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The microstructure and mechanical properties of blended cements hydrated at various temperatures

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Abstract

The development of microstructure and compressive strength of three blended cement pastes hydrated at temperatures ranging from 10°C to 60°C is described. The replacement materials were pulverised fuel ash (PFA), volcanic ash (VA), and ground, granulated blast furnace slag (GGBFS), and the blended cements had the same compositions as those reported in previous studies. The cement pastes were cured under water and tested for compressive strength at various time intervals over a period of 1 year. The only blended cement paste that had substantially improved strength compared with the neat cement paste was that containing blast furnace slag, especially at 60°C. Selected samples were examined by backscattered electrons in scanning electron microscopy (SEM). Generally, the microstructures of the pastes cured at 60°C showed greater apparent porosity than those cured at 10°C. The mechanism of hydration of the various blended cements is discussed.

Keywords: Curing temperature; Backscattered electron imaging; Hydration products; Microstructure; Compressive strength

1. Introduction

The adverse effect of high temperatures, from such diverse origins as hot weather or accumulated heat of hydration, on the hydration of cement pastes can be divided into two parts. First, the microstructure has increased porosity [1,2], and second, the ultimate degree of hydration of the anhydrous cement phases is reduced [2–4]. Overall, elevated curing temperatures tend to have negative consequences, leading to inferior mechanical properties and a cement that is more prone to chemical attack by water or other agents, hence reducing the useful life of a structure. In blended cements, the use of a suitable replacement material can fulfil two roles. First, it may participate in the cementitious hydration products, and second, it may modify the characteristics of the hydrated cement fraction [5]. The improvements brought about by the use of some replacement materials, for example, ground, granulated blast furnace slag (GGBFS) and pulverised fuel ash (PFA), to such properties of the cement as permeability, compressive strength, durability, and reduced heat of hydration, are well known [6–10]. Other advantages that accompany the use of waste materials might include savings in consumption of energy and natural resources. The hydration of the blended cements is not yet fully understood, however, and further research is needed to provide information to increase the knowledge of this field.

Latent hydraulic replacement materials, such as GGBFS, and pozzolanas, such as PFA and volcanic ash (VA), react to different extents [5] with the CH produced by the cement, thereby introducing some microstructural and chemical advantages, e.g. improved strength and durability. There are major differences in the physical and chemical characteristics of these replacement materials. Diamond [11] pointed out that in PFA, there are compositional variations between particles within the same ash, while natural pozzolanas, silica fume and GGBFS, are considered to have a relatively more homogeneous chemical composition and internal structure. The chemical homogeneity (apart from the effects of chemical composition and amorphous fraction) has to have some influence on the reactivity patterns of the replacement materials, hence on some of the properties of the blended cements, e.g. the microstructural characteristics. This effect might be more pronounced in blends incorporating PFA, as there are some particles that behave inertly due to their local chemical and phase composition.

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Publications reporting the effect of curing temperature on the microstructure of composite cement pastes studied by backscattered electron images (BEI) are not common. Pastes incorporating 30% GGBFS made using a $w/s = 0.5$ were studied at 23°C and 70°C by Cao and Detwiler [12]. They showed microstructural images of cement paste at degrees of hydration of 30% and 70%, using nonevaporable water as the indicator of hydration. Assessing the degree of hydration from this parameter leaves uncertainty as to the relative degree of hydration of the slag and the cement, as they respond differently to variation in temperature. The degree of hydration of the cement phases has been reported to be higher at increased temperatures in the early stages, but later, the situation was reversed [2,5], and the same for the compressive strength [13,14]. In this article, we present results related to previous publications [2,5], dealing with the degree of hydration of the anhydrous phases in neat cements and blended cement pastes cured at temperatures ranging from 10°C to 60°C. They will be referred to as Part I [2] and Part II [5], respectively. In the present article, we report a study of the same blended cement pastes, including the evolution of compressive strength over time for three blended cement pastes cured at five different temperatures. In addition, a microstructural study of the blended cement pastes was undertaken to investigate the mechanism of hydration at different temperatures and the effect of the replacement materials on the cement hydration.

2. Experimental

2.1. Cements

Three blended cements made from two ordinary Portland cements (OPCN and OPCS), described in Parts I and II, were studied: OPCN with 30% PFA, OPCN with 60% GGBFS, and OPCS with 22% VA (called OPCVA). Their chemical composition and phase characterisation have been reported previously [2,5]. All of the materials were kindly supplied by the Mexican company, Cementos Apasco, except the slag, which was provided by Frodingham Cement, Scunthorpe, UK.

2.2. Curing and hydration

The blended cements were mixed with freshly boiled distilled water at a water:solid ratio of 0.5. They were cast as 10 × 10 × 100 mm bars in Teflon moulds. The paste specimens were cured for 24 h at 20 ± 1°C and 98% RH in a mist room. The bars were demoulded, grouped according to the set of moulds, and randomly selected to form subgroups for the different curing temperatures. Each group of bars was immersed in a plastic container filled with distilled water, sealed, and submerged in a water bath at constant temperature. The temperatures were 10°C, 20°C, 30°C, 40°C, and 60°C with a variation of ± 1°C. Samples were randomly extracted from the water baths for mechanical testing after periods of 1, 3, 7, 14, 28, 180, and 365 days.

