STUDY REPORT

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Reactive Powder Concrete

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Preface

The low flexural tensile strength of concrete has a number of undesirable consequences for its performance as an effective building material. These include the necessity for auxiliary steel reinforcement and the requirement for thick-sectioned members that are both aesthetically unappealing and consume significant quantities of aggregates. The aggregates are an increasingly scarce resource in many urban areas. An emerging technology with the potential to overcome these limitations is reactive powder concrete (RPC). RPC is a cold-cast cementitious material in which the mechanical properties of the composite matrix are improved by (i) suppression of the weak interfacial transition zone normally developed around the aggregate through improved particle packing and (ii) refinement of the hydrated paste microstructure by extensive use of pozzolanic silica and elevated temperature curing. Tensile capacity is provided by steel micro-fibres rather than conventional reinforcement. The result is a material with the potential for flexural strength up to 50 MPa and ductility and energy absorption values approaching those of steel. The low and non-connected porosity of RPC also render it extremely durable. RPC offers the possibility of building with concrete using slender members in a 'steel-like' fashion with improved seismic response, and may also find application in other areas as a pseudo-plastic cold ceramic.

Acknowledgments

Note

This report is intended for concrete technologists and others interested in the potential of RPC in the New Zealand context.

REACTIVE POWDER CONCRETE

REFERENCE

ABSTRACT

RPCs were produced in the laboratory to examine the effect of a number of process variables including water-to-binder (w/b) ratio, super-plasticiser dosage, curing regime and the choice of silica pozzolan, on the compressive and tensile strength of the hardened material. Compressive strengths in excess of 200 MPa and flexural strength of 15 – 20 MPa were routinely achieved using conventional concrete mixing and curing practices. Mechanical strength was observed to directly correlate with the spatial efficiency that the dry powders' constituents could be packed together. Consequently it is necessary for these powders to exist in distinct size classes with a wide separation between mean particle size diameters. This places a reliance on imported silica fume as an essential component of these mixes. The benefits conferred by the fume's extremely fine sub-micron particles and spherical shape are such that substitution by indigenous geothermal silica pozzolan is impractical.

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1. INTRODUCTION

'Reactive powder concrete' (RPC) is the generic name for a class of cementitious composite materials developed by the technical division of Bouygues, S.A. in the early 1990s. It is characterised by extremely good physical properties, particularly strength and ductility.[1] Table 1 compares the reported properties of RPC with those typically associated with conventional high performance concrete. While RPC is considerably more expensive to produce than regular concrete, its more isotropic nature and greater ductility make it competitive with steel, over which it has a significant cost advantage, for many structural applications. RPC beams can be designed with an equal moment capacity to steel beams at comparable mass and cross-sectional dimensions.[2]

Table 1. Properties of RPC vs conventional high performance concrete

Property	High performance concrete	RPC[3]
Compressive strength (MPa)	60 – 100	180 – 200
Flexural strength (MPa) [central-point loading]	6 – 10	40 – 50
Fracture energy (J/m^2) [ASTM C293]	140	1,200 – 40,000
Young's modulus (GPa)	23 -37	50 – 60

The key features of RPC mix design include very large Portland cement content, extremely low w/b ratios made possible by high dosages of the latest generation super-plasticisers, the presence of a high reactivity pozzolan (typically silica fume), and the incorporation of fine steel fibres for reinforcement. Conventional aggregate is completely replaced by a fine quartz sand with a particle size of between 150 and 425 μm. Consequently, calling the material a 'concrete' is somewhat of a misnomer and it more closely approximates a mortar. The incorporation of 'reactive powder' within the name reflects the fact that all the powder components in RPC react chemically following casting: the cement by conventional hydration, silica fume through pozzolanic reaction with the resulting calcium hydroxide and, albeit to a lesser extent, the quartz sand, by providing dissolved silica for the formation of further C–S–H gel. RPC subjected to high pressure steam curing also includes additional reactive silica to alter the CaO/SiO_2 ratio and favour the formation of tobermorite as the end-product of the hydration sequence.

In some respects, RPC resembles earlier attempts to increase the mechanical capacity of cement-based materials by eliminating in-homogeneity, such as Macro Defect Free (MDF) and, especially, Densified Small Particle (DSP) concretes.[4] However, RPC is unique in attempting to optimise the entire grain size distribution of the composite matrix in order to reach maximum compaction. In particular, there are five central design tenets for RPC:[5]

- enhancement of homogeneity by the elimination of coarse aggregates

- enhancement of the compacted density by optimising the granular mixture and optionally applying pressure before and during setting

- enhancement of the microstructure by heat treatment after hardening

- improved ductility through the incorporation of steel fibres

- maintaining mixing and casting procedures as close as possible to existing concrete industry practice.

1.1 RPC in New Zealand

One of the attractions of RPC as a construction material is the opportunities it offers for the improvement of seismic design with concrete structures.[6] These include simply taking advantage of the higher compressive and shear strengths possible to lighten floor systems and reduce column cross-sections, thus reducing inertial loads and permitting larger deflections of building or bridge frames within the elastic range. The high energy absorption characteristics of RPC may also allow improved post-elastic response of columns, beam-column joints and shear walls. These can be difficult to execute using conventional concrete techniques due to congestion of the necessary reinforcing steel.

Despite this potential, RPC has not received wide attention in New Zealand. Yang[7] reported on the production of laboratory mixes using domestic materials, but generally obtained compressive strengths of between 120 – 150 MPa, rather than the >200 MPa routinely claimed by promoters of the technology. Only one RPC structure is known to be extant in New Zealand: A 175 m pedestrian footbridge, consisting of 10 simply-supported spans, across the railway lines at Papatoetoe, Auckland.[8] The spans, produced in RPC under the tradename 'Ductal®' were cast by Bouygues' VSL subsidiary in Melbourne and shipped across the Tasman for erection on site.

1.2 Objective

The objective of the current study was to remove some of the barriers to the adoption of RPC technology in New Zealand by developing a degree of experience with its production. This included demonstrating that a quality product could be obtained with local constituent materials and establishing an independent benchmark of the performance levels achievable.

2. METHODS

2.1 Mix design

Two basic varieties of RPC were produced: A low temperature mix intended to be cured under ambient conditions as per conventional site practice, and a high temperature mix suitable for manufacturing operations such as pre-cast plants where steam curing facilities are feasible. The experimental mix designs developed are shown in Table 2 below. These are based on a combination of published compositions [1, 3, 5, 9, 10, 11] and adherence to the following principles:

- Pozzolanic silica is added in the stoichiometric quantity necessary to react with all the calcium hydroxide that would be produced assuming complete cement hydration. Using cement chemists' notation, the simplified hydration reaction is:[12, 13]

$$2C_3S + 6H \rightarrow C_3S_2H_3 + 3CH$$

$$2C_2S + 4H \rightarrow C_3S_2H_3 + CH$$
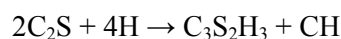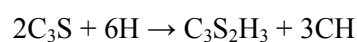
$$[\text{where } C = CaO; S = SiO_2; H = H_2O]$$

The $C_3S_2H_3$ is poorly crystalline, and essentially non-stoichiometric; accordingly it is more commonly referred to as C–S–H or calcium silicate hydrate. The calcium hydroxide [CH] produced by hydration occupies 20 – 25% of the cement paste by volume and makes no contribution to strength and durability. Addition of amorphous silica forms further desirable C–S–H at the expense of calcium hydroxide, according to the 'pozzolanic reaction':

$$CH + S + H \rightarrow C\text{–}S\text{–}H$$

- For mixes cured at 90ºC or higher, the CaO/SiO_2 ratio in the binder is reduced by the addition of further silica. This modifies the hydration sequence further, as shown below, resulting in a lower-lime C–S–H that ultimately converts to crystalline tobermorite [$C_5S_6H_5$], conferring higher strength to the hardened concrete:

$$(C_3S + C_2S) + S + H \rightarrow C_3S_2H_3 + CH + S \rightarrow C\text{–}S\text{–}H \rightarrow C_5S_6H_5$$
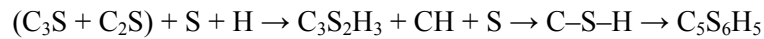
At these elevated temperatures, finely divided crystalline forms of silica are sufficiently reactive to act pozzolanically, so ground quartz flour is normally employed and highly siliceous aggregate can also contribute to this reaction.

- The volume of the binder (cement + silica fume + water) in the composite RPC matrix should exceed the void volume of the aggregate by at least 50% (i.e. each aggregate particle should 'float' in the matrix, rather than touch together as in a conventional concrete). This improves the probability of plastic deformation as a response to stresses such as autogenous shrinkage, rather than accommodation of strain via the formation of micro-cracks.

Table 2. RPC mix design (parts by mass)

Components	Low temperature curing	High temperature curing
Cement	1.00	1.00
Sand	1.10	1.10
Silica fume *or* pozzolan	0.25	0.23
Powdered quartz flour	Nil	0.39
Steel fibres [optional]	0.175	0.175
w/b ratio [as required for workability]	0.11 – 0.26	0.17 – 0.23
Super-plasticiser [% solids on cement]	0.6 – 1.6	1.9 – 2.5

2.2   Material selection

2.2.1 Silica pozzolan

A highly reactive silica pozzolan is an essential component of reactive powder concrete, performing three vital roles for which it needs the following properties:

1.   It must be sufficiently fine to pack closely around the cement grains, improving the density of the composite matrix and minimising the potential for voids between the particles.

2.   It should possess considerable pozzolanic activity, such that the non-cementing portlandite crystals [Ca(OH$_2$)] generated by hydration of the cement react with the silica to form additional C–S–H gel, reinforcing the binding of the composite.

3.   The particles should have a basically spherical shape to act as a lubricant within the fresh mix, improving its ability to flow and be cast into moulds.

Conventionally, the reactive silica used for RPC has been silica fume, which is an industrial by-product of the manufacture and purification of silicon, zirconia and ferro-silicon alloys in submerged-arc electric furnaces. Escaping gaseous SiO oxidises and condensates as extremely fine (0.03 – 0.2 μm) spherical particles of amorphous silica, neatly fulfilling the requirements listed above. One of the potential drawbacks of RPC production in New Zealand is the absence of a domestic source of silica fume: importing this material is an expensive proposition, both because of high demand and an inconveniently light bulk density of 200 – 300 kg/m^3, complicating shipping and handling.

However Microsilica 600, a geothermal silica sinter mined near Rotorua, has been widely used in conventional high performance concrete as a 1:1 replacement for silica fume, and an important part of the study was the evaluation of its suitability for use in locally-produced RPC. According to its producer: *"The NZ Standard NZS 3122:1995 'Specification for Portland and Blended Cements' classifies Microsilica 600 as a silica fume"* and *"Microsilica 600 meets the performance requirements of most international silica fume standards such as the Australian Standard AS358.2 'Silica Fume' and the Canadian Standard CSA-A23.5M86 'Supplementary Cementing Materials for Type U Silica Fume' "* .[14]

In addition to Microsilica 600, two alternative silica fumes were sourced for use as control materials from their suppliers, Australian Fused Materials (AFM), Rockingham, Western Australia and Simcoa Operations Pty, Kimerton, Western Australia. The AFM product is a white undensified fume arising from zirconia manufacturing, a variety that the literature suggests is most suited to use in RPC.[15] The Simcoa silica fume is a more common silicon-smelting by-product and has been densified, i.e. the primary silica particles are agglomerated into micro-pellets by aeration and the application of pressure. This increases the bulk density of the silica fume making transportation a more viable process. This Simcoa product has previously been imported into New Zealand, having been used in Firth-supplied high-performance concrete under the tradename Micropoz™.

