

## INFLUENCE OF INTERFACIAL TRANSITION ZONE ON ENGINEERING PROPERTIES OF THE CONCRETE MANUFACTURED WITH RECYCLED CERAMIC AGGREGATE

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**Abstract.** The influence of the local micro-properties of the interfacial transition zone (ITZ) on macro-properties on the behaviour concretes is studied for recycled concretes made with 20% and 25% recycled ceramic aggregate from sanitary ware waste. This study carried out using nanoindentation and SEM technique aims to explore interface thickness and variations in its elastic modulus. The results show that the minimum and mean values of the elastic modulus and the ITZ thickness impact concrete mechanical and elastic properties to different degrees. Finally, that nanoindentation provides an indispensable tool for studying and assessing the effect of new materials (e.g. recycled aggregates) on the micro-mechanical properties of the interfacial transition zone (ITZ) and its effect on engineering properties (compressive and splitting tensile strength and modulus of elasticity) of these new concretes.

**Keywords:** interfacial transition zone, recycled ceramic concrete, compressive and splitting tensile strength, modulus of elasticity.

### Introduction

The industrial and economic growth observed in more economically developed countries in recent years has brought with it a considerable increase in the generation of wastes. Sustainability performance of the society is an indispensable aspect in attaining the goal of sustainable development (Shen *et al.* 2007). Nowadays, there are intensive investigations carried out in order to utilize the different wastes (construction and demolition wastes, ceramic materials, asphalt, tyres, etc.) in the manufacture of new cements and concretes (Mačiulaitis *et al.* 2009; Kavussi *et al.* 2011).

Concrete is usually considered as a heterogeneous material consisting of three main phases: aggregates, cement paste and the interfacial transition zone (ITZ) between aggregate/paste. All concrete properties are related to the characteristics of these three phases. In particular, an understanding of the inter-relationships between concrete composition, structure and properties depends largely on the knowledge of both the paste and the paste/aggregate interface.

Aggregate/paste, steel/paste and fibre/paste interfacial transition zones have been studied since the 1950's by a number of research teams (Monteiro *et al.* 1985; Ollivier *et al.* 1995; Paulon *et al.* 2004), who explored

the microstructure and properties of the ITZ under the influence of different factors, such as w/c ratio, age, type of aggregate and the presence or different admixtures. Their findings revealed that characteristics of ITZ often differ from those of the bulk cement paste, due primarily to microstructural differences (van Breugel *et al.* 2004): e.g. with greater porosity, less unhydrated clinker phases, precipitation of large portlandite crystals that tend to be aligned perpendicularly to the aggregate surface and the presence of more ettringite. These differences are usually explained by the origin of the ITZ (Wang *et al.* 2009) which includes: a) the so called “wall” effect that prevents dense packing of cement grains against the relatively flat aggregate surface or fibre, or steel surface; and b) the microbleeding effect that leads to accumulation of water underneath the coarse aggregate particles or the steel bars and the flocculation effect of the small cement grains.

The extent of practical impact of these microstructural differences between the ITZ and the bulk paste on the properties of concrete has been debated (Bentur *et al.* 2000; Wang *et al.* 2009) appeared to suggest that such differences of the ITZ did not have significant impact on concrete properties and they were of academic interest only. Whereas other researchers (Akcaoglu *et al.* 2005;

Akçaoğlu *et al.* 2004; Lee, Park 2008) claimed that since the interfacial transition zone is the weakest part of the microstructural system and the place where cracks first appear, it plays a significant role on the mechanical and transport properties of concrete. However, it was argued (Diamond, Huang 2001) that even for concrete with a w/c ratio of 0.50, the ITZ structure need not be assumed to have an adverse effect on permeability or strength. Two committees set up by RILEM to study the topic concluded (Bentur *et al.* 2000) that ITZ properties may have a moderate but certainly not a drastic effect on concrete strength or durability.

The traditional procedures (SEM, BSE, ESEM, TEM and microhardness) for studying cement paste, mortar and concrete microstructure have been supplemented in the last 15 years by nanoindentation (Davydov *et al.* 2011; Stark 2011; Zhu *et al.* 2004a). This technique, has been used to determine the micro-mechanical properties of different phases in hydrated cement (Velez *et al.* 2001), cement paste itself (Hughes, Trtik 2004; Mondal *et al.* 2007; Zhu *et al.* 2007) and the ITZ, aiming to better correlate structure and mechanical properties at the microscale to the macro-mechanical behaviour of these materials.

A number of researches have applied nanoindentation to study the micro-mechanical properties of the ITZ in fibre/paste (Lee *et al.* 2009; Sakulich, Li 2011; Sorelli *et al.* 2008; Wang *et al.* 2009; Zhu, Bartos 1997), steel/paste (Zhu *et al.* 2004b; Zhu, Bartos 2000; 2005) or natural aggregate/paste systems (Mondal *et al.* 2009; Trtik, Bartos 1999). By contrast, no published report was found on research conducted to analyse the variation in the micro-properties of the ITZ in recycled aggregate/paste system, when recycled aggregates were used to replace natural aggregate in normal concrete mixes. With an increasing emphasis on sustainability by using recycled aggregates in concrete, it is important to understand the nature and properties of the ITZ and its impact on concrete properties for such mixes.