2.3. Characterisation

For the compressive strength measurements, four bars were randomly extracted and cut in two sets of halves. From a set of halves, four microcubes of approximately 10 × 10 × 10 mm were carefully cut from the centre of the bar and outwards. The cubes were crushed in a Mayes machine to obtain the ultimate compressive strength. For the microstructural studies, selected samples cured at various temperatures after 1 year of hydration were prepared for BEI in the scanning electron microscope [SEM] (from the set of other halves as referred to above). Details about the sample preparation for both techniques were provided in Part I.

3. Results and discussion

3.1. Compressive strength of the microcubes

Results of compressive strength development for the three blended cements are shown plotted against the square root of time in Fig. 1. The values reported are the mean of four cubes. The standard deviation was always less than 15% with respect to the mean. A general feature observed in all three blended cements was that at the early stages of hydration, the compressive strength was higher with increasing temperature. This is similar to what has been reported in Parts I and II for the degree of hydration of the anhydrous cement phases and the compressive strength of the neat cement pastes.

In the case of the blended cement paste incorporating PFA (Fig. 1A), there appeared to be a high temperature inversion in the strength values. After 180 days, the lowest values were those for cement pastes hydrated at 40°C and 60°C, and the highest, for those hydrated at 20°C and 30°C. The rate of increase in the compressive strength of the PFA cement paste hydrated at 10°C suggests that if the experiment had been continued for a second year, it might have given the strongest cubes. It was previously observed in Part II that for the same range of temperatures, the pozzolanic reaction had the slowest development at 10°C. This inversion in compressive strength with respect to increasing temperature was also noted in the neat cement pastes for compressive strength development and degree of hydration of alite (the main anhydrous cement phase). For the PFA blends, the compressive strengths after hydration for 1 year were always below those of the neat cement pastes at all five temperatures studied. Results for experiments conducted at 10°C, 30°C, and 60°C are shown in Fig. 2A. This

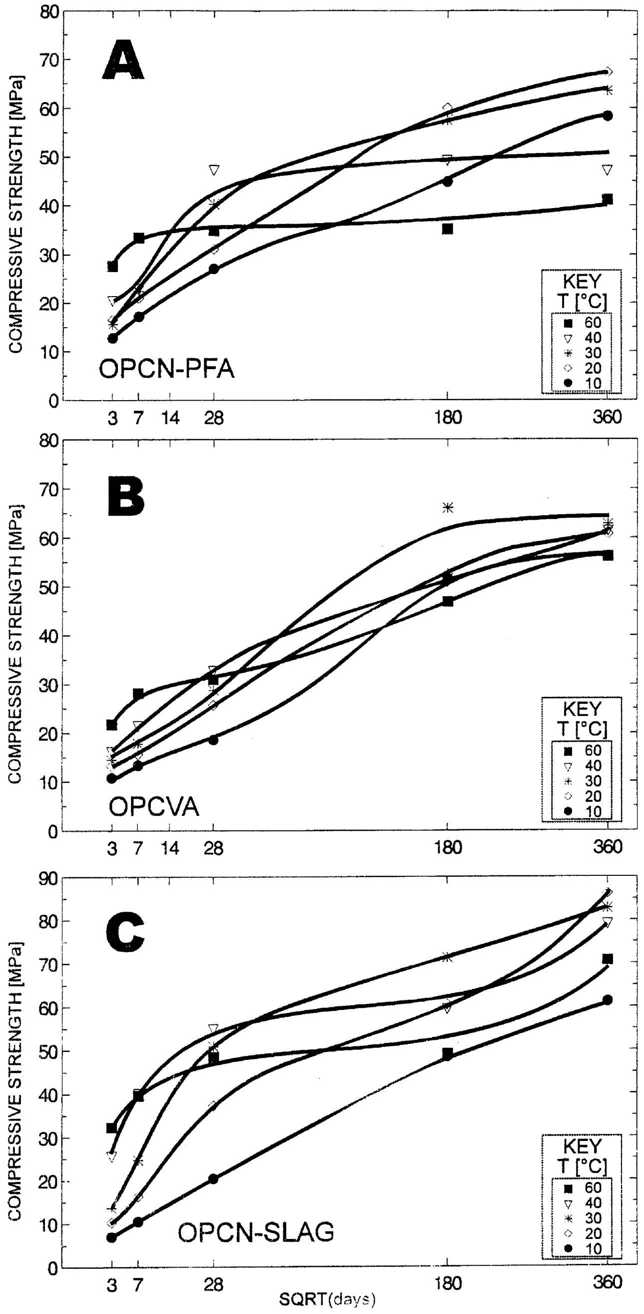

Fig. 1. Compressive strength vs. time for the three blended cement pastes hydrated at 10°C, 20°C, 30°C, 40°C, and 60°C.

indicates that there was no overall improvement in the mechanical properties of the paste by incorporating 30% PFA during the first year of hydration.