The general properties of the silica pozzolans used in this study are given in Table 3 with their oxide composition shown in Table 4. The information is from the respective suppliers.

Table 3. Silica pozzolans used in this study

Pozzolan	Description	Surface area (m^2/g)	Bulk density (kg/m^3)
AFM SF-98 Australian Fused Materials, W.A.	Undensified silica fume [zirconia production by-product]	15	200 – 300
Simcoa SF Simcoa Pty, W.A.	Densified silica fume [ferrosilicon production]	22	500 – 700
Microsilica 600 Microsilica New Zealand	Natural amorphous silica [geothermal sinter]	undisclosed	610

Table 4. Chemical analysis of reactive powders used to produce RPC

Oxide (mass %)	High reactivity pozzolans			Cement
	Simcoa	AFM	Microsilica 600	Golden Bay GP
SiO_2	93.3	93.0	88.9	21.8
Fe_2O_3	0.1	0.4	0.6	2.2
Al_2O_3	0.2	0.2	4.3	3.6
TiO_2	0.1	0.02	1.2	n.d.
Na_2O	0.2	0.01	0.1	0.2
K_2O	0.4	0.01	0.5	0.4
MgO	0.4	0.01	< 0.1	0.9
SO_3	n.d.	n.d.	0.1	2.1
CaO	0.2	0.01	0.3	65.4
P_2O_5	0.1	0.3	0.05	n.d.
$ZrO_2 + HfO_2$	n.d.	4.2	n.d.	n.d.
L.O.I.	4.7	1.0	5.0	4.4

2.2.2 Cement

Due to the very high cement factor, the choice of cement can be an important factor in the performance of RPC. Based on published practice, the ideal cement has a high C_3S and C_2S (di- & tri-calcium silicate) content and very little C_3A (tri-calcium aluminate). This is understandable because C_3A has little intrinsic value as a binding agent and is primarily included in cement due to its role as a flux during the calcination process. Consequently, most RPC made with commercially-available cement employs an ASTM Type V 'sulfate-resistant' blend, which is formulated specifically for low C_3A content. Because this variety is not routinely manufactured in New Zealand, standard Golden Bay Type GP cement was substituted instead. As shown in Table 5, the C_3A content of this cement is not excessively high, despite not being formulated specifically for sulfate resistance.

Table 5. Bogue composition of Portland cements

Cement	C_3S	C_2S	C_3A	C_4AF
ASTM Type V	40	40	4	10
Golden Bay GP	58	21	7	6

2.2.3 Quartz fines

For RPC mixes designed to be cured at temperatures exceeding 90ºC, including autoclaving at elevated pressures, additional silica is necessary to modify the CaO/SiO_2 ratio of the binder. In these cases a powdered quartz flour with a mean particle size of 10 – 15 μm was employed. The quartz flour was supplied by Unimin New Zealand Ltd.

2.2.4 Fine aggregate

The majority of mixes were produced using 'J61W' from Industrial Sands Ltd, Swanson. This is a near-white high purity silica sand widely used for foundry casting and mould-making with a near mono-sized particle size distribution (Table 6). A limited number of trials were also carried out with a more common-place concrete aggregate, an alluvial deposit of rhyolitic Waikato river sand, quarried by Winstone Aggregates.

Table 6. Particle size grading (percentage passing) of aggregates used

Sieve size	75 µm	150 µm	300 µm	600 µm	1.18 mm	2.36 mm	4.75 mm	FM
J61W	1	5	76	98	100	100	100	1.21
Waikato river sand	0	3.6	19.8	60.2	93.4	100	100	1.05

2.2.5 Super-plasticiser

The very low w/b (cement + silica fume) ratios used in RPC are only possible because of the fluidising power of high-quality third generation super-plasticising agents. On the advice of Sika NZ Ltd, Sika ViscoCrete-5 was selected as the most suitable for use. This is described as an aqueous modified carboxylate, designed specifically for ultra-high water reduction applications such as self-compacting concrete. To minimise any air-entrainment effects due to the high-dosage rates necessary, 1% Pronal 753S defoaming agent, also supplied by Sika, was added to the super-plasticiser before use.

2.2.6 Steel fibres

To enhance the RPC ductility, some mixes were produced with micro-fibres of straight carbon steel wire, 13 mm in length and 0.2 mm in diameter, with a minimum on-the-wire tensile strength of 2,000 MPa. These were supplied by BOSFA (Bekaert OneSteel Fibres, Australasia).

2.3 Sample production

The RPC mixes were produced in 1 litre batches using a Hobart N-50A epicyclic mixer compliant with the requirements of ASTM C 305 'Mechanical mixing of hydraulic cement pastes and mortars of hydraulic consistency'.[16] The mixing protocol adopted is shown in Table 7. The extended mixing time is necessary both to fully disperse the silica fume, breaking up any agglomerated particles, and to allow the super-plasticising agent to develop its full potential. The mixing is unusual in that a distinct conversion point is observed several minutes after the final aliquot of water is added, during which the dry-balled ingredients suddenly coalesce to plastic flowing mix.

At the conclusion of the mixing period, the workability of the mix was assessed according to the ASTM C 1437 'flow table' test[17] and the air content estimated by means of ASTM C 185.[18] From the fresh RPC, 50 mm cube specimens for determination of compressive strength and 250 x 25 x 25 mm beams for determination of flexural strength were compacted into moulds by hand tamping in two layers. The specimens were allowed to harden in their moulds for 24 hours at 21°C and 95% relative humidity, before being stripped and subjected to one of the curing regimes specified in Table 8.

Table 7. Procedure for production of RPC mixes

Mixing protocol	Elapsed time (minutes)
Lightly grind cement and silica fume to break-up agglomerates	–
Add all dry powders and aggregate	0
Start mixing	½
Add 87% of water and 50% of super-plasticiser	3
Add steel micro-fibres [if used]	5
Add remainder of water and super-plasticiser	8
Stop mixing and cast test specimens	30

The majority of specimens were cured in tap water at the specified temperature and ambient pressure. However, some mixes designed for high-temperature curing (i.e. those containing quartz fines to modify the CaO/SiO_2 ratio of the binder) were autoclaved in a steel pressure vessel at 160ºC, corresponding to a steam pressure of about 0.5 MPa above atmospheric pressure.

Table 8. Curing regimes

Mix type	Curing regime	Duration	Post-curing treatment
No quartz addition	21ºC in water	1,3,7,28 days	Tested immediately
	65ºC in water	7 days	stored at 23ºC / 65% RH until test
Including quartz fines	90ºC in water	48 hours	stored at 23ºC / 65% RH until test
	160ºC / 0.7 MPa	48 hours	stored at 23ºC / 65% RH until test

2.4   Test methods

The compressive strength of the cured RPC cube specimens was determined with an Avery 1,800 kN capacity testing machine, applying the load at 72 kN/min. The flexural strength of the beams were determined by third-point loading according to Japanese Concrete Institute standard JCI-SF4[19] using an Instron testing machine to provide a constant cross-head deflection of 1/1,500 of the span per minute. The ductility of the beams was estimated by measuring the flexural toughness, defined as the total area under the load-deflection curve of the beam to the point where deflection equals 1/500 of the span.

3.   RESULTS

Thirty-five unique RPC compositions were produced to evaluate the effect of silica pozzolan and aggregate choice, w/b ratio, super-plasticiser content, steel fibre addition and curing regime. The individual mix designs are given in Table 9 and the fresh concrete properties, hardened density, air-content and compressive strength results are summarised in Table 10. Table 11 shows the flexural strength and flexural toughness results. These results are examined in detail in the following discussion.

Table 9. RPC mixes produced

Mix Series	Mix Parameters							
	Label	w/c ratio	w/b ratio	SP (% cement)	Sand	Silica Fume	Quartz Fines	Steel Fibres
Influence of w/b ratio **(low-temperature curing)**	WB 1	0.14	0.11	0.74%	J61W	Simcoa	-	-
	WB 2	0.16	0.13	0.74%	J61W	Simcoa	-	-
	WB 3	0.19	0.15	0.74%	J61W	Simcoa	-	-
	WB 4	0.21	0.17	0.74%	J61W	Simcoa	-	-
	WB 5	0.24	0.19	0.74%	J61W	Simcoa	-	-
	WB 6	0.26	0.21	0.74%	J61W	Simcoa		
Influence of Superplasticiser Dosage **(simple mixes)**	SP 1	0.18	0.14	0.63%	J61W	Simcoa	-	-
	SP 2	0.18	0.14	0.74%	J61W	Simcoa	-	-
	SP 3	0.17	0.14	0.84%	J61W	Simcoa	-	-
	SP 4	0.17	0.14	0.95%	J61W	Simcoa	-	-
	SP 5	0.18	0.14	1.61%	J61W	Simcoa	-	-
Fibre Addition	FM	0.19	0.15	0.74%	J61W	Simcoa	-	Y
Material Substitution	AFM 1	0.14	0.11	0.84%	J61W	AFM	-	-
	AFM 2	0.16	0.13	0.84%	J61W	AFM	-	-
	AFM 3	0.19	0.15	0.84%	J61W	AFM	-	-
	MS 1	0.19	0.15	0.84%	J61W	Microsilica	-	-
	MS 2	0.21	0.17	0.84%	J61W	Microsilica	-	-
	MS 3	0.24	0.19	0.84%	J61W	Microsilica	-	-
	MS 4	0.26	0.21	0.84%	J61W	Microsilica	-	-
	WR 1	0.19	0.15	0.74%	W.R. sand	Simcoa	-	Y
	WR 2	0.19	0.15	0.74%	W.R. sand	Simcoa	-	Y
Influence of w/b Ratio **(high SP dosage)**	SP 6	0.15	0.12	1.61%	J61W	Simcoa	-	-
	SP 7	0.18	0.14	1.61%	J61W	Simcoa	-	-
	SP 8	0.19	0.15	1.61%	J61W	Simcoa	-	-
Mixes Including Quartz Fines **(high temperature curing)**	QZ 1	0.21	0.17	1.89%	J61W	Simcoa	Quartz	-
	QZ 2	0.23	0.19	1.89%	J61W	Simcoa	Quartz	Y
	QZ 3	0.27	0.20	1.89%	J61W	Simcoa	Quartz	-
	QZ 4	0.27	0.17	1.89%	J61W	Simcoa	Microsilica	-
	QZ 5	0.21	0.17	1.89%	J61W	AFM	Quartz	-
	QZ 6	0.23	0.19	1.89%	J61W	AFM	Quartz	Y
	QZMS 1	0.27	0.22	1.89%	J61W	MS600	Quartz	-
	QZMS 2	0.28	0.23	2.50%	J61W	MS600	Quartz	-
	QZMS 3	0.32	0.26	2.49%	J61W	MS600	Quartz	-
Ultra-high Temperature Curing	UHT 1	0.21	0.17	1.89%	J61W	Simcoa	Quartz	Y
	UHT 2	0.21	0.17	1.89%	J61W	AFM	Quartz	Y