The focus of the present paper is on the ITZ between recycled produced from sanitary ware industrial

waste, and cement paste, in concrete mixes with 20% and 25% of the natural aggregate replaced by recycled aggregate. More specifically, it is to explore the thickness of the different ITZs identified (i.e. with the glazed side and ceramic side of the recycled aggregate, as well as with the natural aggregate) and the variations in elastic modulus. The effect of this new aggregate on local micro-properties in the ITZ is quantified and its impact on the engineering properties of concrete is discussed.

## 1. Materials and methods

### 1.1. Materials

The natural aggregate employed can be sub-divided into two categories: the coarse fraction (gravel) of 4/20 mm in size, and the fine fraction (sand), with grains of less than 4 mm in size. The main component,  $\text{SiO}_2$ , accounted for 97% wt. of these aggregates, which also contained  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  as minority oxides. Their mineralogical composition was dominated by quartz, followed to a lesser extent by another series of aluminosilicates belonging to the mica and feldspar groups.

The recycled aggregate employed came from a ceramic sanitary ware factory. This waste was crushed with a jaw crusher and then sieved to obtain the 4/12.5 mm fraction. There are two visually distinguishable sides in the recycled aggregate as shown in Figure 1: the glazed side that formed the external surface of the original sanitary ware, and the ceramic side that corresponded to the internal part of the sanitary ware products.

The physical and mechanical properties of the natural and recycled aggregates are shown in Table 1 (Medina *et al.* 2011) which comply with the requisites established in standard EN 12620 (CEN. 2009b) and Spanish Code Structural Concrete (EHE-08) (Comisión Permanente del Hormigón 2008).

The chemical composition of this ceramic waste was similar to that of other ceramic materials used in construction sector (tiles, bricks, etc.) (Sánchez de Rojas *et al.* 2001, 2003, 2006, 2007). The internal part was composed mainly of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$ , which

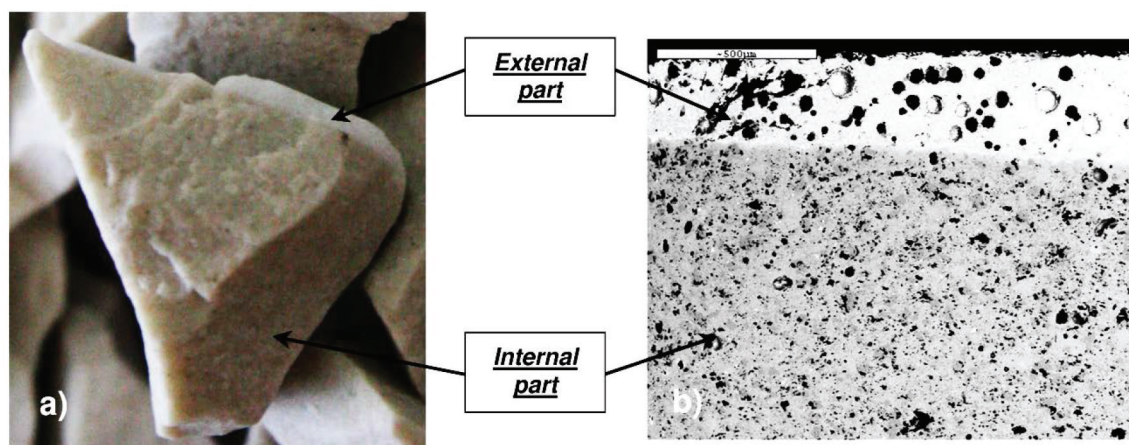


Fig. 1. Recycled ceramic aggregate: a) General aspect; b) BSE image ( $\times 75$ )

Table 1. Physical and mechanical properties of coarse aggregates

Characteristic	Gravel	Ceramic
Dry sample real density (kg/dm <sup>3</sup> )	2.63	2.39
Water absorption (wt. %)	0.23	0.55
Flakiness Index (wt. %)	3	23
“Los Angeles” coefficient (wt. %)	33	20
Total porosity (vol. %)	0.23	0.32

constituted 93.81% of the total, whilst in the external part the proportion of the previous components dropped to 68.24%, with zircon (ZrO<sub>2</sub>) representing 12.62% and calcium oxide (CaO) comprising 11.80%. Meanwhile, the alkalis (MgO, NaO and K<sub>2</sub>O) formed minority components in both parts.

A BRUKER Theta-Theta D8 Advance diffractometer fitted with a 2.2 kW Cu anode but no monochromator was used to determine the mineralogical composition of the materials. The main crystalline components found in the ceramic waste were quartz, orthoclase, mullite, hematite, and zircon (Medina *et al.* 2011).