The blended cement paste incorporating VA (OPCVA in Fig. 1B) showed different characteristics. The high temperature inversion of strength observed in the corresponding neat cement paste [2] was not as evident in this blend. After hydration for 3 days, the compressive strength increased with increasing temperature, whereas after 360 days, the strength values at each temperature were similar to one another. The compressive strength of the blended cement paste OPCVA was less than that of the neat cement OPCS when cured at 10°C and 30°C (Fig. 2B), but at 40°C and 60°C, the compressive strength of the blended cement exceeded that of the neat cement after about 180 days.

For the cement paste incorporating GGBFS, the development of mechanical properties showed a different pattern from those of the pozzolanic blends (Fig. 1C). It is evident that the OPCN–GGBFS blended cement paste benefited from higher curing temperatures. After 3 days of curing, the difference in strength between the pastes cured at 10°C and 60°C was about 25 MPa. After 28 days, the gap was about

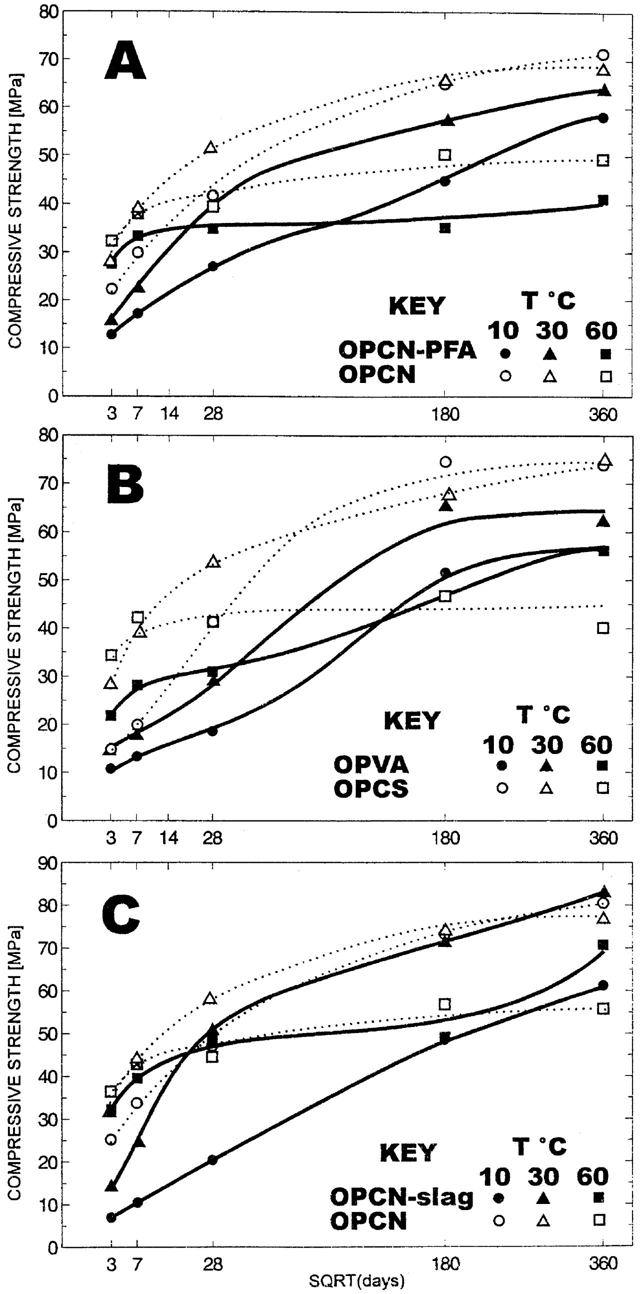

Fig. 2. Comparison of compressive strength vs. time for the blended and neat cement pastes hydrated at 10°C, 30°C, and 60°C.

35 MPa when comparing pastes cured at 10°C and 40°C. The 40°C and 30°C curves crossed that of 60°C after about 7 and 14 days, respectively (Fig. 1C). From these results, it seems that around 30°C was the optimum temperature for strength development in the slag–cement paste blend. This can be clearly observed in Fig. 2C, in which the plot for the blended cement paste hydrated at 30°C followed closely that of the neat cement paste from 28 days of curing. The plot for the blended cement paste cured at 60°C showed similar initial values to that of the neat cement paste, and after a year, it had reached higher values. Although there was some evidence for high temperature inversion in the blended cement pastes, it was not as marked as in the parent cement pastes (Part I). The lowest strength values were given by the paste cured at 10°C, indicating that this temperature was too low to favour hydration of the slag, which made only a minor contribution to the overall compressive strength in the first 28 days, although it subsequently contributed to the overall strength.