Table 10. Summary of RPC test results

| Mix Label	Mix Properties				Compressive Strength							
	Flow	Hardened Density	%Air	D_M/D_O	23ºC			65º/90ºC			160ºC	
					1	7	28	3	7	28	3	28
WB 1	104	2400	5.4	0.859	39	126	175	-	173	-	-	-
WB 2	112	2419	3.2	0.865	45	131	180	-	189	-	-	-
WB 3	120	2430	1.3	0.869	42	132	182	193	197	196	-	-
WB 4	138	2413	0.6	0.863	38	127	178	-	190	-	-	-
WB 5	140	2365	1.2	0.846	27	95	143	-	161	-	-	-
WB 6	148	2289	3.1	0.819	22	87	99	-	128	-	-	-
SP 1	110	2448	1.3	0.876	41	129	174	-	189	-	-	-
SP 2	112	2415	2.6	0.864	36	131	180	-	196	-	-	-
SP 3	120	2460	0.8	0.880	23	136	186	184	205	211	-	-
SP 4	118	2388	3.7	0.854	11	136	168	-	197	-	-	-
SP 5	119	2389	3.4	0.854	0	120	161	-	188	-	-	-
FM	-	2521	1.9	0.86	-	158	212	-	239	-	-	-
AFM 1	-	2401	5.3	0.86	-	117	171	-	180	-	-	-
AFM 2	-	2420	3.1	0.87	-	130	164	-	193	-	-	-
AFM 3	-	2415	1.9	0.86	-	125	173	-	175	-	-	-
MS 1	-	2250	8.6	0.80	-	38	43	-	42	-	-	-
MS 2	-	2371	2.4	0.85	-	132	148	-	168	-	-	-
MS 3	-	2338	2.4	0.83	-	111	130	-	137	-	-	-
MS 4	-	2305	2.5	0.82	-	79	84	-	91	-	-	-
WR 1	-	2510	2.5	0.86	-	115	150	-	169	-	-	-
WR 2	-	2536	1.3	0.86	-	125	173	-	185	-	-	-
SP 6	-	2421	3.5	0.87	-	124	156	-	199	-	-	-
SP 7	-	2399	2.9	0.86	-	120	161	-	188	-	-	-
SP 8	-	2359	3.7	0.84	-	117	159	-	179	-	-	-
QZ 1	-	2389	2.5	0.86	-	-	-	205	201	213	-	-
QZ 2	-	2477	1.8	0.86	72	145	198	226	227	231	252	258
QZ 3	-	2458	2.1	0.85	65	133	162	206	211	204	232	235
QZ 4	-	2289	2.2	0.84	-	-	-	184	-	-	-	-
QZ 5	-	2403	1.9	0.86	-	-	-	205	-	-	-	-
QZ 6	-	2511	0.5	0.87	45	118	144	219	210	221	261	249
QZMS 1	-	2250	5.6	0.81	-	18	42	36	61	65	-	-
QZMS 2	-	2290	3.1	0.82	-	71	89	75	91	89	-	-
QZMS 3	-	2310	0.9	0.83	-	68	104	63	96	100	-	-
UHT 1	-	2489	1.5	0.84	-	-	-	-	-	(fired to 325ºC)		271
UHT 2	-	2519	0.3	0.85	-	-	-	-	-	(fired to 325ºC)		290

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3.1   Effect of silica pozzolan choice

Figure 1 demonstrates the affect that varying the silica pozzolan employed has on the compressive strength of the hardened RPC mix, considered as a function of the w/b (cement + reactive silica) ratio. The data shown relates to the simplest mix compositions, without quartz fines and at a constant super-plasticiser dosage, that have been cured at a mildly-elevated temperature (65ºC) for 7 days to accelerate the achievement of near-ultimate strength. It is evident from the results that an optimal w/b ratio exists that is unique to each pozzolanic silica type, and that Microsilica 600 gives a significantly lower peak strength at a higher water demand.

The existence of an optimal w/b ratio can be readily understood: mixes with insufficient water are stiff and difficult to compact into moulds adequately, resulting in a hardened concrete with entrapped air voids. Figure 2 confirms that mix workability typically increases with increased water content. Conversely, mixes with higher w/b ratios are more susceptible to autogenous shrinkage, which creates voids because C–S–H gel (i.e. hardened cement) occupies a lesser volume than the equivalent quantity of dry cement powder and water. These more fluid mixes are also more susceptible to entraining small air bubbles due to the folding action of the mixing process. RPC mixes appear particularly prone to this problem due to their heavily super-plasticised nature and the cohesiveness resulting from the abundance of fine powder present.

That the compressive strength of the RPC produced fundamentally reflects particle packing can be seen from a consideration of Figure 3. This plots compressive strength against a synthetic 'packing efficiency' parameter, defined as the measured density of the hardened RPC mixes expressed as a ratio of the 'optimal density'. Optimal density is defined as the weighted average density of the solid constituents packed together at 100% efficiency (i.e. with absolutely no gaps between them). The compressive strength is very clearly observed to positively correlate with the packing efficiency parameter, with the best results obtained for mixes that most closely approach the ideal ratio of 1.0.

Sphere packing theory suggests that packing tightness of powders will be maximised in the case of multiple particle sizes where a clear separation exists between the particle size classes. This criterion is fulfilled in those RPC mixes containing silica fume, as witnessed by Figure 4. The median diameter of the silica fume particles (ca. 0.3 µm) is approximately an order of magnitude finer that the cement particles (ca. 15 µm), which are in turn about an order of magnitude finer than the aggregate used. Adherence to this granular size class differentiation has been cited as an important factor in the performance of reactive powder concretes.

In contrast, mixes with Microsilica 600 substituting for silica fume have a clear deficit of material finer than 1 µm, with an obvious overlap in size classification between these particles and the cement grains, as demonstrated in Figure 5. This clash of particle size, and the commensurately poorer packing that results, seems likely to be the main contributor to the far weaker strengths achieved with mixes using Microsilica as the pozzolanic silica. The sample of Microsilica 600 used to produce the RPC appears coarser, and thus worse in this respect, than the particle size distribution specified in the supplier's literature. However, it is acknowledged that this inference is based on a single laser diffraction measurement and that this technique cannot distinguish between dispersed particles or agglomerates and is also subject to substantial bias where particles deviate from an ideal spherical geometry.[20] Therefore any apparent difference may not be completely reliable.
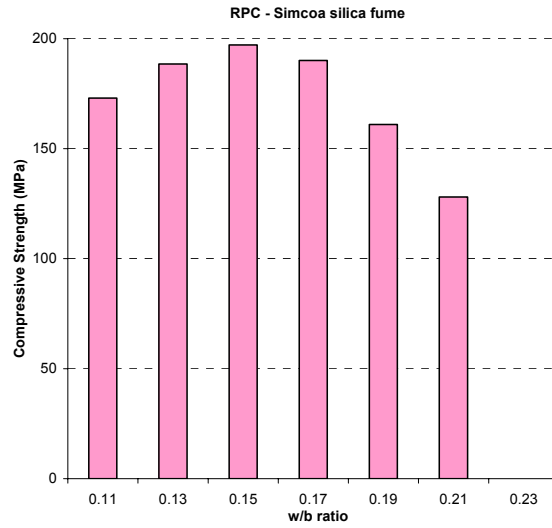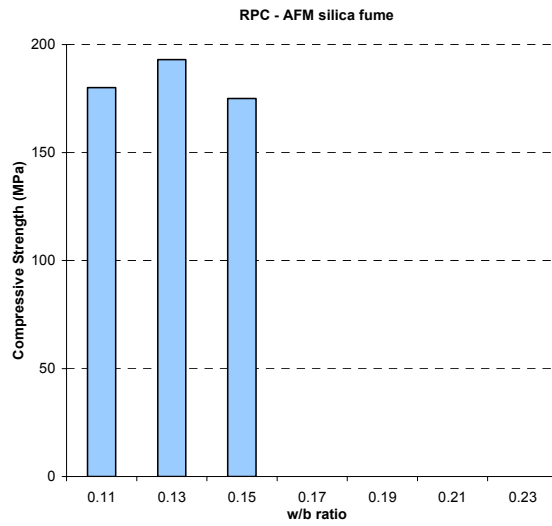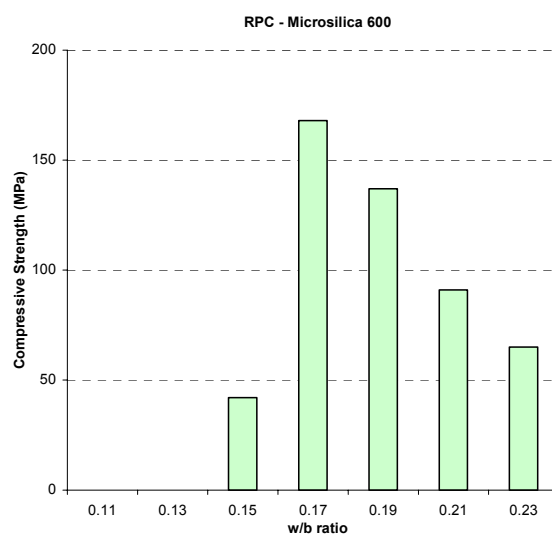
Figure 1.   Influence of silica pozzolan choice on RPC performance
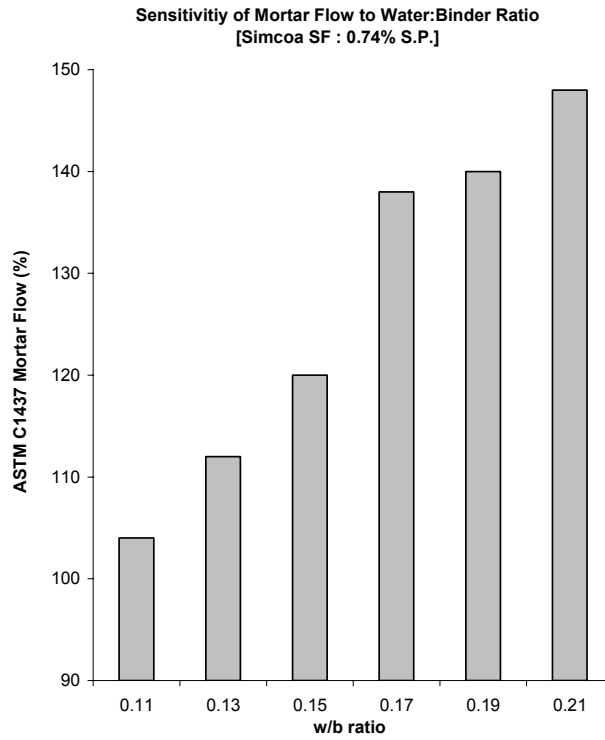
Figure 2.   RPC workability as a function of w/b ratio
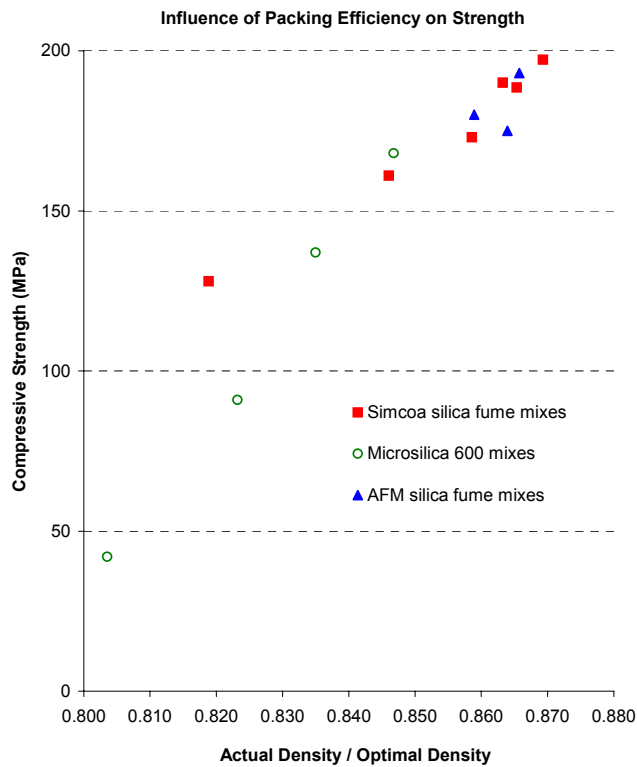