The cement used for mixing the concretes was CEM I 52.5 R type Portland cement in accordance with European standard EN 197-1 (CEN 2011), with content in clinker greater or equal to 95%, and with 5% additional components.

### 1.2. Concrete mixes used

Three types of concretes were prepared: a reference concrete (RC) and two concretes (CC20 and CC25) in which 20 or 25% wt. of recycled aggregate was used in place of natural coarse aggregate.

The concrete was mixed and cured as specified in European standard EN 12390-2 (CEN 2009a) and used to make test specimens measuring Ø 150 mm × 300 mm and 150×150×150 mm for determining the mechanical and ITZ properties, respectively. The design and calculation of these mixes was carried out using the “De La Peña” method (Fernández 2007). Details of the mixes and physical properties (consistency and bulk density) of the concretes are given in Table 2 and Table 3, respectively.

The modulus of elasticity of the concretes has been calculated through an empirical formula recommended by ACI 318-08 (ACI Committee 318 2008) that it suggests this properties of concrete function of its compressive strength and density Eqn (1):

$$Ec = 0.043w_c^{1.5} \sqrt{f_{ck}}, \quad (1)$$

where:  $w_c$  (kg/m<sup>3</sup>),  $f_{ck}$  (MPa) y  $Ec$  (MPa) are 28 days density, compressive strength and modulus of elasticity of concrete, respectively.

### 1.3. Sample preparation for nanoindentation test

The reliability of results obtained from nanoindentation highly depends on the surface quality of the sample to

Table 2. Details of the concrete mixed used

Mix proportion (kg/m <sup>3</sup> )	RC	CC-20	CC-25
Sand	716.51	725.81	728.14
Gravel	1115.82	892.66	836.87
Ceramic	0.00	216.43	270.53
Cement	398.52	387.64	384.91
Water	205.00	205.00	205.00

Table 3. Physical properties of the concrete

Physical properties	RC	CC-20	CC-25
Slump (mm)	75	72	71
Density (kg/m <sup>3</sup> )	2390	2360	2370

be tested. Thus sample preparation (ASTM 2007; Sorelli *et al.* 2008; Wang *et al.* 2009) is a very important part of the nanoindentation studies on cementitious materials. The heterogeneity of the material with its wide variation in the mechanical performance ( $E = 0$  GPa for pores up to  $E > 100$  GPa for unhydrated cement clinker grains and aggregates) demands a complex sample preparation procedure.

Due to low indentation depth only extremely smooth and flat surfaces allow to obtain reliable and representative test results.

After 28 days curing in a water tank, the centre piece of the 150×150×150 mm<sup>3</sup> cubes was extracted using a diamond saw. This was later cut into small samples of the size about 15×15×10 mm<sup>3</sup>. For the further preparation the specimens were embedded in resin disks (Ø 30 mm) followed by a vacuum impregnation with an epoxy resin with an elastic modulus of around 3 GPa. The resin impregnation was required to support and prevent the loss of the weaker phases in the ITZ and the cement paste during the specimen preparation. To obtain the final test specimens for nanoindentation subsequent grinding and polishing were performed, finishing the polishing steps with a ¼ µm diamond particle sprays. Throughout the preparation process only alcohol or oil-based polishing sprays and lubricants were used to avoid further cement hydration and possible dissolution of hydrate phases.

### 1.4. Nanoindentation testing

Nanoindentation is used by many researches in the world to determine micro-mechanical properties of various materials. Nevertheless, we recall briefly the operational principles of nanoindentation. It is making contact between a sample with mechanical properties to be determined and an indenter tip of known geometry and mechanical properties. When in contact an increasing load is applied to the indenter causing the penetration of the indenter tip into the investigated surface. After reaching a predefined maximum load and typically a short hold period at this value the load is withdrawn



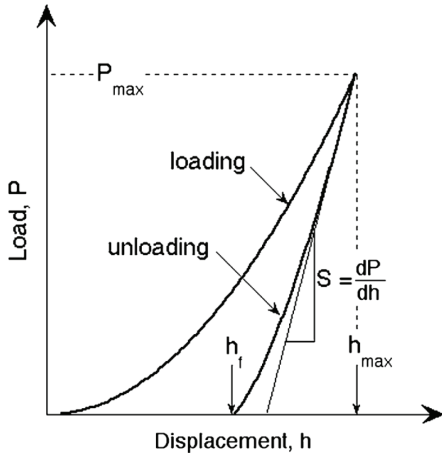


Fig. 2. Theoretical indentation load vs displacement curve

and the penetration depth decreases due to the elastic recovery of the deformed material (Zhu *et al.* 2007; Zhu, Bartos 2000). The change in load ( $P$ ) and depth ( $h$ ) is continuously recorded over the duration of the experiment. A typical outcome (Oliver, Pharr 2004) of nanoindentation testing is an indentation load-displacement (depth) hysteresis curve such as shown in Figure 2.