3.2. Scanning electron microscopy

Backscattered electron imaging enhances observation of the microstructure in terms of compositional contrast, since the brightness of the phases increases proportionally to their average atomic number. For pure Portland cement pastes, it is possible to distinguish in ascending order of whiteness: pores, C–S–H gel, CH, anhydrous phases [15,16]. The replacement materials fit in different places in the grey scale of the cement phases depending on their average atomic number. Their physical shape also helps to identify them in the microstructure. Anhydrous GGBFS grains are nearly as bright as anhydrous cement particles, while hydrated grains of GGBFS are slightly darker than C–S–H gel. Slag grains have angular morphology with sharp edges. Anhydrous VA is normally brighter than C–S–H but darker than CH; grains appear with angular shapes but not with such sharp edges as observed in GGBFS grains. PFA often has a grey shade similar to that of VA and is generally spherical with other irregular shapes as described by Diamond [11].

3.2.1. PFA blended cement

BEI of the PFA blended cement paste, hydrated for 1 year at 10°C and 60°C, are shown in Fig. 3A and B. It was clear to the naked eye that the apparent porosity (pores look black in BEI) was higher in the paste cured at 60°C. This explains the reduced strength observed at 60°C in the PFA blended cement paste (Fig. 2A). A similar observation was reported for the neat Portland cement paste [2] in which the microstructure that developed at 10°C appeared to be more compact and continuous. The microstructure of the PFA blend paste showed greater porosity than that of the neat cement paste, and exhibited many regions with local concentration of pores, which resulted perhaps from the intrinsic physical characteristics of the ash. This porosity probably impeded strength development in the blended cement. The amount of CH deposit observed in the microstructure at 60°C was low, indicating that CH had been consumed in the pozzolanic reaction. On the other hand, the reduced reactivity of the ash at 10°C was indicated by the presence of some CH crystals dispersed in small clumps in the microstructure. These observations confirmed those based on the CH content determined from TGA data that was presented in Part II [5]. The presence of partially hydrated cement grains in the microstructure (labelled P in Fig. 3B) of the paste cured at 60°C for 1 year is consistent with the reduced degree of hydration of the silicate and ferrite phases observed from the QXDA data reported in Part II. Only very few and small grains of anhydrous or partially hydrated cement grains were, however, observed at 10°C, consistent with the QXDA data. So increasing the curing temperature reduced the degree of hydration at later ages, which is consistent with the literature.

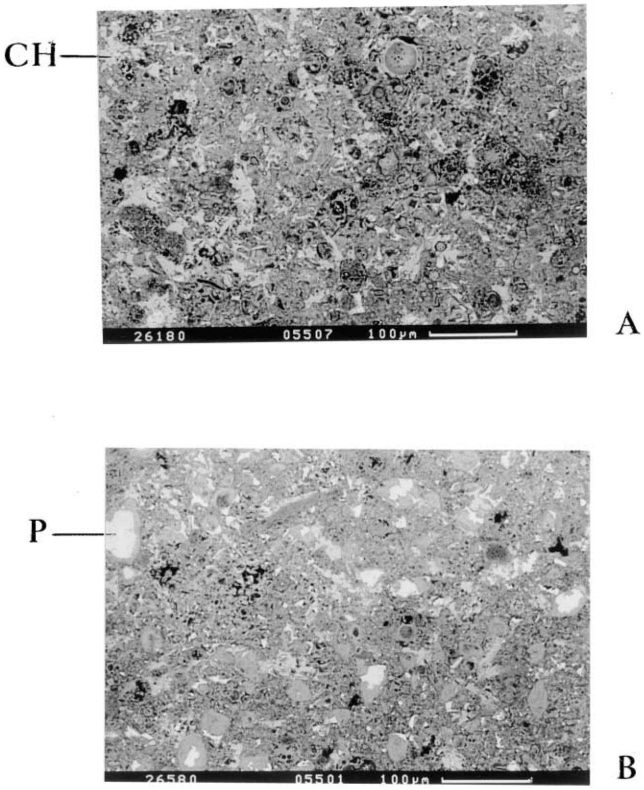

Fig. 3. BEI micrographs for the PFA cement paste hydrated at (A) 10°C and (B) 60°C for 1 year [CH = $Ca(OH)_2$; P = partially hydrated cement grain].

