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TRANSITION ZONE IN HIGH PERFORMANCE CONCRETE DURING HYDRATION

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ABSTRACT

A number of transition zone studies are available in the literature for normal strength concrete. High performance concrete behaves differently from normal strength concrete and very few transition zone studies are available for high performance concrete. The weakest unique transition zone between cement paste and coarse aggregate particle controls many important properties of concrete such as compressive strength.

In this paper the relationship between transition zone microstructure and compressive strength property of high performance concrete using Australian industrial silica fume and Australian aggregates is investigated. Based on the scanning electron microscopic studies of transition zone, it has been found that high performance concrete transition zone thickness increases while compressive strength increases. Further, high performance concrete transition zone thickness is much less than the thickness of transition zone of normal strength concrete. This thin transition zone also enhances compressive strength. It has been observed that silica fume presence in the transition zone significantly enhances compressive strength. Silica fume particles consume calcium hydroxide, which is present in transition zone, and make the zone dense and uniform. © *1997 Elsevier Science Ltd*

Introduction

High performance concrete is more homogeneous than normal strength concrete. High performance concrete with small aggregates is similar to a strong rock. The aggregate, matrix and the transition zone control the concrete properties. Structure changes in matrix and transition zone due to silica fume addition which enhances compressive strength of high performance concrete. Normal strength concrete transition zone thickness is up to fifty μm around coarse aggregates which makes a weak bond between aggregate and matrix.

Breton et al. (1) discussed the main transition zone formation mechanisms developed by Barnes, Diamond and Dolch (2), Ollivier and Grandet (3), Monteiro (4), and Zimbelman (5). The transition zone models developed by the above researchers, differ from each other. A single unique model of transition zone is not available yet. All the transition zone models consist of Calcium Hydroxide (CH) crystals and Calcium Silicate Hydrate (CSH). The CH region is not continuous within transition zone.

Detwiler and Mehta (6) found that the physical effect of micro filling of silica fume is primarily responsible for strength enhancement in high performance concrete. Pozzolanic reaction has less effect initially. Ting and Patnaikuni (7) found that compressive strength is significantly enhanced up to 56 days. This later strength enhancement is believed to be due to pozzolanic action. Studies of physical effect and chemical effect at transition zone is not available yet for high performance concrete. Microcracks propagate at transition zone at early stages before any external stress is applied possibly due to shrinkage. High performance concrete behaves in a more brittle manner than normal strength concrete. This paper investigates the transition zone structure of high performance concrete in order to modify and enhance the strength of the transition zone using industrial silica fume. Significant strength enhancement in normal strength concrete is due to cement hydration mainly during first 28 days but for high performance concrete strength enhancement takes place mainly up to 56 days. Cement hydration and silica fume's physical effect of microfilling and Pozzolanic effect both enhance strength up to 28 days and after that pozzolanic reaction at matrix and transition zone enhances the strength of high performance concrete.

Terminology

The following terms are used in this paper.

Normal High Strength Concrete (NHSC)	- 50 to 100 MPa
Very High Strength Concrete (VHSC)	- 100 to 150 MPa
Microcrack (MCK)	- Up to 10 micrometers opening
TZ	- Denotes transition zone between cement paste and coarse aggregate particle
TZMCK	- Denotes transition zone microcrack
CH	- Denotes $Ca(OH)_2$
CSH	- Denotes Calcium Silicate Hydrate
CASH	- Denotes Calcium Aluminium Silicate Hydrate
CSKH	- Denotes Calcium Silicate Potassium Hydrate
CSF	- Denotes Condensed Silica Fume
OPC	- Ordinary Portland Cement
W/B	- Denotes Water to Binder ratio

Casting and Curing of VHSC Cylinders

Australian silica fume supplied by PIONEER industries and 7 mm maximum size Kilmore Basalt (from Victoria) were used in the production of very high strength concrete. The chemical and physical properties of CSF and OPC are given in Table 1.

