume

3D visualisation of trapped wetting phase

3D visualisation of isolated pore space
Passive treatment systems designed to remediate contaminated water from acid mine drainage

Four CT images of the same section: before drainage; after 4, 8 and 12 days (passivation at ≈300 h).
Passive treatment systems designed to remediate contaminated water from acid mine drainage

Rocks and mineral grain filter (such as calcite, aragonite or dolomite) with size grain between 1-2 mm
Passive treatment systems designed to remediate contaminated water from acid mine drainage

Directional variability

Grain orientation on right view sections

Grain orientation on top view sections
Tomography analysis of fly ash pelletization process

Core carbonation dependent on the reaction time
Tomography analysis of fly ash palletization process

Core analysis

ROI selection

Surface determination on selected ROI

Extracted ROI with surface determination

Extracted core volume: 16.04%

Extracted core

Extracted core volume: 32.92%
Tomography analysis of fly ash pelletization process
Porosity analysis
Tomography analysis of fly ash palletization process

Porosity analysis

<table>
<thead>
<tr>
<th>Material</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material volume [mm³]</td>
<td>22409.8594</td>
</tr>
<tr>
<td>Defect volume [mm³]</td>
<td>44.6834</td>
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<tr>
<td>Defect volume ratio [%]</td>
<td>0.20</td>
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</tbody>
</table>
Tomography analysis of fly ash palletization process

Inclusions analysis
Composition mapping by microbeam X-ray fluorescence microXRF

Standardless procedure for elemental composition
Elemental composition of fly ash pellets by microXRF

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>1.92</td>
</tr>
<tr>
<td>Cl</td>
<td>8.54</td>
</tr>
<tr>
<td>K</td>
<td>5.57</td>
</tr>
<tr>
<td>Ca</td>
<td>76.11</td>
</tr>
<tr>
<td>Cu</td>
<td>0.45</td>
</tr>
<tr>
<td>Zn</td>
<td>3.23</td>
</tr>
<tr>
<td>Pb</td>
<td>0.65</td>
</tr>
<tr>
<td>Ti</td>
<td>2.74</td>
</tr>
<tr>
<td>Br</td>
<td>0.79</td>
</tr>
</tbody>
</table>

Inclusions on pellet surface
Volumetric analysis of waste based composite materials
volcanic ash & metallic insertions

Composite material made by volcanic ash (matrix) and metallic swarf (insertions)

Selected ROI

Total volume of metallic insertions from selected ROI

<table>
<thead>
<tr>
<th>Values (grid coordinate system)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min.:</td>
</tr>
<tr>
<td>Max.:</td>
</tr>
<tr>
<td>Mean:</td>
</tr>
<tr>
<td>Deviation:</td>
</tr>
<tr>
<td>Volume [mm³]</td>
</tr>
<tr>
<td>Number of voxels:</td>
</tr>
<tr>
<td>Between cursors [%]:</td>
</tr>
</tbody>
</table>
Volumetric analysis of waste based composite material foam matrix & wood fibers

Space resolution 30 µm/voxel
Top: wood to matrix ~ 8.25%
Bottom: wood to matrix ~ 7.0
The reservoir rocks are composed of limestone (calcite) and sandstone (66 wt.% calcite, 28 wt.% quartz and 6 wt.% microcline).

CO2-rich acid brine will likely promote the dissolution of carbonate minerals (calcite) and aluminosilicates (microcline).

These coupled dissolution and precipitation reactions may induce changes in porosity and pore structure of the repository rocks.