3.2.2. VA blended cement

Micrographs of the OPCVA blended cement paste cured at different temperatures are shown at various magnifications in Figs. 4–6. After curing at 10°C and 30°C for 1 year, the microstructure appeared to be more continuous and considerably less porous than that at 60°C. Most of the CH had been consumed at 60°C (Fig. 6A and B), whereas at 10°C (Fig. 4A and B), some CH clusters remained, sometimes surrounding VA particles (e.g. Fig. 4B). In Part II [5],

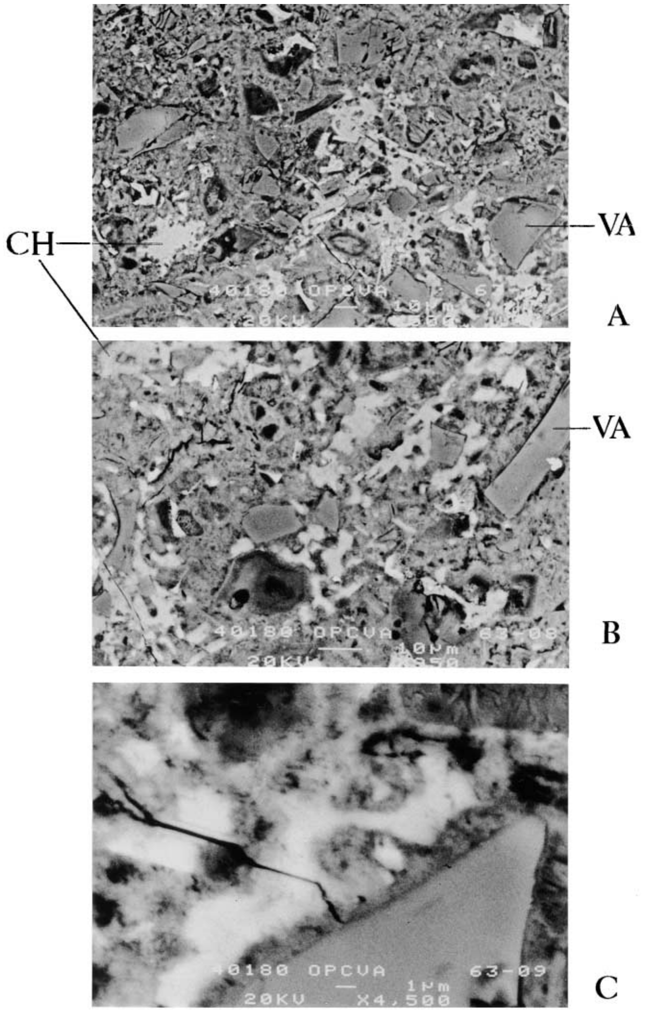

Fig. 4. BEI micrographs for the VA cement paste hydrated for 1 year at 10°C. (CH = $Ca(OH)_2$; VA = volcanic ash grain).

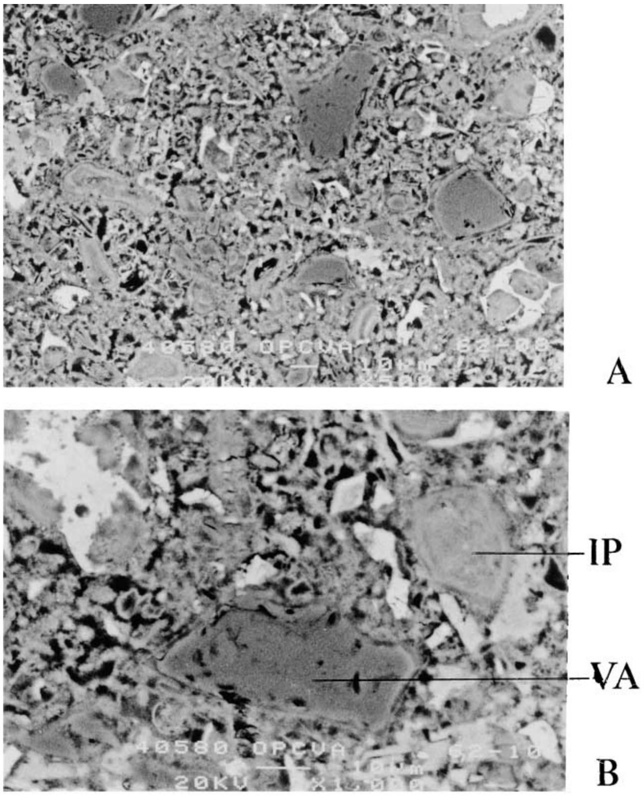

Fig. 6. BEI micrographs for the VA cement paste hydrated for 1 year at 60°C. (IP = inner product; VA = volcanic ash grain).

it was reported that the degree of hydration of the cement grains in the OPCVA blended cement paste was higher after curing at 10°C for 1 year than at 60°C. Accordingly, BE images show that nearly all of the cement grains were hydrated at 10°C, whereas some partially unhydrated cement grains remained at 60°C, e.g. in the upper parts of Fig. 6A and B. In Fig. 5, at 30°C, a VA grain is shown next to a fully hydrated cement grain.

