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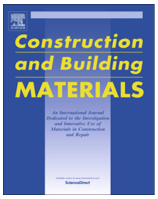

Effects of high-pressure/temperature curing on reactive powder concrete microstructure formation

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HIGHLIGHTS

- Pressure decreases the total pore volume, but increases the capillary pore volume.
- Heat curing accelerates the hydration, pozzolonic and crack progression.
- Heat curing induces crystalline hydrate formation inside the capillary network.
- Both treatments result transformation in micro structural composition.
- Both treatments do not affect the ITZ phase.

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ABSTRACT

Reactive powder concrete (RPC) comprises cement with quartz sand, superplasticizer, silica fume, and water which is processed by heat curing and/or pressure. This paper presents the effect of treatments (static pressure of 8 MPa and heat curing at 240 °C for 48 h) on microstructure formation. Results indicated that pressure decreased the total pore volume, but increased the capillary pore volume due to the movement of grains. The space created could allow additional C–S–H growth during hydration (and later pozzolonic reaction). Heat treatment accelerated the propagation of microcracks (formed during shrinkage) due to thermal expansion of the solid phases, volumetric expansion of the air and increased pressure within entrapped voids. It induced further crystalline hydrate formation inside the capillary pore network. Pressure following by heat curing treatment firstly increased the capillary pore volume and then accelerated both the hydration and pozzolonic reactions with subsequent increased in skeletal density.

1. Introduction

Reactive powder concrete (RPC) is an advanced concrete technology produced using an enhanced composite powder mix, and processed by high-pressure setting and post-setting heat treatment [1]. Exclusion of macro defects and improved homogeneity of the microstructure is achieved by limiting maximum aggregate diameter to 0.6 μm, a water–binder ratio typically <0.18, lowering of the $CaO–SiO_2$ ratio by addition of condensed micro silica, and enhancing ductility using steel fibre reinforcement [1–5]. RPC can achieve ultra-high performance in terms of mechanical properties, where unconfined compressive strength can range between 200 and 800 MPa, with fracture toughness of up to 40,000 J/m^2, and an ultimate elongation of up to 0.007 m/m^{-1} [1]. The world's first major structure built with RPC is the Sherbrooke pedestrian/bikeway bridge (in Canada, 1997) [6], having a span of 60 m and with a compressive strength of 150 MPa and no steel bar reinforcement. The main beam of the bridge was treated by heat curing at 90 °C for two days in a vapour-saturated atmosphere. The diagonal stiffeners of this bridge were cast in a 75 mm tube metal frame to which the static pressure treatment at 2 MPa was immediately applied, followed by heat curing after one day.

Other RPC bridges have been developed around the world such as the Sunyudo (Peace) Footbridge with a single span of 120 m (Seoul, Korea), the Sakata-Mirai Footbridge with no reinforcement

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and lighter by 80% (Sakata, Japan), the Shepherds Creek Road Bridge with a thin permanent precast RPC formwork panels (Australia), and the Wapello Country Bridge with no reinforcement and used for a highway (USA) [7]. The thinnest precast concrete structure achieved using RPC is 20 mm thick and area dimensions 5 × 6 m; a train station roof supported by a single column (150 MPa) [8]. Its potential for even greater mechanical properties give RPC the opportunity for many applications beyond those of conventional concrete, e.g. advanced lightweight composites [9] or impermeable containers for hazardous fluids or nuclear waste [10].

Heat treatment is typically applied to RPC after the final setting time using temperatures between 90 and 200 °C [11]. This accelerates the pozzolonic reaction and also modifies the micro structures of hydrates by changing the C–S–H chain length from trimer to pentamer [11]. Whilst temperature curing between 200 and 250 °C microstructural change and the formation of xonotlite with some dehydroxylation of cement gel can occur [12]. This xonotlite is formed spontaneously when water extracted from hydrates is trapped in the centre of samples during transient heating [13]. Applying heat curing only in an autoclave has produced RPC with an unconfined compressive strength >200 MPa [14–16]. Earlier work suggested that the combination of pressure and heat curing during processing may result in higher strengths than by heat treatment alone because the addition of pressure treatment to the fresh concrete increases the density and decreases the porosity [1,9]. Both density and porosity have close correlation to the bulk mechanical properties of concrete [17]. More recently, Helmi et al. [18] observed that heat treatment during the hardening stage has a significant effect on the early age compressive strength, whereas static pressure treatment during the setting stage has an insignificant effect. However, in combination when heat treatment is applied after treatment by static pressing, the strength increases by a further significant amount due to pore-filling by tobermorite/xonotlite formation from the pozzolanic reaction and enhancing the paste-aggregate bonding mechanism.

Spherical pores (caused by air entrapment) in RPC generally remain air-filled with some calcium hydroxide crystals following standard curing, but can be partially-filled with the calcium-silicate hydrate (C–S–H) mineral, tobermorite ($Ca_{4+x}(H_{2-2x}Si_6O_{17})\cdot 5H_2O$) when autoclave cured [16] or the much harder mineral, xonotlite ($Ca_6(Si_6O_{17})(OH)_2$) if the curing temperatures are sufficiently high. Some researchers have suggested that this would lead to reductions in measured bulk porosity by pore filling with a C–S–H product and consequently alters the pore size distribution by reducing the modal pore diameter [11–16]. Helmi et al. [18] found that for a compressive strength/bulk density (f_c/ρ_d) plot (using Neville's equation – see Eq. (1) [17]), the integer, n for non-pressure treated and pressure-treated samples were significantly different, but the measured compressive strength was not.

$$f_c = f_{c,0}(1-n)^x \qquad (1)$$

where: n = porosity, f_c = unconfined compressive strength, $f_{c,0}$ = compressive strength of hardened paste (at zero porosity), and x = scaling integer. This assumes that $f_{c,0}$ equals 500 MPa for cement paste at w/c = 0.45. When heat treatment was applied in both cases, the heat treated samples fitted the same f'/ρ_d plot as the non-heat treated. This appears to be consistent with the general hypothesis that pore network geometry (resulting from entrapped air) is altered during the process. If true, the heat treatment component of the process could result in (i) increased pore air pressure by reduction in modal pore diameter, (ii) reduction in microcracks within the mortar phase, and/or (iii) change in macro defect stress distribution.