The determination of the elastic recovery by analysing the unloading data according to a model for the elastic contact problem enables to calculate the modulus of elasticity ( $E$ ). Details of the theoretical background and methodology for the elastic modulus determination

have been reviewed and discussed by Oliver and Pharr (Oliver, Pharr 2004) leading to the following Eqns (2) and (3) (Zhu *et al.* 2004b):

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A}; \quad (2)$$

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu_i^2)}{E_i}, \quad (3)$$

where:  $S = dP/dh$ , is the slope of the upper portion of the unloading curve during the initial stages of unloading (also called contact stiffness). In Eqns (2) and (3)  $E_r$  stands for the reduced elastic modulus and  $A$  is the projected contact area of the indenter tip and  $E$  is Young's modulus and  $\nu$  is Poisson's ratio of the tested materials.

Finally,  $E_i$  and  $\nu_i$  represent the material parameter of the indenter tip (e.g. diamond,  $E_i = 1141$  GPa and  $\nu_i = 0.07$ ).

The nanoindentation experiments in this study were performed using an Agilent G200 Nano Indenter® fitted with a Berkovich indenter tip.

To study the ITZ properties, grids of indentation testing points with varying distances to the actual aggregate-paste interface were selected. The spacing between the indentation test points used were usually 10  $\mu\text{m}$  or 20  $\mu\text{m}$  to cover a sufficient area and also to avoid possible overlapping of the test areas. Nano-indentation testing with a maximum load of 2 mN (corresponding to an average indentation depth of 300 nm

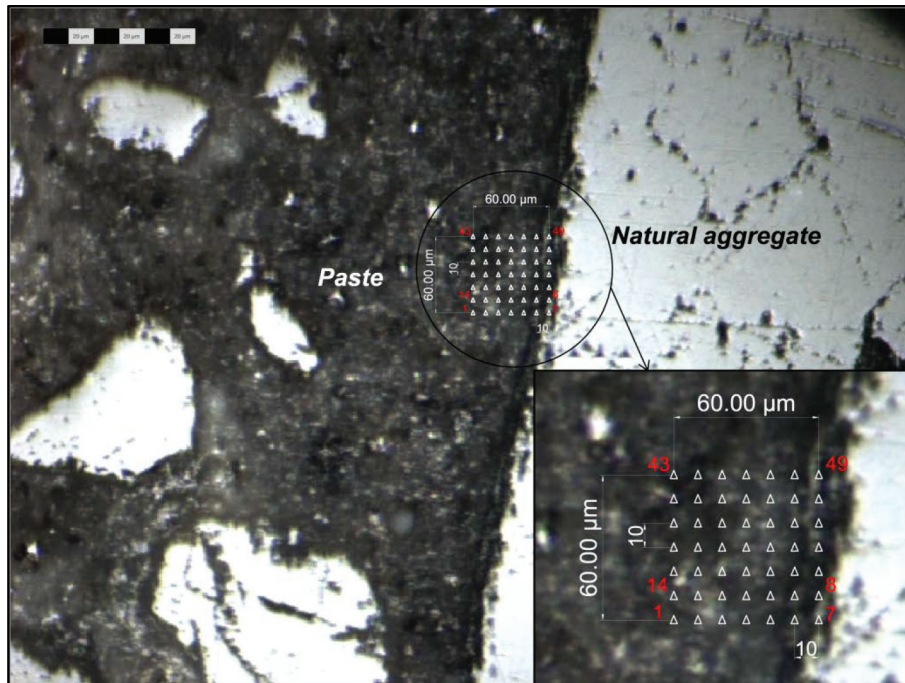


Fig. 3. BSE image of natural aggregate interfacial transition zone, where the dimension of the indented area is 60×60  $\mu\text{m}$  with an indent spacing of 10  $\mu\text{m}$

in most cases) was carried out at each grid points. The perpendicular distance of each indentation point to the actual aggregate – paste interface was measured using an image analysing and the AutoCAD® 2012 tool (Figs 3–5).

Some selected indentation areas are shown in the BSE images of the ITZ between aggregates/paste in Figures 3–5. The indentation areas are contained within

the rectangles outlined in the figures; the number, which always begins from 1, refers to the sequence of the indentation tests carried out in each chosen indented area, which facilitate the measurement of the distance to ITZ for each test point.

