Raman spectroscopy of interfacial transition zone in concrete doped with limestone powder and metakaolin

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Interfacial transition zone (ITZ) in concrete
• Concrete = three-phase composite
• Cement paste + aggregate + ITZ

Schematic diagram of ITZ in concrete with ordinary Portland cement according to Al Bayati et al. (2016), modified.
Raman spectroscopy - principle

Scheme of Raman spectroscopy (Fenn et al. 2011)

Energy-level diagram showing the states involved in Raman spectra

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Introduction to Raman spectroscopy and its use in the research of building materials

- Raman spectroscopy in comparison with other methods (XRD, WDX) represents a non-destructive research method
- Identification of solid, liquid, gaseous substances, gels and pastes
- The laser beam is directed at a specific point in the sample and is also possible to analyze different layers of the sample
- Possibility of measurements in the field without sampling
- Distribution of the phases in the sample
- Study the hydration of cement paste in situ

Disadvantages
- Impossibility to identify black or very dark samples, metals, highly fluorescent materials
- Some phases have too intense Raman signal to drown out the signal of other phases

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**Materials and methods**

Strength characteristics and absorbability were tested after 28 days of curing.

Raman spectroscopy of selected samples was performed on a WITec Confocal Raman Imaging Microscope System alpha300 R + with excitation of 532 nm (power incident on the sample - 35 mW, objectives Zeiss EC Epiplan 20x/0.4 and Zeiss EC Epiplan-Neofluar Dic 50x/0.8, scan bitmap (step) = 1 pixel/µm, integration time at each point = 200 ms. Before the Raman measurement, the selected area was scanned using optical profilometry WITec TrueSurface.

### Mixture proportions for OPC-1, VPC-1 and VP-5; in grams

<table>
<thead>
<tr>
<th></th>
<th>OPC-1</th>
<th>VPC-1</th>
<th>VP-5</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I 52,5 N (Mokrá)</td>
<td>900</td>
<td>450</td>
<td>-</td>
</tr>
<tr>
<td>CEM I 52,5 R (Mokrá)</td>
<td>-</td>
<td>-</td>
<td>855</td>
</tr>
<tr>
<td>Fine-grained limestone</td>
<td>-</td>
<td>180</td>
<td>45</td>
</tr>
<tr>
<td>(Štramberk)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Metakaolin Mefisto K05</td>
<td>-</td>
<td>270</td>
<td>-</td>
</tr>
<tr>
<td>Sand (Bzenec)</td>
<td>900</td>
<td>900</td>
<td>900</td>
</tr>
<tr>
<td>Aggregate (Olbramovice)</td>
<td>900</td>
<td>900</td>
<td>900</td>
</tr>
<tr>
<td>Aggregate (Olbramovice)</td>
<td>900</td>
<td>900</td>
<td>900</td>
</tr>
<tr>
<td>Water</td>
<td>545</td>
<td>710</td>
<td>545</td>
</tr>
</tbody>
</table>
Results

Mechanical properties after 28 days of curing

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Concrete sample OPC-1 A) Bright-field image under the confocal optical microscope; B) Raman image of the ITZ of the sample; C) Sample profile analysed by optical profilometry.
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Sample VPC-1

A) Bright-field image under the confocal optical microscope; B) Bright-field image of the sample underlain by Raman image in the studied area; C) Raman image of studied area of the sample; D) Sample profile analysed by optical profilometry.
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Distribution of portlandite
Distribution of portlandite

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C-S-H is the main hydration product of Portland cement in studied samples

Concrete OPC-1 and VP-5 has higher compressive and tensile strength and lower density and absorbability compared to concrete VPC-1

In concrete with ordinary Portland cement and concrete with limestone powder, the interfacial transition zone has higher thickness and more portlandite is formed in this zone than in the sample with limestone powder and metakaolin

In concrete with the addition of metakaolin and limestone powder, a thin rim of calcite is formed around quartz particles, this can reduce the strength of the concrete

<table>
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<tr>
<th></th>
<th>OPC-1</th>
<th>VPC-1</th>
<th>VP-5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness of ITZ (µm)</td>
<td>20-40</td>
<td>0-10</td>
<td>10-30</td>
</tr>
<tr>
<td>Compressive strength after 28 days (MPa)</td>
<td>52.0</td>
<td>27.0</td>
<td>55.1</td>
</tr>
</tbody>
</table>
References

• Al-Bayati H, Tighe S, Baaj H 2016 Effect of different Treatment Methods on the Interfacial Transition Zone Microstructure to Coarse Recycled Concrete Aggregate. In: Conference of the Transportation of Canada, Toronto pp 1-29

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Thank you for your attention

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