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# Experimental study of the chemo-mechanical properties of the interfacial transition zone of concrete

A Girboveanu<sup>1,3\*</sup>, M Jebli<sup>1</sup>, F Jamin<sup>1,2</sup>, V Huon<sup>1</sup>, M S El Youssoufi<sup>1,2</sup>,  
and D P Georgescu<sup>3</sup>

<sup>1</sup> LMGC, Univ. Montpellier, CNRS, Montpellier, France.

<sup>2</sup> MIST Lab., IRSN, CNRS, Univ. Montpellier, France.

<sup>3</sup> Laboratory of the Department Reinforced Concrete Structures Department – Technical University of Civil Engineering, 124 Bld. Lacul Tei, 020396 Bucharest, Romania

Corresponding author's e-mail: andrei-sorin.girboveanu@phd.utcb.ro

**Abstract.** This article presents an experimental study of the chemical and mechanical properties of the cement paste / aggregate interphase. Interfacial Transition Zone (ITZ) represent the contact zone of the cement paste with the aggregate with a thickness of about 20 - 50  $\mu\text{m}$ . This zone has different mechanical and chemical properties compared to the bulk paste. Cement paste / limestone aggregate composite samples were performed to allow the study of the chemical and mechanical properties of ITZ. The cement paste is made with Portland cement with a water/cement mass ratio of 0.4. Calcium concentration profiles in the ITZ show a high concentration of calcium due to the presence of portlandite and ettringite. The nano-indentation tests indicate weakness of ITZ compared to bulk cement paste.

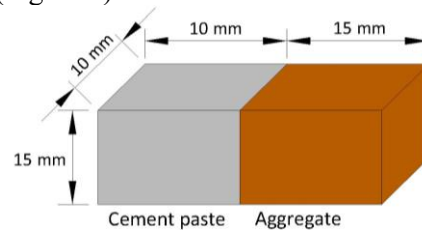
## 1. Introduction

At the end of the hydration process, concrete is composed of aggregates, hardened cement paste and Interfacial Transition Zone (ITZ). The hardened cement paste is formed by the hydration products: the matrix of hydrated calcium silicates (C-S-H) and crystals of portlandite (CH) and ettringite (Afm). Portlandite, as a hexagonal crystals form, is a highly soluble chemical species. Portlandite is found with greater quantity at the ITZ. The ITZ is a cement paste zone in the nearest parts of aggregates, whose thickness varies between 20 and 50  $\mu\text{m}$ . Several phenomena, including the wall effect and micro-bleeding, favour the formation of this zone of higher porosity <sup>1</sup>. As a result, ITZ is characterized by lower mechanical properties than bulk cement paste <sup>2,3</sup>. Nature of the aggregate also influences the chemical structure of this zone. Limestone aggregates, so-called reactive, allow better adhesion than siliceous aggregates<sup>4</sup>. The main objective of this study is to characterize, from a chemical and mechanical point of view, the microstructure and properties of the ITZ.



## 2. Experimental protocol

In this part, we will present the experimental protocol of the realization and the conservation of the samples. Also the methods used for the chemical and mechanical characterization of the ITZ. The composites samples consist of a cement paste and a limestone aggregate, with parallelepiped shape of  $10 \times 10 \times 30 \text{ mm}^3$  total volume (Figure 1).



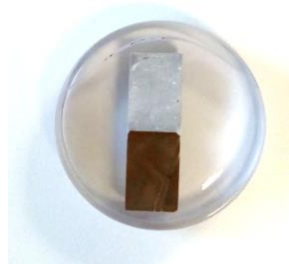
**Fig. 1.** Sample's geometry

The cement paste was prepared with Calcia cement from Beffes (France) and limestone aggregates from Villeneuve Lès Maguelone (France) quarry. Materials characteristics are presented in Table 1.

**Table 1.** Materials characteristics.

<b>Aggregate</b>	<b>Type</b>	Limestone
	<b>Density</b>	$2.7 \text{ g/cm}^3$
<b>Cement paste</b>	<b>Formula</b>	CEM I 52.5 R CE CP2 NF
	<b>W/C ratio</b>	0.4

After fabrication, the composites are kept in lime-saturated water for 90 days to prevent early degradation. In order to allow Scanning Electron Microscope (SEM) observations and nano-indentation, the composites were impregnated in epoxy resin and polished to provide a sufficiently smooth surface (Figure 2).



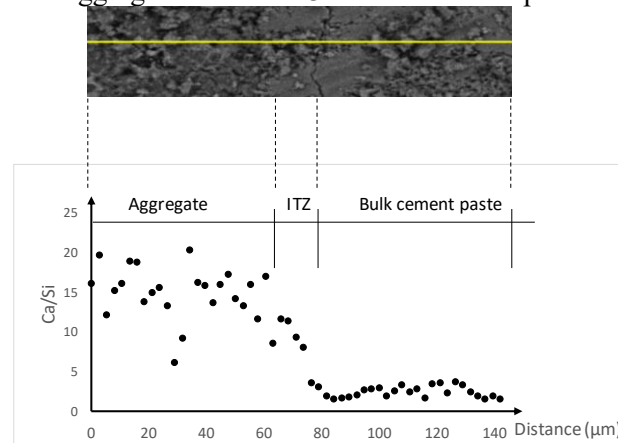
**Fig. 2.** Polished section

## 3. Results

### 3.1. Scanning electron microscope (SEM)

SEM allows chemical analysis by EDS (Energy Dispersive x-ray Spectrometry) by analyzing several points on the same predefined line. It is possible to obtain the longitudinal profile of concentration of several chemical elements, in particular calcium (Ca) and silicium (Si). Classically, Ca/Si molar ratios for hydrated calcium silicates (C-S-H) are between 1,2 and 2,3 and for Portlandite (CH) the ratio is greater.