Figure 3.   Influence of packing efficiency on compressive strength
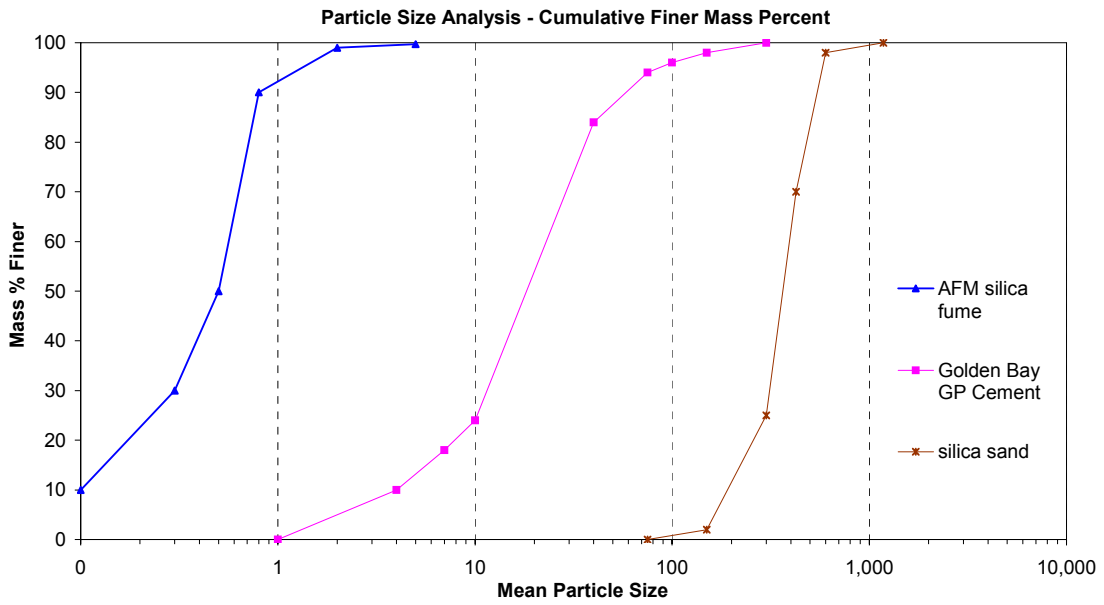
Figure 4.   Particle size analysis of reactive powders for mixes containing silica fume
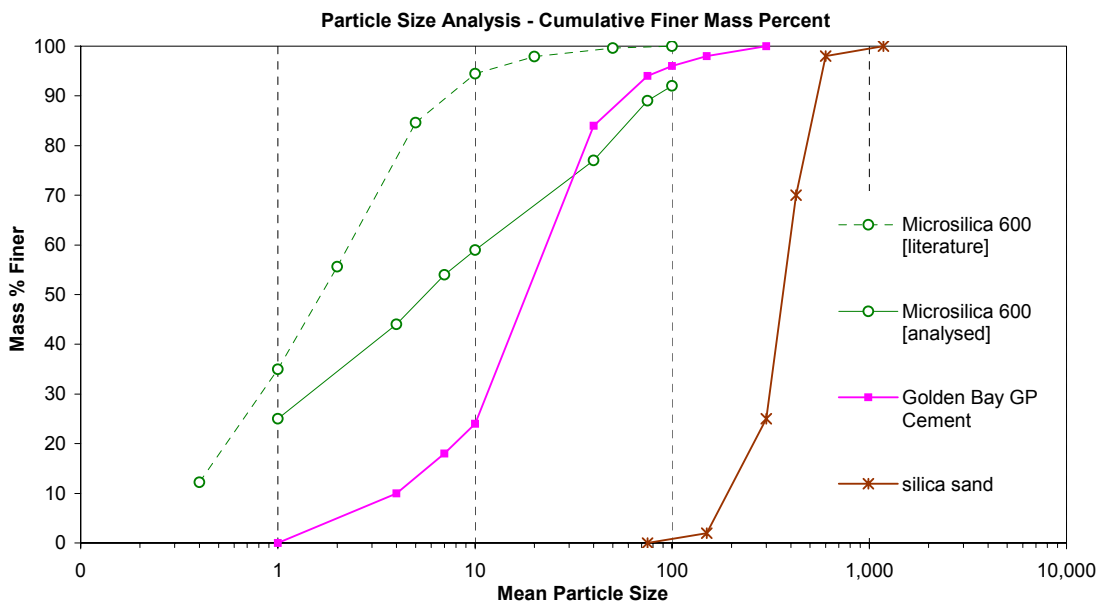

Figure 5.   Particle size analysis of reactive powders for mixes containing Microsilica 600

The particle shape of the silica pozzolans is examined in Figures 6, 7 and 8, which show lower and higher magnification scanning electron photomicrographs of each material. The densified nature of the Simcoa silica fume is very obvious, with the primary particles agglomerated into large and approximately spherical clumps of material (Figure 6). The surface of these agglomerates resolves into the individual primary particles with nominal diameters of around 0.3 μm. In contrast, the AFM silica fume is far more dispersed (Figure 7), with only occasional agglomerates of irregular shape and size. The individual particles are less uniform in size than the Simcoa material, but their almost perfectly spherical character is very in evidence. The lubrication benefits of this characteristic were apparent during the testing with mixes containing AFM consistently more workable than equivalent mixes with the other silica pozzolans at the same w/b ratio and super-plasticiser content. Despite this additional workability benefit, the Simcoa silica fume appeared to give slightly better hardened properties, contrary to the suggestion that the undensified materials are generally preferred for RPC production.

The relatively coarser particle size of Microsilica 600 is evident in Figure 8 and also the fact that the individual particles are, at best, angular to sub-rounded fragments and chards rather than spheres. This is likely to provide a further contribution to sub-optimal packing and also helps explain particular difficulties with the rheology of these mixes. Many of the Microsilica RPC compositions were observed to be thixotropic, i.e. the viscosity of the plastic mix was related to the shear force applied. In this case the relationship was one of inverse proportionality, with the mix often satisfactorily fluid when at rest but stiffening to a near-solid consistency as shear was applied. Obviously this makes physical mixing with an epicyclic mixer difficult and consequently the w/b ratio of the Microsilica mixes needed to be significantly increased to ensure fluidity relative to those containing silica fume. It seems reasonable to interpret this phenomenon as the angular Microsilica 600 grains 'locking-up' the matrix of cement, silica and aggregate particles. On the other hand, the more spherical silica fumes do act as ball-bearings allowing the mix to flow.