Binder content (cement + CSF) of 576 kg/m^3 with 15% of CSF and W/B ratio of 0.23 were used in the mix proportions. Sulphonated Naphthalene Formaldehyde type super plasti-

TABLE 1

Characterisation of CSF and OPC

Chemical /Physical properties	OPC %	CSF %
SiO_2	19.9	90.08
Al_2O_3	4.2	0.45
Fe_2O_3	5.6	0.09
CaO	64.2	3.65
MgO	-	0.28
SO_3	-	0.06
Na_2O	-	0.00
K_2O	-	0.31
Surface area	365 m^2/kg	23995 m^2/kg
Loss of ignition	1.0	4.95

ciser was used in the production of very high strength concrete. The coarse aggregate and fine aggregate were oven dried at average of 39°C to constant mass to avoid water film on the aggregate surface.

A horizontal pan concrete mixer of 0.08 m^3 capacity and a vibrating table which can accommodate 28 cylinders of size 75 mm × 150 mm at a time were used in the production of very high strength concrete cylinders. 75 mm × 150 mm cylinders were used as it was established that these cylinders give the same results as 100 mm × 200 mm cylinders (8,9,10). The concrete cylinders were cured in a lime saturated water tank. Water circulation and temperature control systems were maintained for the water tank.

Compressive Strength Testing of Cylinders

75 mm × 170 mm VHSC cylinders were cast initially and the rough top ends of the concrete cylinders were cut by a diamond saw to obtain the size of 75 mm × 150 mm. Both the smooth ends of the cylinders were capped with unbonded rubber caps and tested using a system developed by Ting and Patnaikuni (7). 75 mm × 150 mm VHSC cylinders were tested on a high-precision computerised MTS testing machine on 3rd, 7th, 14th, 28th and 56th day. The rate of loading used was 20 MPa/minute as specified by Australian Standard AS 1012.9 (11).

Microstructure Examination of VHSC Samples

Very high strength concrete samples for microstructural study were prepared from the 75 mm diameter × 20 mm height discs. Approximately 10 mm × 10 mm samples were cut by diamond saw. Then these samples were polished by using a fine diamond wheel and silicon carbide powders of grades 4, 6 and 8. The polished samples were checked under stereoscopic microscope to ensure that scratches or foreign particles are not present.

VHSC samples were coated with gold using a sputter coating machine. The samples were coated in one single operation without repeat coating. Gold coating was preferred by the authors over carbon coating as carbon coating requires higher vacuum pressure which may

TABLE 2

Experimental Results

Age (days) ↓	Average transition zone thickness (μm)			Compressive strength (MPa)		
Batch→	K1	K2	K3	K1	K2	K3
03	9.1	7.9	6.3	70.6	76.8	82.0
07	11.4	-	11.5	90.3	99.9	102.1
14	11.7	10.8	11.7	112.8	113.2	116.4
28	-	11.3	12.2	120.5	128.1	123.6
56	12.5	12.0	12.5	130.9	139.5	125.5

cause additional microcracking. A thin colloidal graphite layer was applied from the stub (sample holder) to the gold coated sample in order to provide continuity of conductivity. Microstructure examination was carried out using JEOL JSM-35 CF model scanning electron microscope operated at 25 keV. An energy dispersive X-ray analyser was used to identify the phases observed.

Experimental Results

Both the compressive strength testing and the microstructural study were carried out at the concrete ages of 3, 7, 14, 28 and 56 days. The experimental results are tabulated in Table 2.

7 day microstructure examination for batch K2, and 28 day microstructure examination for batch K1 could not be performed as the scanning electron microscope was not available on those days due to breakdown. K1, K2 and K3 are three identical batches of concrete designed to give the same compressive strength but were cast on different days.

Mean values of compressive strength versus transition zone thickness of three different batches K1, K2, K3 are plotted in the Figure 1. Transition zone in silica fume concrete refers to the thickness of the interfacial zone at the aggregate boundary where silica fume has reacted with lime and alkali to produce additional CSH.