Validation of test results after the indentation testing was performed for each point to identify abnormal or discontinuous indentation test curves shapes, high sur-

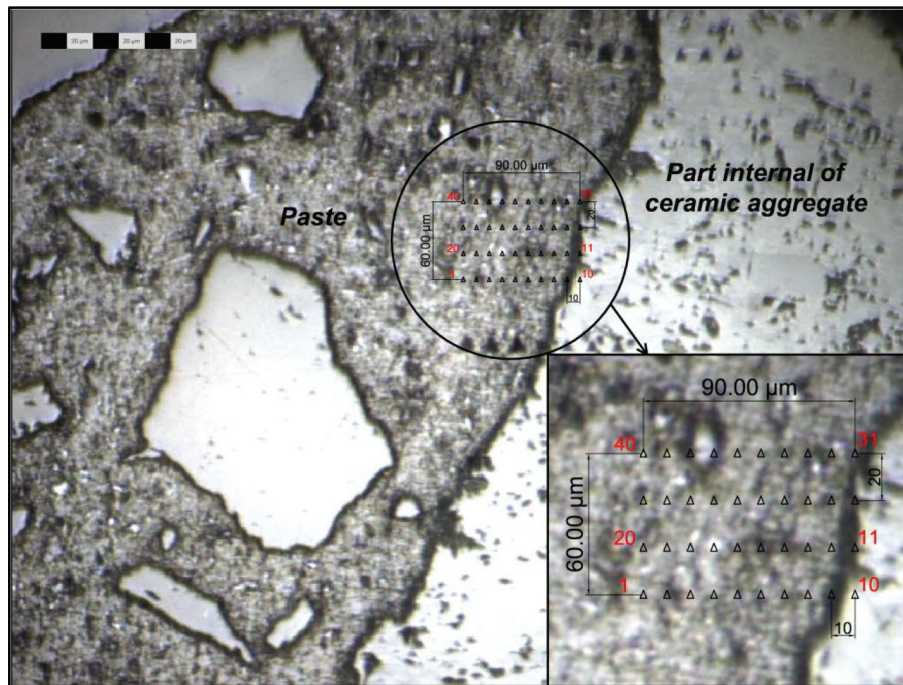


Fig. 4. BSE image of part internal of ceramic aggregate interfacial transition zone, where the dimension of the indented area is  $90 \times 60 \mu\text{m}$  with indent spacing of 10 and 20  $\mu\text{m}$  in X and Y direction respectively

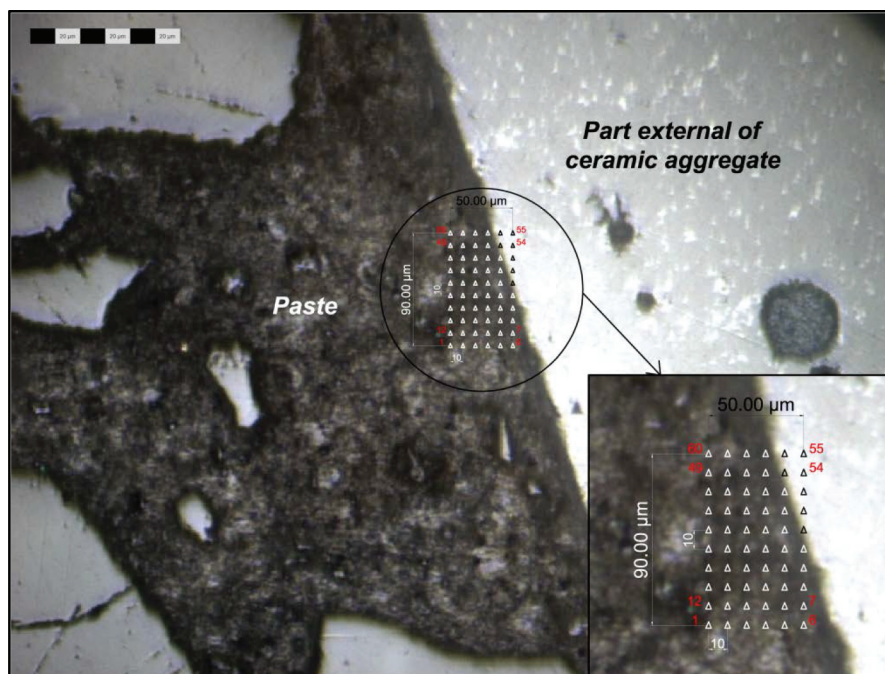


Fig. 5. BSE image of part external of ceramic aggregate interfacial transition zone, where the dimension of the indented area is  $50 \times 90 \mu\text{m}$  with an indent spacing of 10  $\mu\text{m}$



Table 4. Number of indentation test performed in each type of aggregate

Type of aggregate		Number of indentation tests		
		ITZ	Paste	Aggregate
Ceramic aggregate	Internal part (matrix)	90	112	18
	External part (glaze)	104	80	13
Gravel		89	100	10

face roughness, large voids or cracking. When such imperfections were detected, which was only rarely (< 2% of the total), the respective data were excluded in further data analysis. The total number of valid test points obtained and used is provided in Table 4, covering the various aggregates, ITZs and the bulk paste.

## 2. Results and discussion

### 2.1. Characterization of the aggregates

Figure 6 shows the results obtained of elastic modulus from aggregates. The natural aggregate have higher value than ceramic aggregate, as a consequences of its mineralogical composition (see point 2.1). This value is in accordance with the result obtained by other authors (Sakulich, Li 2011; Zhu *et al.* 2007) that they observe an elastic modulus of 100–120 GPa.