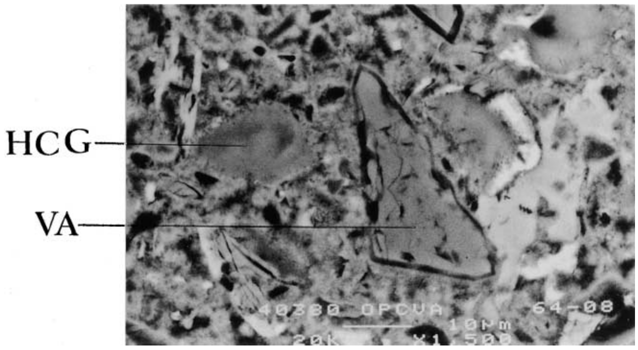

Fig. 5. BEI micrograph for the VA cement paste hydrated for 1 year at 30°C. (HCG = hydrated cement grain; VA = volcanic ash grain).

Another interesting feature is the presence of AFm clusters, which were not observed at 60°C, but were noticeable at 10°C and 30°C. This observation was also made and discussed in Part I, in the case of the neat cement paste. Inner products (IP) of hydrated cement pastes have been found to absorb more sulfur when hydrated at 60°C than at lower temperatures [17]. On curing at 10°C, the AFm crystals are large enough to be detected by SEM, but at 60°C, the sulfur is more dispersed, and any AFm is intimately mixed with the C–S–H gel.

The morphology of the VA grains varied. Some grains had angular corners but rounded edges, while others were elongated with prism-like shapes. It must be remembered that our images provide only a two-dimensional view of a three-dimensional matrix. In BE images of pastes hydrated at 10°C, the unreacted VA particles looked very similar in grey level to the hydrated cement grains. On the other hand, they appeared distinctly darker than the hydrated cement grains when curing occurred at 60°C. The unreacted VA particles must have the same grey level irrespective of the curing temperature, therefore the cement grains hydrated at the two extreme temperatures must exhibit different grey levels, i.e. they are lighter at the higher hydration temperature. This latter feature was also noted when comparing the microstructures of the neat cement pastes [2]. This suggests that the CSH that developed at 60°C was denser than that formed at 10°C. On comparing the OPCVA microstructures with those of the neat OPCS cement paste (Part I), the enhanced hydration of the cement after 1 year is also evident in the former.

3.2.3. GGBFS blended cement

The BE images for the slag cement pastes are shown in Fig. 7 (at 10°C), Fig. 8 (at 30°C), and Fig. 9 (60°C) at various magnifications. The blast furnace slag particles were sharply angular. When they hydrated, they exhibited rims of hydration products that formed within the boundaries of the original slag grains. The micrographs of the pastes cured at 10°C (Fig. 7) show some very dark slag-like shaped voids that are probably from slag grains pulled out during the polishing or grinding stages. The temperature dependence of the hydration of the slag can be noticed by comparing the micrographs of the pastes hydrated at different temperatures. At 10°C, many anhydrous or only slightly hydrated slag grains remained, whereas at 30°C (Fig. 8), some partially and fully hydrated slag grains were present in the microstructure. At 60°C (Fig. 9), the advancement of the slag hydration had proceeded even further, as indicated by the thicker rims that had developed around the slag grains. A partially reacted GGBF slag grain is marked S in Fig. 9B, while C indicates an unreacted clinker grain, and IP indicates inner product from the hydration of alite. The smaller slag grains had clearly reacted to a greater extent than those of larger size at 60°C.

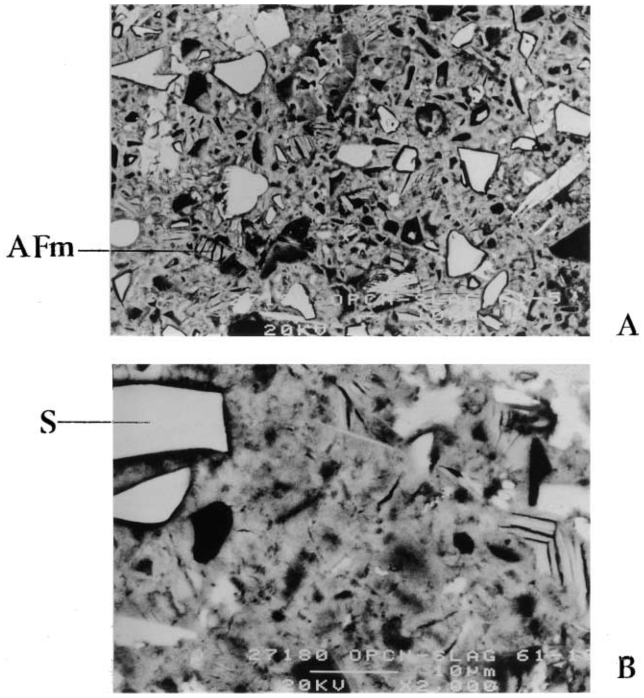

Fig. 7. BEI micrographs for the GGBFS cement paste hydrated 1 year at 10°C. (AFm = monosulfate; S = GGBFS grain).