As a general observation, the rheological properties of these mixes were complex and, except for a limited number of simple compositions, not easily characterised by standard workability tests such as hydraulic mortar flow. Optimisation of mix properties for commercial production would clearly necessitate more sophisticated techniques.
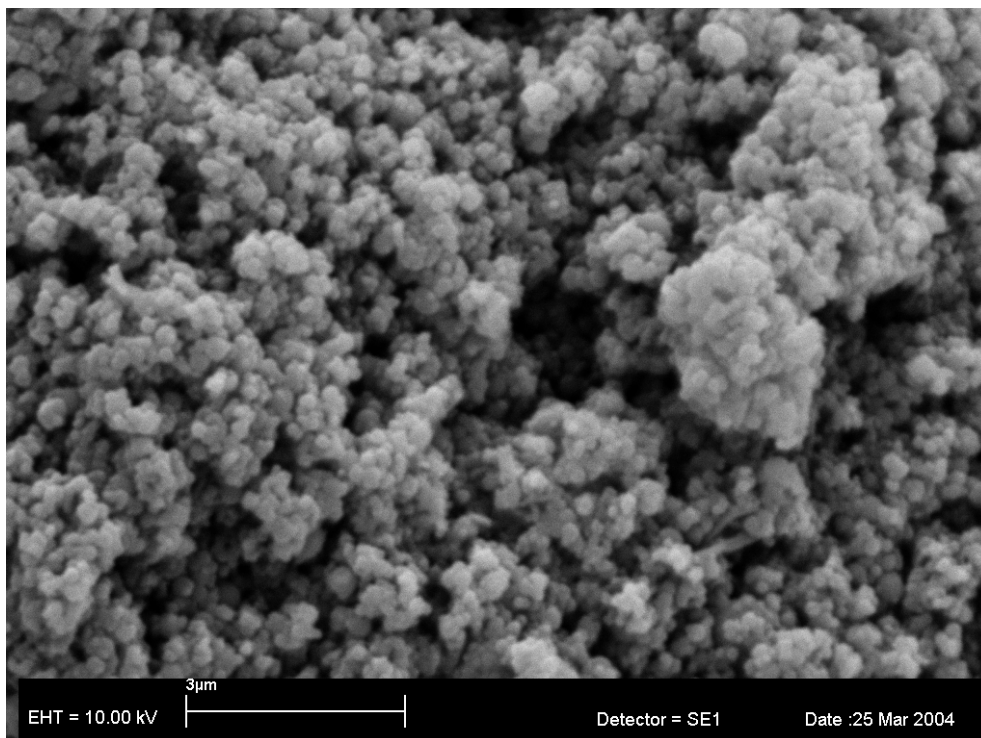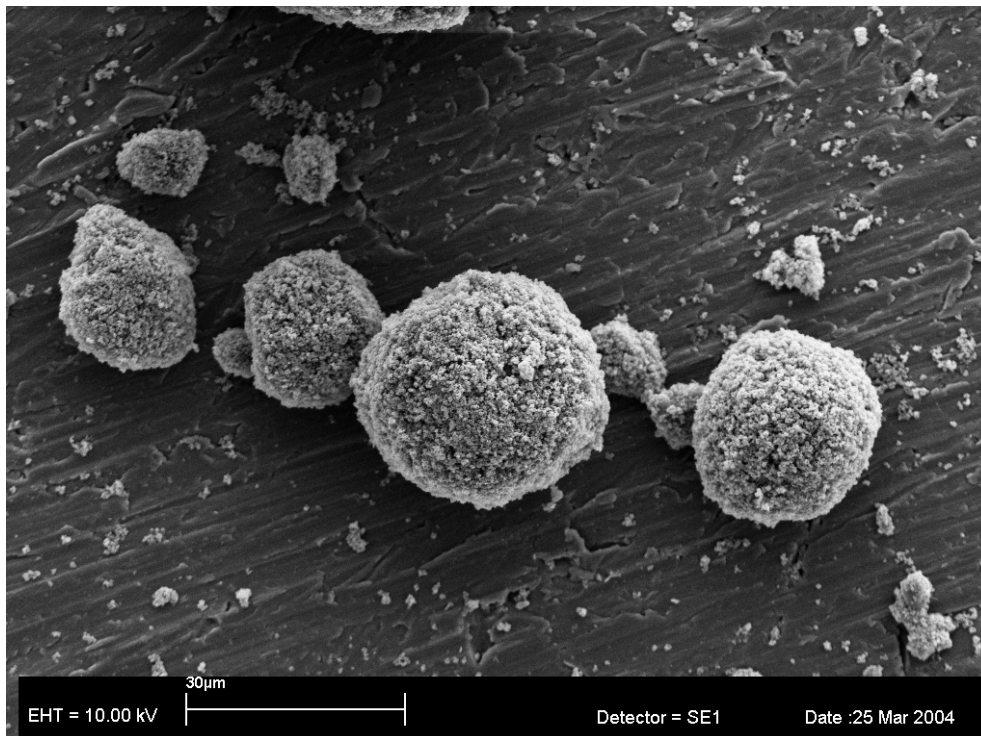
Figure 6.   Scanning electron micrographs of Simcoa silica fume (densified)
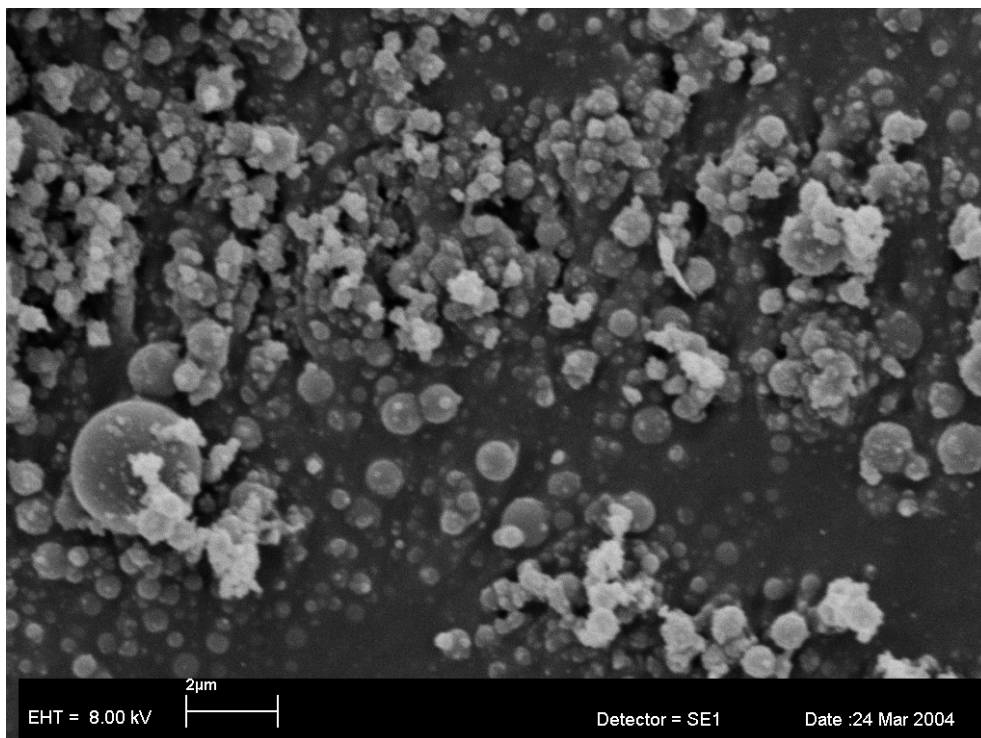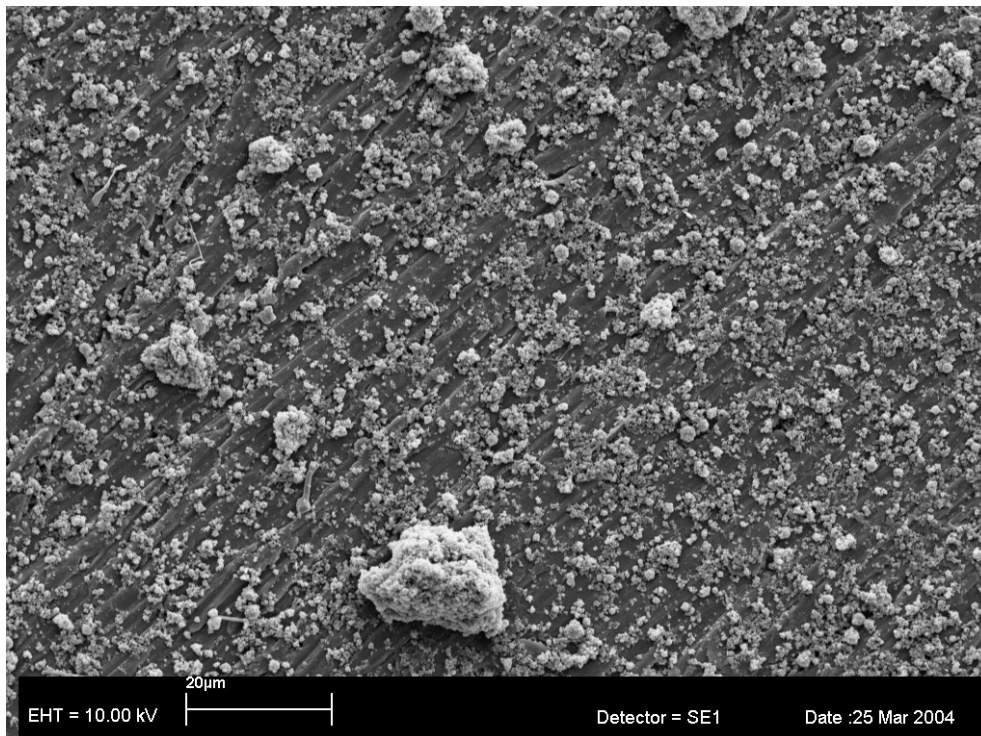
Figure 7. **Scanning electron micrographs of AFM silica fume (undensified)**
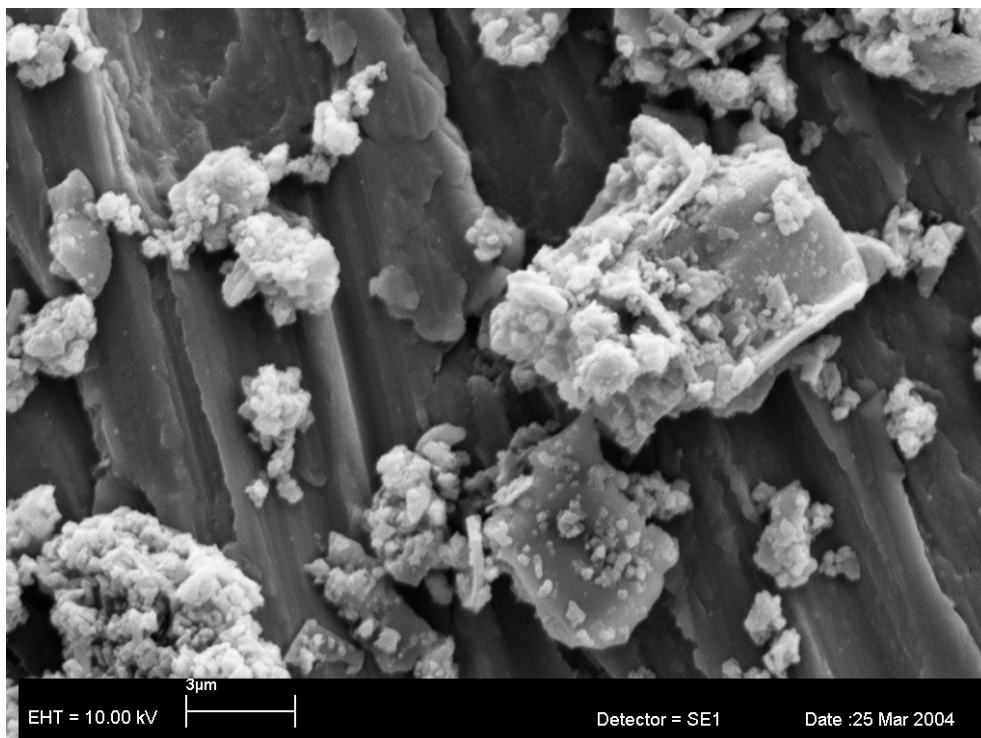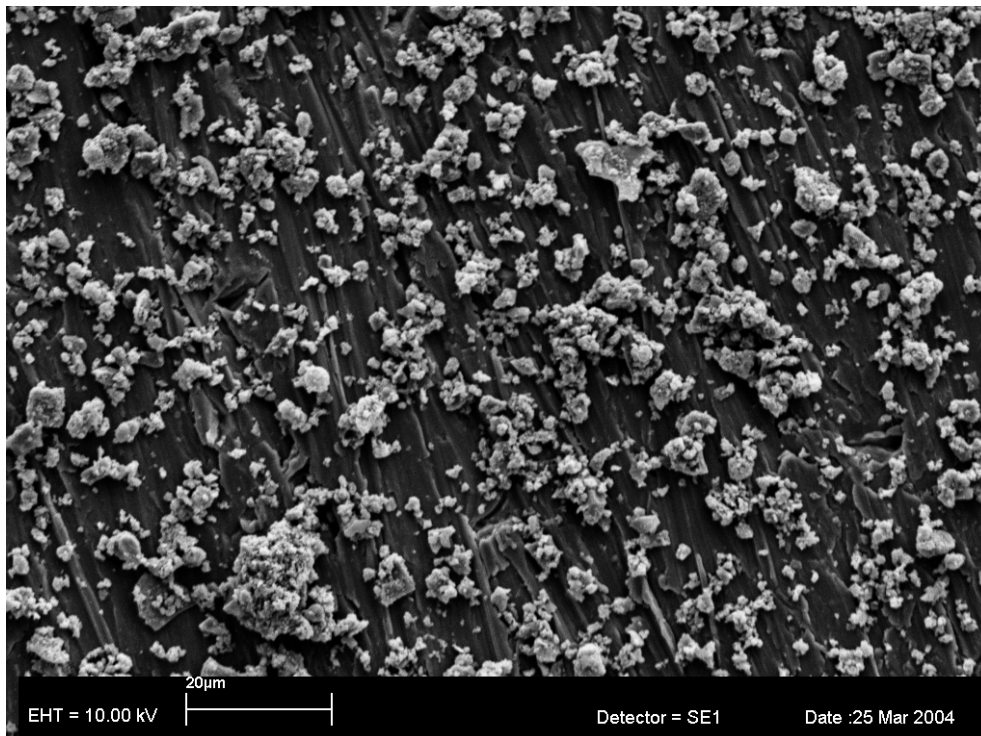
16

Figure 8.   Scanning electron micrographs of Microsilica 600

3.2   Effect of super-plasticiser dosage

Production of a cohesive and flowing RPC mix required extremely large quantities of super-plasticiser compared to high-performance or even self-compacting concrete. Sika recommend ViscoCrete-5 dosages of 0.1 – 0.3% solids by weight of cement for these applications. In contrast, a minimum dosage of about 0.6% was required to allow an RPC mix to 'convert' as described earlier. Dosages as high as 2% were necessary for the more demanding mixes containing both quartz fines and steel fibre.