With respect to the ceramic aggregate, the internal part has a higher modulus than the external part. The different values obtained between natural aggregate and ceramic aggregate by elastic modulus are due to different mineralogy of these materials, because aluminosilicates have lesser value (80–90 GPa) than quartz.

### 2.2. Characterization of the ITZ

Table 5 shows the variations in the elastic modulus detected with nanoindentation at the ITZ studied (internal part/paste, external part/paste and natural aggregate/paste) and the values of the standard deviation. Variations observed in the elastic modulus across the ITZ concurred with the findings reported in research papers on natural aggregate (Lutz *et al.* 1997; Sakulich, Li 2011; Zhu *et al.* 2004b). In the area closest to the surface

(5–15  $\mu\text{m}$ ), the modulus declined to a minimum value that ranged from 19.9 to 25.4 GPa. In this point ( $x = 15 \mu\text{m}$ ) we can observe important differences in function of the type of aggregate-paste interface. It subsequently rose in the 15–30  $\mu\text{m}$  area and flattened at around 30–40 GPa at distances of over 35  $\mu\text{m}$ . The value recorded at the points adjacent to or at less than 10  $\mu\text{m}$  from the aggregate surface was impacted by proximity to the surface, an observation also reported by Zhu and Bartos (Zhu *et al.* 2004b). This is mainly because the affected surface area by the indentation could be 5–7 times greater than the indentation depth. Also, the interface observed in 2D may slightly differ from the real interface in 3D, which could lead to a small error of the distance to the interface measured.

As regards the lowest of the minimum values observed ( $19.9 \pm 2.7$  GPa) was found for the natural aggregate/paste interface (Figure 8), followed by the value for the interface between the external (glazed) part of the recycled ceramic aggregate/paste ( $23.7 \pm 2.5$  GPa) and lastly the area between the internal part/paste ( $25.4 \pm 2.5$  GPa): i.e. the elastic modulus for the ceramic aggregate were 19.1 and 27.6% greater, respectively, than for the natural aggregate. These differences may be explained to the shape and chemical composition of the recycled ceramic aggregate. The ceramic materials (Sánchez de Rojas *et al.* 1993; Luxan *et al.* 1989; Mehta 1981; Uzal, Turanli 2003) have pozzolanic activity when they are in powder and this activity is smaller (Medina *et al.* 2012a, b) when they have coarse size. This research group has evaluated this activity with an accelerated chemical test that it is based on lime uptake by the material when it was cured in a saturated lime solution. The results obtained shown that this ceramic waste displayed acceptable pozzolanic activity, having been fixed more than 50% after 28 days. This value is similar to other ceramic materials (clay tile, bricks, etc.) but it's more active than fly ash at this age and it is less than silica fume.

The Figure 7 shows the points where we have analysed the value of C/S ratio from gel C-S-H of the different ITZs. The results obtained are shown in the Table 6. You can observe that the C/S ratios are similar and in accordance the interval values (1.2–2.3) established by Hewlett (1998) and Taylor (1997). These results indicate that the incorporation of ceramic aggregate doesn't present any negative effective in the formation of gel C-S-H.

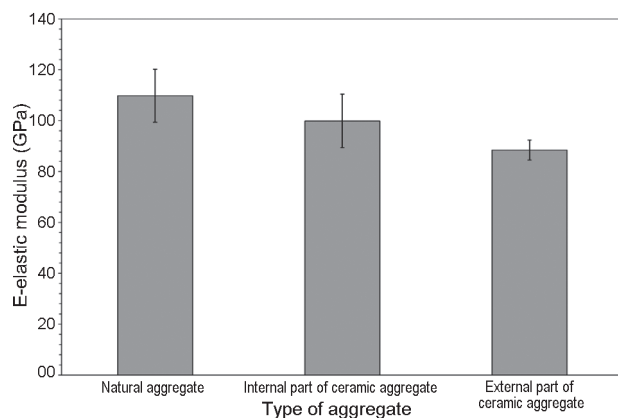


Fig. 6. Elastic modulus of coarse aggregates

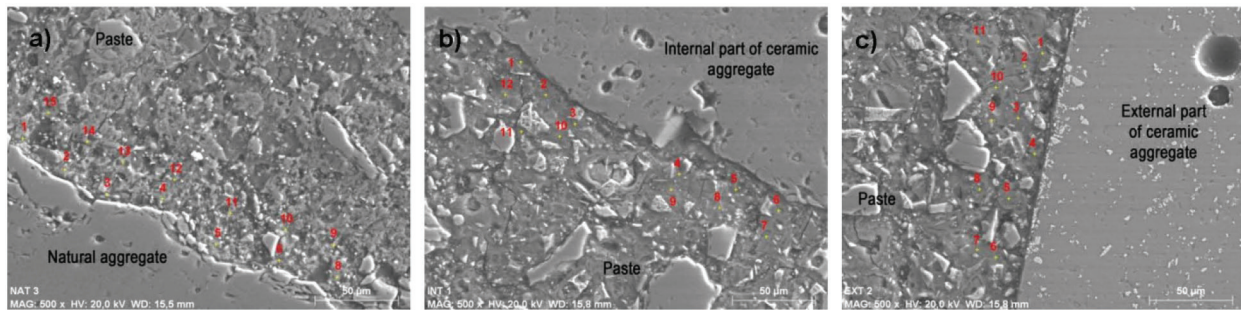


Fig. 7. BSE image of different ITZs (500X): a) Aggregate/paste; b) Internal part of ceramic aggregate/paste; c) External part of ceramic aggregate/paste