The aim of this study was to further the understanding of the process-structure-property relationships for RPC in terms of pore network geometry and interfacial transition zone. The purpose is to develop a basic conceptual model for heat treatment and static pressure setting, which could enable material design and optimisation of RPC at the microstructural scale for use in advanced applications.

2. Materials and method

The powder ingredients comprised CEM I 52.5 Portland cement according to BS EN 197-1:2011 [19] (Cemex, UK) that it had been used by [20]; condensed micro silica grade 940-D (Elkem, Switzerland) [20]; and ground granulated blast furnace slag (Hanson, UK) [21]. Some properties of these materials is shown in Table 1. Quartz sand conforming to BS 1881-131 and containing the grades *A* (2.36 mm–1.18 mm), *C* (0.60 mm–0.30 mm) and E (0.15 mm–0.09 mm), mixed in a ratio of 2:1:1 (by mass), were used for the aggregate component (David Ball Ltd., UK). A high range water-reducing admixture based on a polycarboxylate polymer was used as a super plasticiser (Grace Construction product Ltd, UK). The composition (by mass) of the RPC mix used for this study is presented in Table 2. The materials were mixed dry at a speed of ~120 rpm for 2 min in a forced action paddle mixer before increasing the speed to ~450 rpm for a further 2 min. The water and super plasticiser were premixed and added to the mixer incrementally with further mixing for 10–12 min until the mixture was consistent.

The mixture was used to cast prisms (with dimension 40 × 40 × 160 mm) in a steel mould and vibrated on a table for 3 min. The mould was covered to avoid immediate evaporation and laid in a room at temperature about 10 °C, in order to minimise the evaporation of water during mixture and setting in early age samples. Four combinations of treatments were applied in this study: (*A*) without pressure and cured in water; (*B*) without pressure and heat cured in a drying oven; (*C*) with pressure and cured in water; (*D*) with pressure and heat cured in a drying oven. A static pressure of 8 MPa was applied 5 h after casting, and all samples were demoulded after 2 days. After that a staged heat curing cycle was applied as follows: preheated at 40 °C for 2 h before increasing to 240 °C at a rate of 50 °C/h; cured at 240 °C for 48 h; decreased at 50 °C/h down to 40 °C. After this step, heat-cured samples were stored in water at 20 °C until testing. The specimens used to determine the compressive strength were 40 mm cubes in accordance with BS EN 196-1:2005. All strength data presented is the mean average for three representative samples.

A Philips XL30 field emission gun environmental scanning electron microscope (FEG-ESEM) was used. Micrographs were recorded using an Everhart-Thornley type Secondary Electron (SE) detector and a Back Scattered Electron (BSE) detector supplied by K. E. Developments. The FEG-ESEM operating conditions were 20 kV accelerating voltage, 4.0 spot size, and 10 mm working distance. Three representative sub-samples were clean fractured from bulk specimens of each treatment type (*A–D*), i.e. 12 in total. Sub-samples were mounted fracture face down and cold mounted under vacuum in 2-part epoxy resin. Mounted samples were ground using SiC paper (400, 600, 800 and 1200 grit) on 20 cm diameter wheels at a rotational speed of 250 rpm, followed by polishing using 6 μm then 1 μm diamond pastes. Polished samples were washed using acetone and dried under a hot air blower, followed by sputter coating with ~15 nm thick carbon using an Edwards 306 vacuum Coater. Representative micrographs were recorded for randomly selected aggregate particles and analysed using ImageJ 1.47v (National Institutes of Health, USA). Images were smoothed using a 2px median filter, before bracketing the upper/lower limits of the greyscale histogram by manually adjusting contrast/brightness. The porosity could then be segmented manually using the default threshold algorithm. On each SEM sample, three cross-sectioned aggregates were randomly selected and grids measuring 5 μm (thick) × 50 μm (wide) were applied from the aggregate surface at distances ranging from 0 to 75 μm (15 grids in total). The number and total area of pores were assessed using the 'analyse particle' tool. Porosity was defined by dividing the total area of particles with the area of grid. The mean

Table 1
Properties of cement, silica fume and GGBS used in this study.

Properties	Cement[a] wt.%	Silica fume[a] wt.%	GGBS[b] wt.%
Chemical			
SiO_2	20.09	>90.00	36.50
Al_2O_3	4.84	–	12.50
Physical			
Loss in ignition	2.36	<3	8.50
Specific gravity	3.15	2.20	1.00
Blain finesse	395	22,400	–

[c] Ground granulated blast furnace slag (Hanson, UK) [21].

[a] CEM I 52.5 Portland cement (Cemex, UK); [20].

[b] Condensed micro silica grade 940-D (Elkem, Switzerland) [20].