Beyond the fairly sharply-defined minimum quantity necessary to get the mix to convert (i.e. proceed from a dry-balled state to a cohesively flowing material), additional super-plasticiser appeared to have little influence on mix rheology. This is demonstrated by Figure 9, which shows the fairly consistent flow of a simple Simcoa silica fume mix with a constant w/b ratio and varying super-plasticiser dosages from 0.6 to 1.6 %. In general, particle size, shape and total water content appeared to be much more important controls on workability.
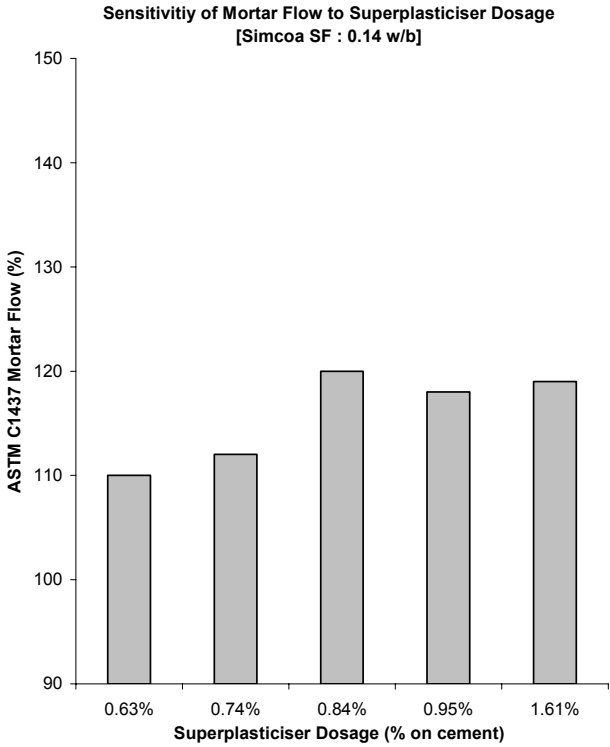
Figure 9.   The effect of super-plasticiser dosage on RPC mortar flow

Similarly, within broad limits the super-plasticiser content has little affect on the ultimate compressive strength of the hardened RPC mixes as indicated by Figure 10, although there is some suggestion of a small peak in performance. As expected, increasing super-plasticiser dose severely retards the rate of initial hardening, with dosages in excess of 1% typically taking more than 24 hours to gain significant strength. Once initial set is achieved, strength gain is observed to be dramatic however.

Effect of Superplasticiser Dosage
[Simcoa SF : 0.14 w/b ratio]

Figure 10.   The effect of super-plasticiser dosage on RPC compressive strength

3.3   Influence of curing regime

Figure 11 and Figure 12 demonstrate the affect of differing curing conditions and duration on two series of mixes; the first a simple mix containing 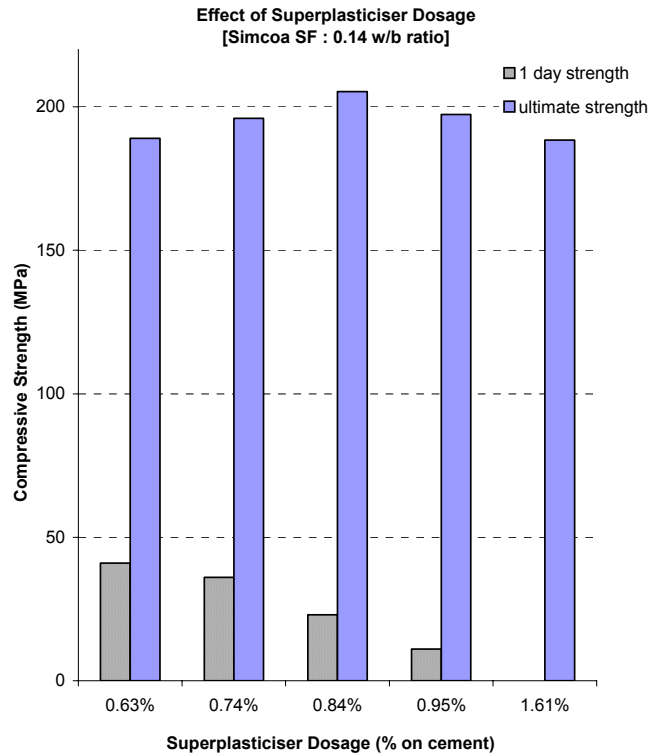only silica fume, cement and aggregate, and the second also containing additional quartz fines to modify the CaO/SiO_2 ratio and alter the hydration sequence for high temperature curing, as previously described. The simple mix (Figure 11) gains strength continuously over a 28 day curing period at 23ºC and also shows a marked beneficial effect from elevated curing at 65ºC for 7 days, which confers a 25 MPa strength margin at 28 days (211 MPa vs 186 MPa). Understandably, 23ºC wet-curing of the modified quartz mix (Figure 12) yields less impressive strength because the quartz dilutes the cement factor with a material that is effectively inert at this temperature. Forty-eight hours curing at 90ºC proves sufficient to compensate for this effect, and the economy in cement may well outweigh the more difficult curing regime for commercial RPC, which is likely to be produced in a factory environment such as a pre-stressing yard. Autoclave curing at 160ºC provides a further boost in strength to approximately 230 MPa, but the need for a pressure vessel to carry out this operation probably precludes its practical application.

X-ray diffraction analysis of the hardened RPC demonstrated that the silica fumes used were effective pozzolans because no Ca(OH)2 hydration reaction products were detected. A typical low-temperature (23ºC) curing XRD pattern is shown in Figure 13. Interestingly, the high-temperature (90º and 160ºC) patterns are very similar, with no evidence of tobermorite crystallising from the low-lime modified C–S–H gel as expected (Figure 14). This suggests that the quantity or reactivity of the quartz fines added requires further investigation if the strength of the RPC is to be maximised. Both XRD patterns demonstrate peaks due to di- and tri-calcium silicate (C2S & C3S), indicating that a significant fraction of the cement is unhydrated and simply acts as an inert filler in the 10 μm diameter size range. This is, of course, consistent with the extremely low water-to-cement ratio of these materials.

Influence of Curing Regime - Simple Mixes
[Mix SP3 : Simcoa SF : 0.14 w/b : 0.84% SP]

- 65ºC - 7 days
- 23ºC - continuous
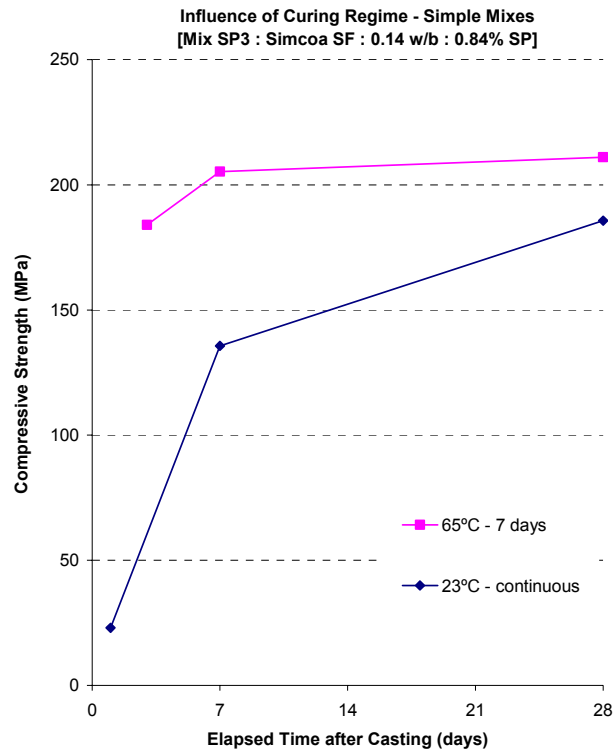
Figure 11.   Strength gain by low temperature curing

Influence of Curing Regime - Heat-treated Mixes
[Mix QZ3 : Simcoa SF : Quartz fines : 0.20 w/b : 1.89% SP]

- 160ºC [0.7 MPa] - 48 hours
- 90ºC - 48 hours
- 23ºC - continuous
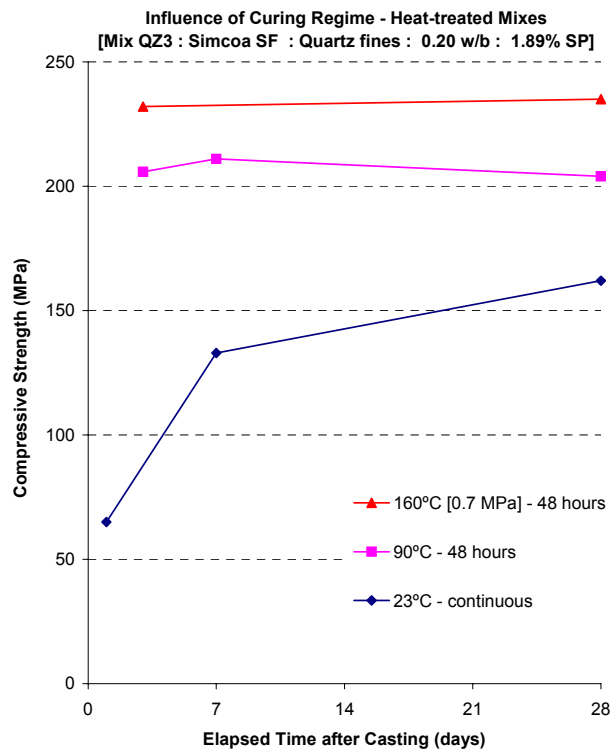
Figure 12.   Strength gain by higher temperature curing [modified CaO/SiO$_2$ ratio]