Table 5. Elastic modulus (GPa) vs distance from the aggregate surface by type of aggregate

Distance from the interface ( $\mu\text{m}$ )	Elastic modulus (GPa)		
	Ceramic aggregate		Gravel
	Internal part (matrix)	External part (glaze)	
5	46.7 $\pm$ 5.3 <sup>a</sup>	61.0 $\pm$ 6.7	70.0 $\pm$ 5.9
10	31.0 $\pm$ 3.7	36.5 $\pm$ 3.4	37.3 $\pm$ 1.6
15	<b>25.4<math>\pm</math>2.5</b>	<b>23.7<math>\pm</math>2.5</b>	<b>19.9<math>\pm</math>2.7</b>
20	27.8 $\pm$ 3.0	24.8 $\pm$ 2.6	22.7 $\pm$ 3.3
25	27.3 $\pm$ 3.3	25.8 $\pm$ 2.5	29.1 $\pm$ 3.8
30	29.4 $\pm$ 4.2	29.9 $\pm$ 3.2	31.1 $\pm$ 4.5
35	33.5 $\pm$ 3.8	32.6 $\pm$ 3.0	30.3 $\pm$ 1.6
40	34.3 $\pm$ 4.1	35.4 $\pm$ 2.6	32.6 $\pm$ 3.1
45	34.0 $\pm$ 2.7	33.3 $\pm$ 3.5	33.9 $\pm$ 3.5
50	30.8 $\pm$ 3.7	31.9 $\pm$ 2.2	31.2 $\pm$ 2.2
55	31.7 $\pm$ 3.2	39.4 $\pm$ 3.5	35.3 $\pm$ 4.7
60	34.0 $\pm$ 2.4	37.8 $\pm$ 3.4	34.9 $\pm$ 2.6
65	33.8 $\pm$ 4.4	34.2 $\pm$ 2.6	34.7 $\pm$ 4.1
70	34.4 $\pm$ 3.0	36.4 $\pm$ 3.2	34.6 $\pm$ 2.6

<sup>a</sup> The + represents one standard deviation

The elastic moduli were classified into two groups for comparison and assessment of the variations in the areas analysed (Fig. 8): one group was calculated for distances within the interface ( $10 \mu\text{m} < d < 30 \mu\text{m}$ ) and the other for distances in the paste ( $35 \mu\text{m} < d < 70 \mu\text{m}$ ). As the figure shows, the mean elastic modulus remained practically constant in the two areas (ITZ and paste) in concrete with both natural and recycled aggregate, an indication of similar behaviour. The mean  $E$  value for the ITZ was found to be around 84% of  $E$  value for the bulk paste for all the aggregate-paste systems studies. These findings are consistent with the results reported by Trtik and Bartos (1999) and Mondal *et al.* (2009), who observed that the elastic moduli at the interface ranged from 30 to 90% of the value in the paste, depending on the type of aggregate.

As Table 5 shows, the ITZ was 10–30  $\mu\text{m}$  thickness in all cases, which concurred with the values observed by Zimbelmann (1985), Delagrave *et al.* (1997) and Trtik and Bartos (1999). Moreover, that value was smaller than both

the normal thickness  $10 \times 50 \mu\text{m}$  found in natural aggregate/paste interfaces (Lee, Park 2008; Zheng *et al.* 2005), and the 30 to 60  $\mu\text{m}$  reported for recycled concrete aggregate/paste interfaces (Li *et al.* 2009; Poon *et al.* 2004). Such results constitute further evidence that the inclusion of this new type of recycled aggregate had no adverse effect on interfacial transition zone micro-properties. To some extent, it contributes to improve the ITZ properties, as the enhanced lowest  $E$  value within the ITZ suggests.

### 2.3. Engineering properties

In light of the foregoing, three basic engineering properties (compressive strength, splitting tensile strength and modulus of elasticity) were analysed with a view to assessing the beneficial effect of recycled ceramic aggregate on the macro-properties of recycled concrete.

Table 7 clearly shows that the inclusion of recycled sanitary ware as aggregate improved the engineering properties of conventional concrete (Medina *et al.* 2011, 2012a, b).