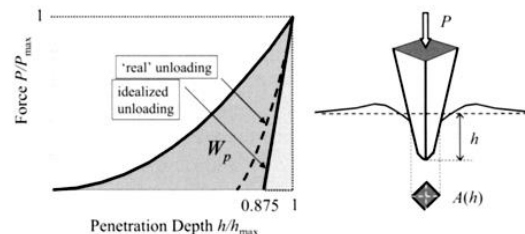
The profiles of the Ca/Si molar ratio in perpendicular direction to the ITZ (Figure 3) show zones of high calcium concentration in the zone of 15-20  $\mu\text{m}$  from the aggregate. The Ca/Si molar ratio at the ITZ varies between 15 close to aggregate and about 3 for bulk cement paste.



**Fig. 3.** Gradient of the Ca/Si molar ratio at the ITZ

### 3.2. Nano-indentation

Nano-indentation is a technique used to characterize the rigidity of materials on a very small scale and is extracted from hardness measurement techniques. Indentation measurements are deduced from the material penetration by a needle to a depth of less than one micrometre. This penetration allows the analysis of the mechanical materials response. Depending on the impression dimension of the indentation tool used, it is possible to deduce the local hardness. The force / depth curve slope at the beginning of the discharge portion allows the measurements of the indentation modulus, by assuming that the response of material is elastic (Figure 4). The realisation of sufficiently large indentation grids gives access to the mechanical properties of material via statistical indicators.



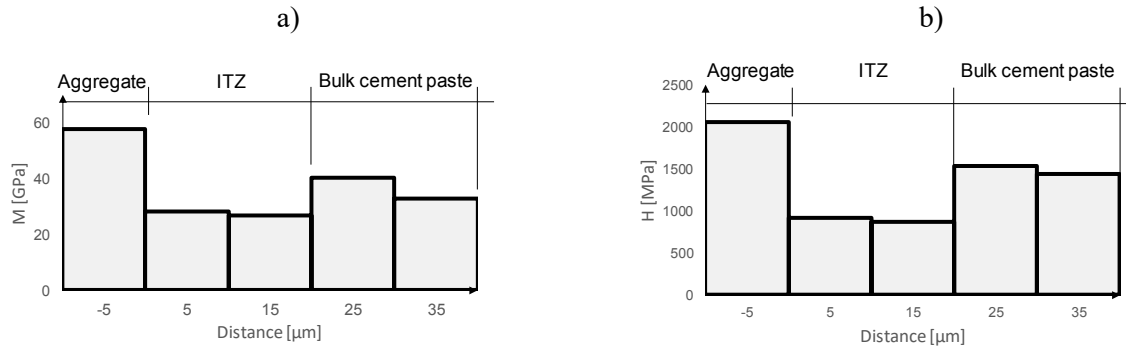
**Fig. 4.** Principle of the nano-indentation test. Measurement of indentation module (left) and hardness (right) <sup>5</sup>

In this study, nano-indentation was used to quantify the hardness and stiffness gradient at the ITZ. For this purpose, indentation grids were made at the ITZ with 10 points for a same distance from cement paste / aggregate. The mechanical property gradient was achieved by averaging all values of hardness and indentation modulus for points located at the same distance from cement paste / aggregate contact.

The figure 5 a) and b) shows the averaged results on the 10  $\mu\text{m}$  of indentation modulus and hardness. We can identify three zones:

- A first zone, very rigid and hard, represent the aggregate.
- A second zone, less rigid, where a decrease in mechanical properties over a distance of 20  $\mu\text{m}$  is observed. This zone represents ITZ.
- A third zone, a rise in values of indentation module and hardness are observed. This zone corresponds to the bulk cement paste.

Nano-indentation results show that the 20  $\mu\text{m}$  zone in locality of aggregate is associated with ITZ. The ratio between average indentation modulus of ITZ and that of cement paste is about 0.75, and the ratio between the average hardness of ITZ and that of cement paste is about 0.6.



**Fig. 5.** Indentation modulus gradient at the ITZ a); Hardness gradient at ITZ

#### 4. Conclusion

The results of chemical observations by SEM and mechanical nano-indentation analyses indicate a zone with a thickness of about 20  $\mu\text{m}$  with properties different from those of the bulk cement paste. From chemical point of view, this zone is characterized by higher Ca/Si molar ratio, suggesting a high content of portlandite (CH). Despite that the portlandite crystals are harder than the C-S-H particles, they have a lesser contribution of the mechanical properties of the ITZ. The results of the nano-indentation tests at ITZ show less good mechanical properties compared to bulk cement paste on the same thickness.

In conclusion, the SEM and nano indentation analyses revealed a zone of about 20  $\mu\text{m}$  in thickness characterized by a high concentration of portlandite, high porosity and lower mechanical properties compared to the bulk cement paste

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