20

Operations: X Offset 0.075 | Background 0.174,1.000 | Import
Unoriented powder - File: 9197-A.raw - Type: 2Th/Th locked - Start: 5.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 60. s - Temp.: 25 °C (Room) - Time Started: 0 s - 2-Theta: 5.000 ° - Theta: 2.500 °
00-046-1045 (*) - Quartz, syn - SiO2
01-085-1378 (C) - Hatrurite, syn - Ca3(SiO4)O
00-033-0302 (*) - Larnite, syn - Ca2SiO4
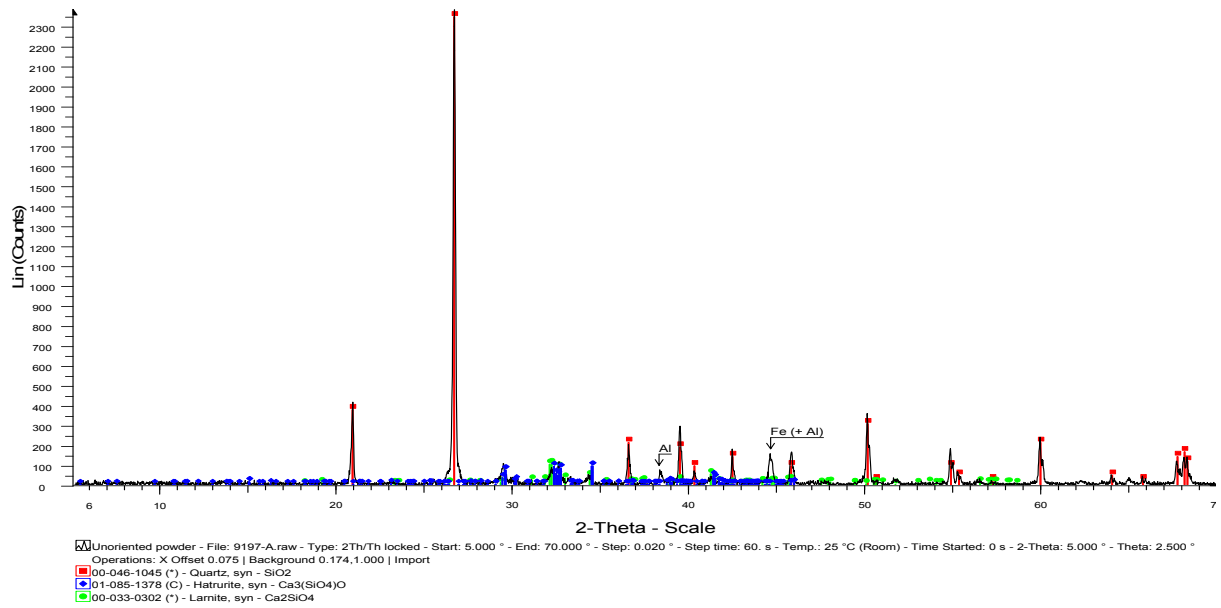
Figure 13. XRD pattern of RPC mix 'QZ3' after curing at 23ºC for 28 days

Operations: X Offset 0.108 | Background 0.174,1.000 | Import
Unoriented powder - File: 9197-HT.raw - Type: 2Th/Th locked - Start: 5.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 60. s - Temp.: 25 °C (Room) - Time Started: 0 s - 2-Theta: 5.000 ° - Theta: 2.500 °
00-046-1045 (*) - Quartz, syn - SiO2
01-086-0402 (C) - Hatrurite, syn - Ca3SiO5
00-033-0302 (*) - Larnite, syn - Ca2SiO4
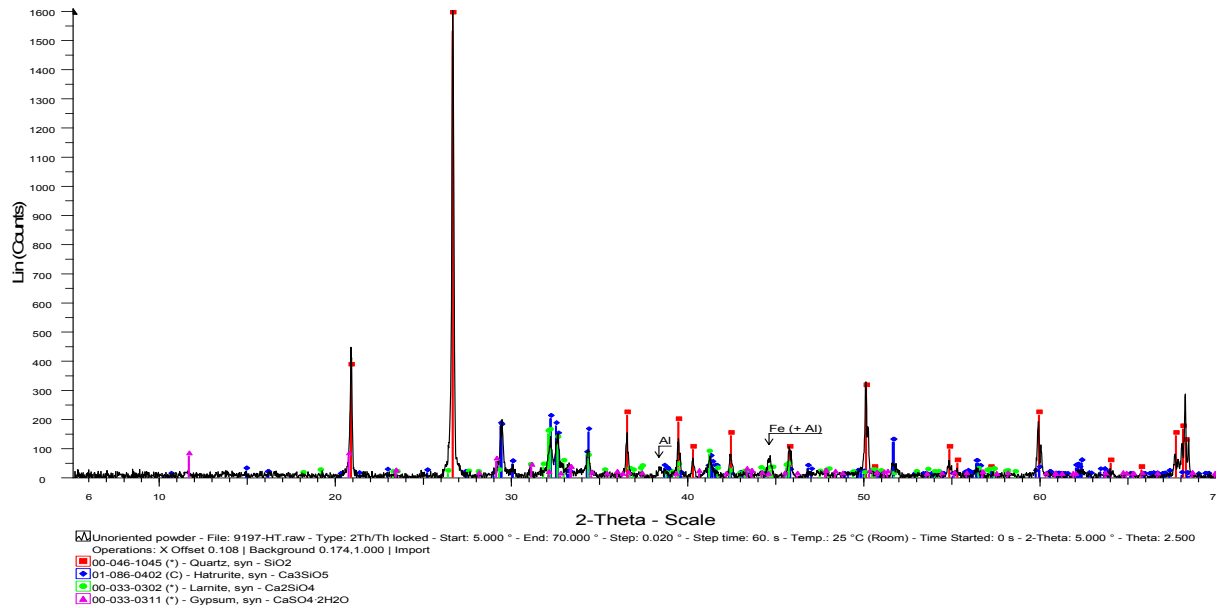00-033-0311 (*) - Gypsum, syn - CaSO4·2H2O

Figure 14. XRD pattern of RPC mix 'QZ3' after curing at 160ºC for 48 hours

A limited number of pyro-processing trials were carried out in which the hardened RPC was subsequently heat-treated at 250º – 400ºC. This technique has been claimed to be capable of producing compressive strengths of 600 – 800 MP. Little success was achieved, with most specimens fracturing as the vapour pressure generated by free water and the thermally decomposed cement hydrate exceeded the material's tensile capacity. The highest strength obtained by conditioning in this fashion was 290 MPa.

3.4 Quartz addition

As noted, the addition of quartz fines to the RPC composition allows for both effective accelerated high-temperature curing and a reduction in cement content due to the need to adjust the CaO/SiO$_2$ ratio in the C–S–H phase. Despite the need for extremely heavy super-plasticiser dosages to fluidise the mix, this gave satisfactory results with the silica fumes. As demonstrated by Figure 15, compressive strengths exceeding 200 MPa were achieved after 48 hours curing at 90ºC. As before, however, substitution of Microsilica 600 for the silica fume was unsatisfactory, resulting in a decrease in strength of 50% or more. This meant the performance of these mixes often failed to exceed that achievable with a conventional high-performance concrete (Figure 16). Again this is probably attributable to the inefficiency of particle packing: Figure 17 demonstrates how the quartz fines introduce even further congestion of particle diameters in the 5 – 20 μm range.

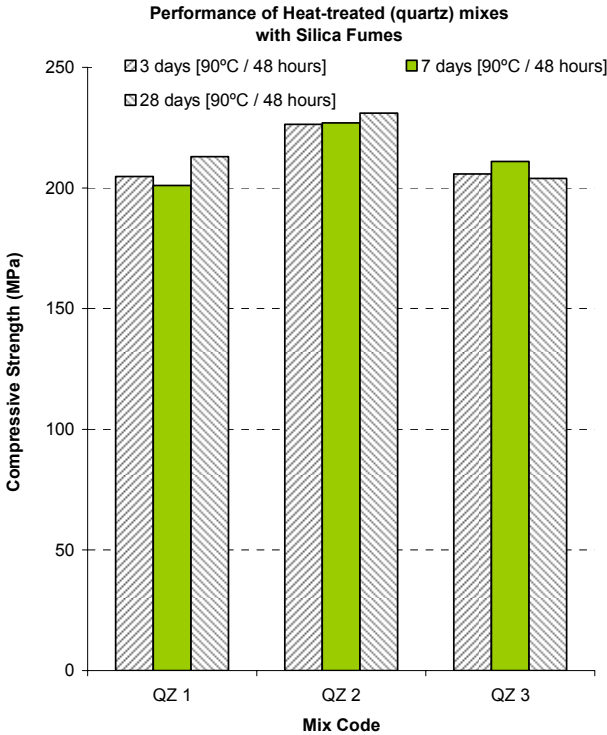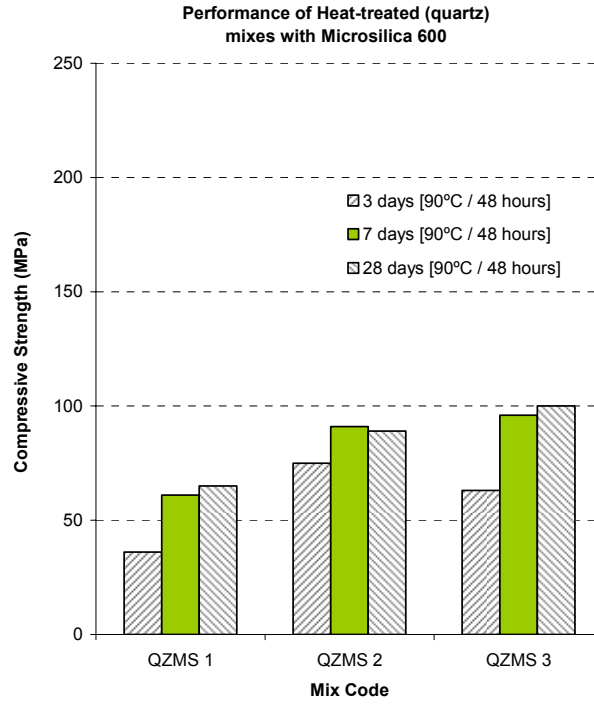**Figure 15.   Compressive strength of quartz mixes containing silica fume**

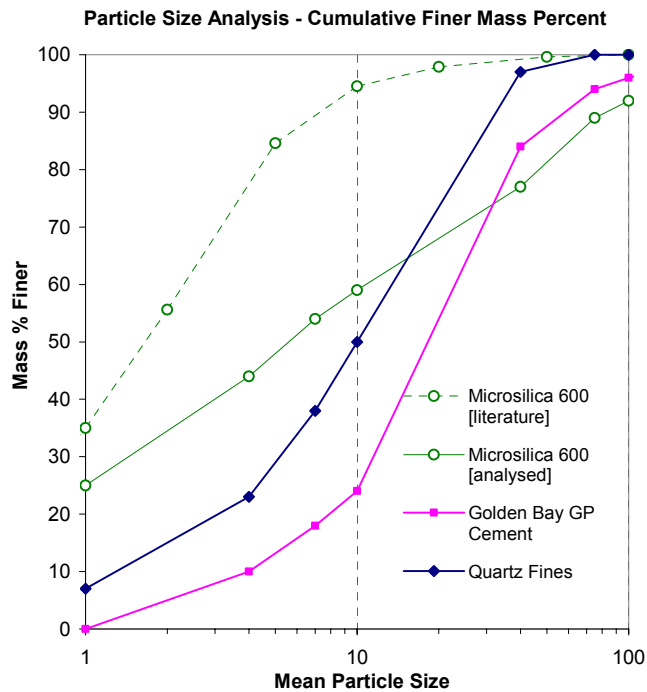**Figure 16. Compressive strength of quartz mixes containing Microsilica 600**

Figure 17. Comparison of particle size of quartz fines, cement and Microsilica 600

