Measurement of thin oil layers by PIXE - α

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原则的 PIXE - α 方法

PIXE analysis of the silver coating of a Roman coin
(III century BC)
Characterisation of the blue pigments of Madonna del Roseto
(Luca della Robbia, XV century
Florence, Italy)

Analysis of the black pigment

General advantages of the method

• Non destructive analysis
• Analysis of light elements (Z < 26)
• Surface analysis (some mm)
• Samples analysis with dimensionnal or administrative constraints
• Field equipment
PIXE - α adaptation to thin layer thickness measurement principle

Radioactive source specifications

- Pure α emission (>>> $^{210}$Po )
- High activity (37MBq)
- Annular geometry
- Source: sealed but thin
- Resistance to radiolysis

$^{210}$Po desintegration scheme

$T = 138.4$ years

- $210$Po
- $a_1 = 5.3$ MeV
- $a_2 = 5.3$ MeV
- $(90.09\%)$
Po 210 radiological and chemical safety

- Chemical safety:
  - Very low level of the toxicity limit $4 \times 10^{11}$ mg/m$^3$
  - Polonium compounds are very volatile

- Radiological safety (annual incorporation limit)
  - Workers: $2 \times 10^4$ Bq
  - Public: $2 \times 10^3$ Bq

From Journal Officiel des Communautés Européennes (JOCE, 1980)

Chemical reaction for source preparation

Spontaneous electrochemical replacement (Haïssinsky, 1937)

\[
\begin{align*}
\text{Po}^{2+} + 2e & \rightarrow \text{Po} \\
(\text{Ag} \rightarrow \text{Ag}^+ + e) \times 2 \\
\text{Po}^{2+} + 2\text{Ag} & \rightarrow 2\text{Ag}^+ + \text{Po}
\end{align*}
\]

Electrodeposition cells
Sources socket MYLAR / Silver (0.2 mm)

Annular truncated concave source

Annular source

Confined barrier:
- Layer of aromatic epoxy resin (e < 0.5 µm)
- KAPTON window (e = 2 µm)
- NYLON grid (mechanical protection)

Po Source preparation – deposition kinetics

Po deposition yield vs contact time

210Po Source ready to use
Technological transfer from CEA to INFN

Experimental program

- Objectives:
  - Influence of the thickness of demoulding oils on the aesthetic quality of concrete facings
  - Studies on the influence of oils deposition method on film thickness

- Materials:
  - 2 types of concrete
  - 2 types of oils

- Oil deposition methods:
  - Spraying with conic nozzle at 20 cm from surface
  - Spread after spraying with a rubber scraper
Studied materials

Concrete composition

<table>
<thead>
<tr>
<th>Properties</th>
<th>Sand (4/8)</th>
<th>Gravel (0/4)</th>
<th>Sand (0/4)</th>
<th>Cement (CEM I 52.5)</th>
<th>Water</th>
<th>E/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (kg/m³)</td>
<td>2688</td>
<td>258</td>
<td>206</td>
<td>826</td>
<td>187</td>
<td>2.86</td>
</tr>
</tbody>
</table>

Oils characteristics

<table>
<thead>
<tr>
<th>Properties</th>
<th>Vegetable-based oil (V)</th>
<th>Mineral-based oil (M)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/mL)</td>
<td>0.91</td>
<td>0.85</td>
</tr>
<tr>
<td>Color</td>
<td>Dark yellow</td>
<td>Yellow</td>
</tr>
<tr>
<td>Flash point (°C)</td>
<td>&gt; 80</td>
<td>&gt; 90</td>
</tr>
<tr>
<td>Viscosity at 20°C (mPa.s)</td>
<td>0.85</td>
<td>0.93</td>
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<td>0.85</td>
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</tr>
<tr>
<td>Extract angle (°)</td>
<td>52</td>
<td>52</td>
</tr>
</tbody>
</table>

Calibration with mylar disks

<table>
<thead>
<tr>
<th>Mylear Disc</th>
<th>Mass (mg)</th>
<th>Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Disc #1</td>
<td>10.2</td>
<td>3.07</td>
</tr>
<tr>
<td>Disc #2</td>
<td>12.6</td>
<td>3.16</td>
</tr>
<tr>
<td>Disc #3</td>
<td>12.6</td>
<td>3.16</td>
</tr>
</tbody>
</table>

The calculation of each equivalent thickness is done according to the following equation:

Equivalent thickness = Thickness of the disc × density of the mylar / density of the studied oil
an approximate equation of these curves is of the form: \( y = a + bx + cx^2 \).

By fitting on the results we obtain the equations coefficients, thus:

For the mineral oil:
\[
y = 40.59 + 1.33 \times 10^3 x - 9.12 \ln x
\]

For the vegetable oil:
\[
y = 36.93 + 1.17 \times 10^3 x - 8.28 \ln x
\]
Comparison between gauging and weighting results

<table>
<thead>
<tr>
<th>Oil Thickness (µm)</th>
<th>Sprayed film</th>
<th>Scraped film after spraying</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mineral oil</td>
<td>Between 1 to 2</td>
<td>6.0</td>
</tr>
<tr>
<td>Vegetable oil</td>
<td>Between 2 to 3</td>
<td>1.6</td>
</tr>
</tbody>
</table>

Conclusion

- PIXE method really adapted for this kind of measurement
- Range of measurement : 1 to 10 µm
- Film thicknesses are between 1 to 2 µm after scraping sprayed film
- A film of 1 to 2 µm is enough to obtain a high quality facing allowing minimization of oil quantities
- The studied vegetable oil allows obtaining a facing with a better aesthetical quality than the mineral oil
- Many other applications are possible and some have been tested (teflon for ex.)
- Evolution to Curium 244 source (half life,...)

Thank you for your attention