Transition zone thickness increases almost linearly with compressive strength up to 100 MPa strength. From a strength of 100 MPa the slope of the line changes but remains nearly

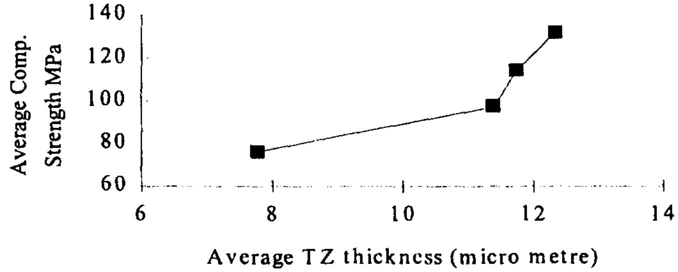


FIG. 1.
TZ-Compressive strength relationship.

linear. Transition zone thickness is increased from approximately 8 μm to up to 13 μm during hydration period from 3 to 56 days. After 14 days the increase in thickness is very small.

Transition zone formation models for normal strength concrete, developed by Olliver and Grandet, Zimbelman, and Monteiro, which were discussed by Breton et al. (1), consist of needle ettringite structure. The needle structure was not found in high performance concrete throughout 56 days of hydration. This needle structure is responsible for less strength in normal strength concrete, but since it is not present in high performance concrete, there is no reduction in strength.

In the case of NHSC calcium hydroxide forms were found near coarse aggregates on third day of hydration. The CH forms were either minimised or fully eliminated by 56 days of age due to consumption by silica fume. High content of CSH and CASH were found at transition zone at 56 days of age. CSKH structure was also found mostly during the hydration. Silicon count was increased significantly after 14 days of age and it is believed that this is due to the reaction of silica fume particles in the transition zone. After 14 days of age transition zone thickness almost became constant and this is due to the consumption of calcium hydroxide in transition zone. Silica fume also chemisorb alkalis present at transition zone. Transition zone becomes dense and uniform after 14 days of age. TZMCK forms at very early stage near coarse aggregate surface and the microcrack is being pushed towards matrix during hydration as shown in Figure 2 and Figure 3. Formation of transition zone during hydration in high performance concrete could be presented as shown in Figure 2. The exact formation mechanism can not be described by one model as described by Breton (1). Only one dense film of transition zone was observed in high performance concrete during hydration.

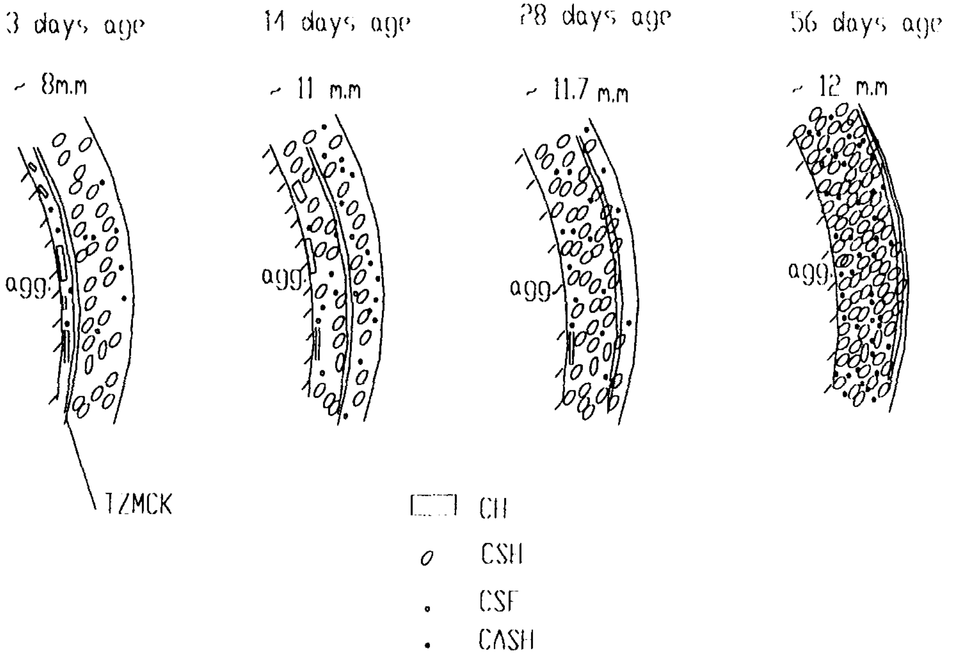
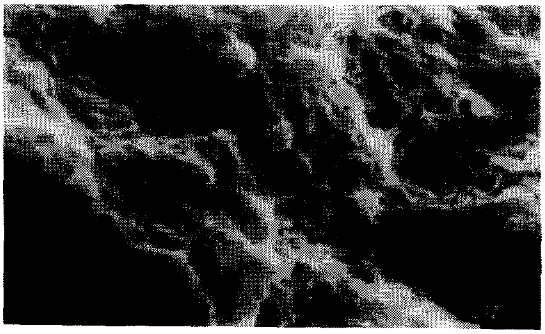