Table 6. Microanalysis of gel C-S-H on different ITZs

Element	Natural aggregate/paste	Internal part of ceramic aggregate/paste	External part of ceramic aggregate/paste
O	34.06±0.30	39.18±0.75	39.23±0.45
Na	0.00±0.08	0.11±0.15	0.12±0.13
Mg	0.39±0.14	2.49±1.30	1.80±0.74
Al	2.44±0.17	4.13±1.03	3.40±0.84
Si	18.76±0.60	16.00±1.75	16.36±0.81
P	0.00±0.01	0.19±0.16	0.20±0.11
S	0.51±0.05	0.67±0.40	0.88±0.47
K	0.85±0.07	0.32±0.17	0.24±0.06
Ca	41.02±0.98	34.92±3.27	35.88±1.59
Ti	0.05±0.02	0.19±0.15	0.25±0.21
Fe	1.42±0.19	1.78±0.53	1.81±0.44
C/S	2.18	2.18	2.19

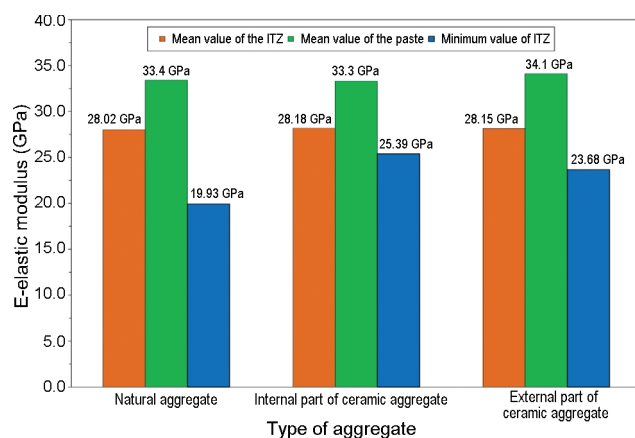


Fig. 8. Mean and minimum values of elastic modulus (GPa) in interfacial transition zone and mean value in paste

The greater compressive and splitting tensile strength in these new concretes (Medina *et al.* 2012b) can be attributed to the intrinsic properties of the ceramic aggregate (Medina *et al.* 2011) and the effect of the microproperties of interfacial transition zone on the mechanical behaviour (Holschemacher 2004). Note that the minimum elastic modulus for both the internal and external part of ceramic aggregate/paste interface was higher than for the natural aggregate/paste interface.

Splitting tensile strength rose more than the other parameters because this strength is closely related to the ease with which micro-cracks form (Akçaoğlu *et al.* 2004; Tasdemir *et al.* 1998) at the ITZ when the concrete is loaded. This increase was 19.70% higher in CC-20 and 25.65% in CC-25 than in the RC. The primary reason for the appearance of such micro-cracks is that the elastic modulus differs in paste and aggregate (Fig. 6); and that difference is greater between natural aggregate/paste than between recycled aggregate/paste.

The estimated value of the modulus of elasticity in concretes (Table 7) was observed to rise slightly with the

Table 7. Engineering properties of the 28-day concretes

Type of concrete	Compressive strength (MPa)	Tensile strength (MPa)	Modulus of elasticity (GPa)*
RC	35.87	2.69	30.1
CC-20	38.53	3.22	30.6
CC-25	39.83	3.38	31.3

\*Calculated through an empirical formula suggest by ACI 318-08.

percentage of ceramic aggregate and was 1.7% higher in CC20 and 4.0% higher in CC25 than in the reference concrete. This behaviour is closely related to the slightly higher elastic modulus found in the (internal and external) ceramic aggregate/paste interface than in the natural aggregate/paste interface. These findings are consistent with Lee and Park (2008).

## Conclusions

A number of conclusions about the use of nanoindentation in the research of the interfacial transition zone between recycled ceramic aggregate/paste in recycled concrete can be drawn from this study:

1. The general distribution pattern of the elastic modulus in both recycled ceramic/paste and natural aggregate/paste interfacial zone is as expected, and similar to those reported previously.
2. The minimum value of the elastic modulus was greater in the ITZ between (internal and external part) ceramic aggregate/paste than in the natural aggregate/paste interface. This appears to correlate well with the improved tensile strength properties of the concrete containing such recycled aggregates.
3. The minimum E value was found at around 15  $\mu\text{m}$  from the actual aggregate surface. There is, however, no significant difference in mean E value of



the ITZ between the recycled ceramic aggregate/paste and the natural aggregate/paste systems.

4. The thickness of the ITZ was found to be 10–30  $\mu\text{m}$  for both the natural aggregate-paste and recycled aggregate-paste system, which is thinner than found for other types of recycled aggregates reported. The results also suggest that the ITZ is not adversely affected by the inclusion of recycled aggregate used in the study.

The findings of the present study confirm that nano-indentation is an indispensable tool for studying and assessing the effect of new materials (recycled aggregate) on the micro-mechanical properties of the interfacial transition zone (ITZ) and their impact on the macro properties of future recycled aggregate concretes.

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