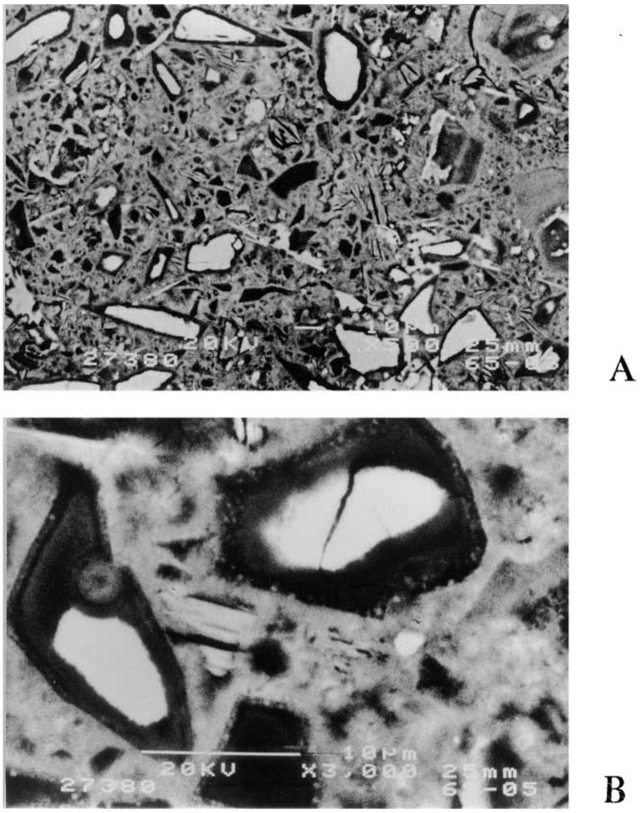

Fig. 8. BEI micrographs for the GGBFS cement paste hydrated 1 year at 30°C.

At 30°C and 60°C, the hydration products resulting from the slag formed a rim around the cores of the unreacted slag grains, which is clearly visible in Fig. 8B and Fig. 9B. At 10°C, these rims were less apparent, although there was a very thin dark area surrounding the slag grains (Fig. 7). While this might indicate a change in the mechanism of hydration of the slag grains at different temperatures, it is more likely to be due to different reactivities associated with the slower rate of hydration of the slag grains at 10°C.

X-ray diffraction of the slag cement pastes hydrated at 10°C and 60°C showed the presence of monosulfate, a hydrotalcite-like phase, and portlandite in both samples, but with more monosulfate present at 60°C. Ettringite was observed after curing at 10°C, but its peaks after curing at 60° were very weak indeed. TGA data indicated that the amount of CH present after 1 year was only about 6% at 10°C, 5% at 30°C, and 3.6% at 60°C [5]. In accordance with these observations, relatively little evidence for the presence of calcium hydroxide was obtained by SEM in any of the slag blend pastes. The few CH crystals remaining were probably finely distributed throughout the matrix. AFm crystals were identified by EDS and were abundant at all curing temperatures. The inner hydration products of the slag that formed at 60°C had relatively high Mg contents tentatively identified as a hydrotalcite-like phase. There were no such IP at 10°C, and smaller amounts of magnesium were widely dispersed in the outer products.

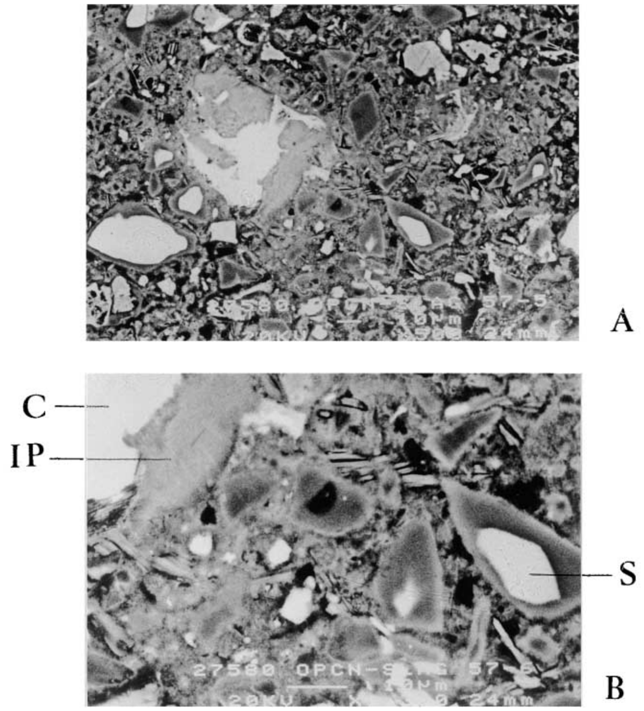

Fig. 9. BEI micrographs for the GGBFS cement paste hydrated 1 year at 60°C. (C = anhydrous cement grain; IP = inner product; S = GGBFS grain).