3.5 Steel fibre addition

Steel fibres are added to the RPC mix primarily to improve the normally poor tensile strength of composite cementitious materials. However, they were also generally observed to provide a marked improvement in the measured compressive strength of otherwise equivalent mixes to which they were added: Figure 18 gives a typical comparison for mixes with and without fibre. The improved compressive strength likely reflects the contribution of the steel fibre to the tensile capacity of the RPC, given the accepted view that concrete under a uniaxial compressive load fails because of lateral strain induced by Poisson's ratio effects.[12,13] However, it was also noted that the presence of the fibre typically produced a denser and more readily compacted mortar with less entrapped air, attributes that are also beneficial to the material's mechanical properties.
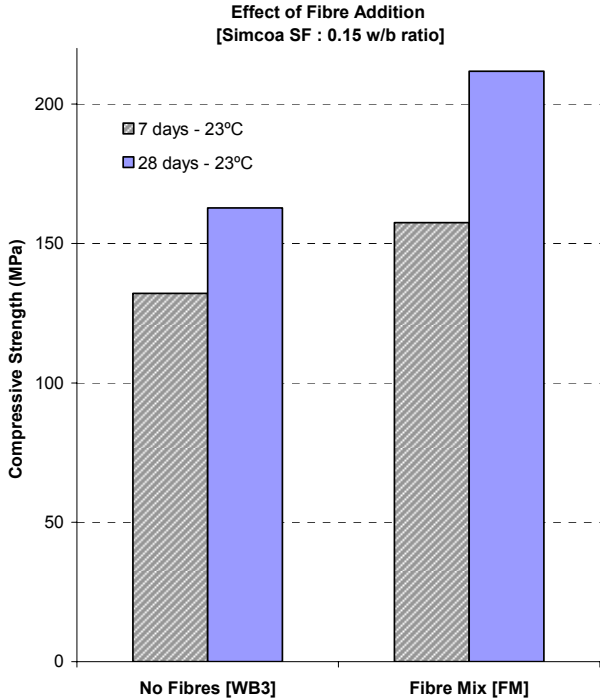
Figure 18.   The affect of fibre addition on the compressive strength of an RPC mix

The best recorded performance of a steel fibre-reinforced RPC beam specimen under third-point loading is demonstrated in Figure 19. The stress – deflection curve exhibits fairly linear behaviour up to first cracking, a significant strain-hardening phase until the ultimate flexural load of 19 MPa is achieved and an extended post-ultimate load strain softening phase during which the beam retains a significant proportion of its structural capacity. Other flexural strength and flexural toughness results are summarised in Table 11.

Figure 19.   Stress – deflection curve of RPC beam tested in flexure (third-point loading)

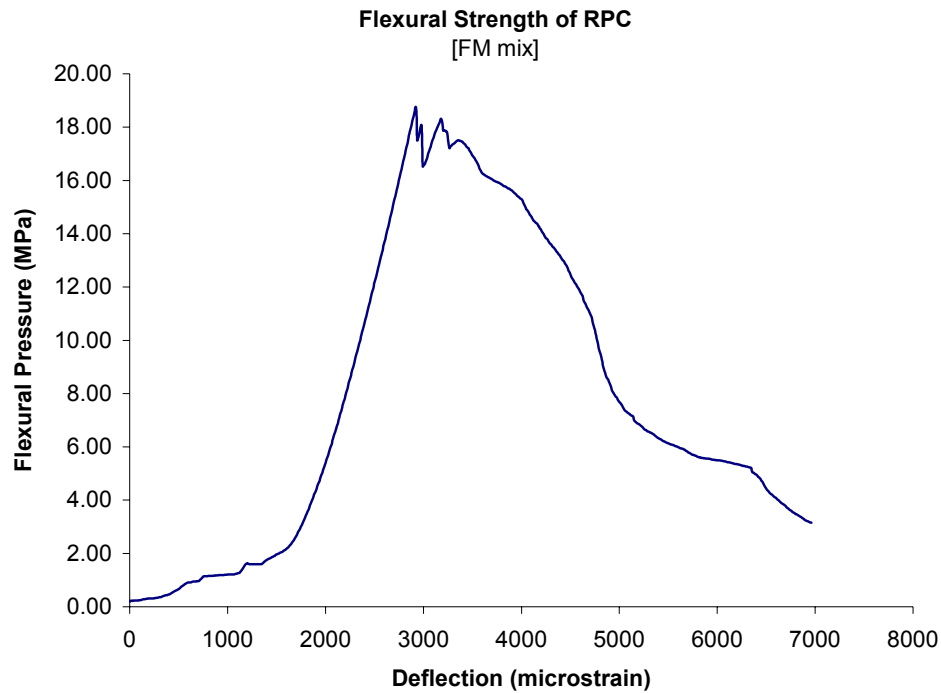In comparison with some literature values,[1,2] the achieved flexural strength and flexural toughness results are disappointing, failing to approach the reported ultimate load capacities of 25 – 50 MPa. However, these literature results are typically achieved on notched specimens using a central-point loading technique (e.g. ASTM C 293), which tightly constrains the point at which the specimen can fail. In contrast, the third-point loading method used in this study allows the beam to fail at its weakest point in the centre third of its span, tending to minimise the measured strength. Published results using both central-point and third-point loading indicate the latter method will give flexural strengths that are lower by at least 50%.[10] Other researchers have noted that the percentage of fibres in relation to the strength of an RPC matrix is low, making it essential to have the fibre correctly optimally orientated to the imposed load. In the case of the tested beam specimens it was observed that the fibre distribution was often poor, with a particular tendency to segregate due to gravity before initial set was achieved. This appears to be a clear instance where optimisation of the mix rheology is necessary to take full advantage of the material's potential.

Table 11. Measured flexural strength and flexural toughness of fibre-reinforced mixes

Mix	F1		QZ2F		QZ6F		WR1		WR2	
cement	1		1		1		1		1	
silica fume	0.25 Simcoa		0.23 Simcoa		0.23 AFM		0.25		0.25	
quartz fines	-		-		0.39		-		-	
sand	1.1		1.1		1.1		1.1 Waikato R		0.89 Waikato R	
steel fibres	0.175		0.175		0.175		0.175		0.175	
w/b	0.15		0.19		0.19		0.15		0.15	
curing regime	21°	90° / 48 hr	21°	90° / 48 hr	21°	90° / 48 hr	21°	90° / 48 hr	21°	90° / 48 hr
Flexural Strength (MPa) - 3rd point loading JCI-SF4										
at 7 days	13.9	-	11.2	-	-	-	-		-	
at 28 days	18.8	n/a	14.8	16.2	13.1	14.6	12.7	n/a	13.2	n/a
Flexural Toughness (J)										
at 7 days	684	-	576	-	-	-	-		-	
at 28 days	837	n/a	714	895	594	778	421	n/a	431	n/a

3.6   Sand substitution

High purity quartz sands such as J61W (used for the majority of mixes here) are expensive and comparatively rare aggregates, imposing a significant cost-burden on a commercial RPC. To investigate alternatives, two RPC mixes were produced using Waikato river sand, a rhyolitic alluvial deposit routinely used for conventional ready-mixed concrete production. The results are summarised in Table 12. Mix 'WR1' used a straight 1:1 replacement of the silica sand, resulting in a measurable degradation of the mechanical properties of the RPC, particularly flexural strength. This was considered to be potentially attributable to the higher voids content of the river sand giving rise to a mix in which the aggregate particles were in contact, resulting in a greater tendency for micro-cracking in response to autogenous shrinkage. Consequently, the mix was repeated with the paste volume (cement + silica fume + water) increased to 2.13, equivalent to the paste:voids volume ratio in all the earlier mixes. This produced an improvement in the compressive strength of the hardened mix but little change in the flexural performance, suggesting that homogeneity of the cement matrix is a critical parameter for compressive strength, but that performance in flexure more closely depends on intrinsic aggregate quality. The potential of a quartz aggregate to contribute to the pozzolanic reaction, giving a more effective aggregate – cement matrix bond under the shear stress produced in bending tests, may also be significant.

Table 12. Summary of test results for mixes containing Waikato river sand

Mix code	Volume ratio: paste to voids	Compressive strength (MPa)	Flexural strength (MPa)	Flexural toughness (J)
WR1	1.71	169	12.7	421
WR2	2.13	185	12.9	431

4. CONCLUSIONS

Fluid RPC mixes with hardened compressive strength exceeding 200 MPa and third-point flexural strength approaching 20 MPa can be readily produced using mixing, casting and curing techniques familiar to the pre-stressed concrete industry and unremarkable constituent materials. In addition to a sufficiently reactive pozzolanic silica source, performance of RPC strongly depends on the optimisation of packing of the powder constituents, necessitating tightly-constrained size classes for the granular materials, with mean particle diameters differing by at least an order of magnitude. This is exemplified by the ideal of silica fume, cement and aggregate particles of approximately 0.1, 20 and 300 μm diameter respectively. Unfortunately, the dictates of such a particle size distribution effectively disqualify the indigenous Microsilica 600 amorphous silica as a suitable pozzolanic material, due to both the coarser mean particle size and the characteristically angular particle shape. Commercial production of RPC would therefore require the importation of silica fume, which is comparatively expensive because of demand and the difficulty of handling a low density powder. However, the results reported here do suggest there is no reason to prefer undensified silica fume over the more easily transportable densified variety. Estimations by Yang suggest that even with imported silica fume, RPC is cost-comparable with other high performance concrete when measured in the more rational terms of monetary cost for a given structural capacity, rather than dollars per cubic metre of material. Nevertheless, it is clear that RPC is a niche product best suited to applications that take advantage of its properties for lighter and more durable structures.

Presuming the imperative exists for further development of RPC with New Zealand materials, the experience gained here suggests that successful optimisation of mix design will require the use of a purpose-designed rheometer, similar to those developed for self-compacting concrete. The high content of fine granular powders, combined with heavy super-plasticiser dosage, results in fresh RPC mixes possessing an almost tar-like consistency. They are far more cohesive than conventional mortars or concrete and often show marked thixotropic properties, making workability evaluations by standard flow/slump techniques unfeasible except for the simplest and most idealised of compositions. While empirical judgement is of some assistance, the difficulties experienced in this study with segregation of the steel fibre after casting demonstrate that mix design needs to be considered on a more rational basis.

One of the most frequent claims for RPC is that it increases the mechanical homogeneity of concrete, i.e. its tensile strength much more closely approaches its compressive capacity. While the flexural strengths measured for the samples produced in this study are comparatively high for cementitious materials, they do not support the isotropic claim. The ultimate flexural loads were typically about 10% of the compressive value, a fairly characteristic ratio for normal concrete. Regardless of arguments about flexural measurement techniques, this ratio indicates significant potential for improvement in the mixes produced. The proponents of the 'Multi-Scale Concept' of fibre reinforcement[21,22] hold that fibre with relatively large length:diameter ratios, as employed here, mostly assist in stitching together macro-cracks, which primarily increases the capacity of the concrete post-ultimate load (i.e. its ductility). Significant improvements in tensile strength require a high proportion of short fibre in the matrix, which serve to bridge micro-cracks at the point of initiation and prevent them coalescing to form said macro-cracks. This suggests that the mechanical performance of the RPC might be improved by replacing some of the sand with mineral micro-fibres such as wollastonite, a possibility for further investigation.

Other potential avenues for extending the current work are suggested by the x-ray diffraction evidence that much of the very high cement content (typically $800 - 1,000$ kg/m^3) remains unhydrated and merely acts as an inert filler. Some fraction of the material can presumably be

substituted for another granular powder with similar size characteristics at a significant cost benefit; Microsilica 600 would seem to be an obvious candidate. A further critical examination of the viability of substituting concrete sands of more common mineralogy and size distribution for the high purity mono-sized quartz sand ordinarily considered essential for RPC may also be worthwhile. The preliminary results with Waikato river sand indicate the impact may not be strikingly detrimental, providing the tenets of granular class separation and void to paste volume ensuring 'floating' aggregate are respected. Once a mix design is finalised, other macroscopic engineering properties such as post-cure drying shrinkage, swelling and creep should also be verified.

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