FIG. 2
Transition zone structure during hydration.

3 days of age

7 days of age



14 days of age

28 days of age

56 days of age

FIG. 3.
Transition zone during hydration-batch **K3**.

Silica fume addition in concrete modifies the transition zone and enhances compressive strength but the effective modification of transition zone could be either affected or slowed down by TZMCK, which forms at transition zone before three days. It is possible that, due to the TZMCK opening, furthur reaction of silica fume particles nearer to coarse aggregate surface could be delayed.

It is also observed that, when the aggregates are very close, of the order of less than 100 μm, transition zone thickness around each aggregate varies. Transition zone of one aggregate is thicker than the other aggregate transition zone.

Conclusions

- Transition zone and TZMCK forms before 3 days of age around coarse aggregates.
- Transition zone thickness increases while compressive strength increases.
- High performance concrete transition zone thickness is much lesser than normal strength concrete and is up to approximately 15 μm thick while for normal strength concrete it is up to 50 μm.
- Silica fume particles strengthen the transition zone structure by the consumption of calcium hydroxide.

References

1. D. Breton, A. Carles-Gibergues, G. Ballivy and J. Grandet, Contribution to the formation mechanism of the transition zone between rock-cement paste, Cement and Concrete Research, 23, 335-346 (1993).
2. B.D. Barnes, S. Diamond and W.L. Dolch, The contact zone between portland cement paste and glass aggregate surface, Cement and Concrete Research, 8, 233-244 (1978).
3. J.-P. Ollivier and J. Grandet, Processus de formation de l'aure'ole de transition, Liaion pates de ciment materiaux associes, Coll. Int. Toulouse, France, 1982 p. a-14 a' A-22.
4. P.J.M. Monteiro, Improvement of the aggregate-cement paste transition zone by grain refinement of hydration products, 8th Int. Cong. on the Chem. of Cement, Rio, Brasilia, 3, 433-437 (1986).
5. R. Zimbelman, Contribution of cement-aggregate bond, Cement and Concrete Research, 15, 801-808 (1985).
6. R. Detwiler and P. Mehta, Chemical and Physical effects of silica fume on the mechanical behaviour of concrete, ACI Materials journal, 86, 609-614 (November-December 1989).
7. E.S.K. Ting and I. Patnaikuni, Compressive strength testing of concrete using a new unbonded restrained capping system, Proceedings of the 2nd International conference on the concrete future, Kuala Lumpur, Malaysia, 1993.
8. E.S.K. Ting, I. Patnaikuni, R.S. Pandyala and H.A. Johansons, Effect of Cylinder Size on the Compressive Strength Test Results of Very High Strength Concrete, Proceedings of the International conference on the concrete future, Kuala Lumpur, Malaysia, 1992, pp. 253-256.
9. E.S.K. Ting and I. Patnaikuni, Influence of mix ingredients of the compressive strength of very high strength concrete, Proceedings of the 17th conference on our world in concrete & structures, Singapore, August 1992, pp. 207-215.
10. I. Patnaikuni, H.A. Johansons and E.S.K. Ting Suitable loading rate for very high strength concrete, Proceedings of the International conference on structural failure, Durability, Retrofitting, Singapore, July 1993, pp. 704-709.
11. AS 1012.9-1986 Method of testing concrete, part 9-Method for the determination of the compressive strength of concrete specimens, Standards Association of Australia, Sydney, 1986.