3.3. Further discussion

The blast furnace slag grains became surrounded by rims of hydration products, as clearly observed at 30°C and 60°C (Figs. 8 and 9), and as reported by Barker [18] in similar blended cements cured at 20°C. In the case of the blended cements based on PFA and VA, no such clear rims were formed (Figs. 3 and 4). Outer products resulting from the hydration of the anhydrous silicate grains can be observed near to the VA grains (Fig. 4C), and products of the pozzolanic reaction have also been deposited, as indicated by the grain of VA surrounded by CH with an intermediate layer of C–S–H. These differences indicate a different mechanism of hydration, related to the variation in chemical composition of the replacement materials. Whereas the BFS has a composition with nearly enough Ca and Si to sustain formation of C–S–H gel, the VA and PFA have insufficient Ca to form C–S–H gel, except by reaction with the matrix formed by the products of hydration of the cement phases.

Most of the cement grains have been totally hydrated in the blended cement pastes incorporating GGBFS (Fig. 9) and VA (Fig. 6) that were cured at all the temperatures studied. This can probably be attributed to enhanced cement hydration in the presence of the replacement materials [5]. It was also in part an effect of additional water; the water:solid ratio was maintained at 0.5, so that the effective water:cement ratio was much higher. Since the replacement materials reacted more slowly than the cement, more water was available for reaction with the cement in the early stages of hydration. The hydration of the cement fraction might also be favoured as the CH is consumed (since a hydration product is being removed from the system), shifting the equilibrium towards a greater degree of hydration of the silicate phases, leaving lower levels unreacted.

Although the microstructure of the slag–cement paste at 60°C was more porous than that at 10°C, the compressive strength was higher at 60°C for the times studied here. The pastes cured at 20°C and 30°C attained the highest compressive strengths, which may be due not only to a more advanced degree of hydration of the slag, but also to a denser microstructure with a finer distribution of pores (Fig. 8A and B). At 10°C, the lower strength observed can be attributed to a much reduced reactivity of the slag. In order to improve the mechanical properties, the slag cured at 10°C needs to reach higher levels of hydration, to consume more water, and to form additional C–S–H gel. Intermediate curing temperatures gave better mechanical properties than those at 60°C and 10°C, although the slope of the compressive strength curve at 10°C is highly positive (Fig. 1C). The high temperature inversion in the compressive strengths of the slag blend pastes was similar, except for the 10°C curve, to that observed in the neat cement pastes. This suggests that a mechanism, similar to that proposed by Verbeck and Helmuth [4] for neat cement pastes, is operating, leading to increased porosity of the hydrated paste by rapid formation of dense hydration products around the slag grains at 60°C, which retards the rate of slag hydration and strength development.

There was a difference in the grey levels shown by the hydration products of the slag at 30°C and 60°C. The latter have a lighter grey level than the former. This observation suggests that the hydration products of the slag formed at 30°C were less dense than those developed at 60°C. A similar observation was noted for neat cement pastes based on these cements [2,17] and has been reported previously [19,20].

Particle size is another important factor that affects the reactivity of the replacement materials. Finer grains of slag [21] or VA enhance their reactivity and reduce the effect of the increased water:cement ratio, especially at early ages, in these blended cements. The disappointing compressive strength development of the PFA blended cement paste at 40°C and 60°C may be explained by the physical properties of the ash, which had about 30% of particles with a size greater than 70 μm.

4. Conclusions

It was generally observed for all the blended cement pastes investigated that increased hydration temperature brought about increased porosity; the differences were most marked between 10°C and 60°C. Microstructural evidence was obtained for the increased degree of hydration of the cement fraction in blended pastes with respect to the neat cement pastes, which is consistent with the data obtained by quantitative X-ray diffraction analysis reported in Part II [5]. The additional water available for the hydration of the Portland cement has to be considered together with the presence of the replacement material. In the pozzolanic cement pastes based on PFA and VA, CH was almost absent in the cements cured at 60°C, whereas at 10°C, some CH clusters were visible in the microstructure. This confirms the effect of temperature on the pozzolanic reaction as reported in Part II. In the cement incorporating GGBFS, the remaining CH, after partial consumption by the slag, was finely distributed throughout the microstructure.

The microstructure displayed by the cement incorporating VA after hydration at 10°C, 30°C, and 60°C suggested that the hydration mechanism was via dissolution of the ash grains, since no hydration product rims were observed around the VA particles. The high temperature inversion in the compressive strength was not as marked in OPCVA paste as in the neat cement paste. The strength curve at 60°C attained similar values to those obtained at 30°C and 40°C after 1 year.

The slag grains in the blended slag cement paste cured at 10°C had only a very dark, thin rim of hydration products. On the other hand, at 30°C and 60°C, rims of hydration products were clearly observed. The C–S–H resulting from the slag hydration at 30°C exhibited a darker grey level than that formed at 60°C. From the temperatures selected, 30°C appeared to be the optimum for strength development of the slag blend, whereas 10°C was evidently less favourable to the reactivity of the slag.

While the cement incorporating PFA displayed poor strength development compared with that of the neat cement paste, a similar high temperature inversion was observed as for the neat cement paste, leading to greater strengths after 1 year, when the blended cement was hydrated at 10–30°C than at 40–60°C.

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