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# FACTORS INFLUENCING STRENGTH DEVELOPMENT OF CONCRETE CONTAINING SILICA FUME

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## ABSTRACT

Existing data on the relationships between temperature, pozzolanic activity and cement hydration are reviewed with particular emphasis on condensed silica fume (CSF)-ordinary Portland cement blends. CSF concrete with a range of fume contents has been cured at two temperatures (20°C and 50°C) for periods up to 91 days. Strength development and relative strength are considered in relation to temperature, cement hydration and pozzolanic action. The observed results establish that relative strength varies directly with CSF content and that the strength enhancement at early curing periods, which is achieved by increase in curing temperature, is a result of increased reaction rate between $Ca(OH)_2$ and CSF.

## Introduction

### Alite and Belite Hydration

The rate of development of compressive strength of concrete made from ordinary Portland cement is principally determined by the rate of hydration of its four major constituents, alite, belite, aluminate and ferrite. Typical proportions for these major constituents are in the range alite (45-60%), belite (12.5-25.0%), aluminate (7.0-12.0%) and ferrite (6.0-10.0%). The aluminate and ferrite which constitute the minor phases, consequently make only a minor contribution to strength development relative to the two silicate phases, especially alite.

A number of workers have investigated hydration of the individual cement compounds in Portland cement paste. Work by Copeland et al [1] suggests that hydration of the pure cement compounds is slower than that of the same compounds in cement paste. Also, water/cement ratio (w/c) influences the rate of hydration, the tendency being that the greater the w/c ratio, the greater the hydration rate. Alite and belite hydrate at very different rates, belite being much slower to hydrate than alite [1,2]. Although differences in the cements in relation to factors such as specific surface, impurities and differences in curing conditions produce a wide variation in hydration rates, it is undoubtedly the case that in OPC a very substantial proportion of alite has hydrated within the first few days, and by 28 days, hydration is near to completion. For belite, hydration is much slower and even at 28 days, there is still a significant proportion of unhydrated material remaining. Therefore, the major contribution to strength development of concrete within the first few days of curing is derived from alite hydration, with small contributions from belite hydration and hydration of the aluminate and ferrite phases. As curing progresses, belite hydration makes an increasing contribution to the strength development, and beyond 28 days, it is the principal contributor to strength gain, that from alite hydration becoming negligible.

## Factors Influencing Hydration Rate

Hydration reactions of cements can be accelerated by a number of different factors of which temperature and pozzolana additions are two practical examples. Copeland et al [1] demonstrated that temperature has a very significant accelerating effect on alite hydration. For an OPC paste of w/c ratio 0.57 cured for 0.5 days, there is negligible hydration of alite at 5°C, about 30% hydration at 25°C and nearly 60% hydration at 50°C. After 2 days curing, hydration is near to completion at 50°C whereas at 25°C only 60% has hydrated and at 5°C only about 30%. Only after about 7 days curing does alite hydration at 25°C approach that at 50°C, and at 5°C it takes about 30 days. Belite hydration in cement paste is also accelerated by temperature, but to a lesser degree than alite. The effect of temperature on belite hydration is delayed relative to its effect on alite hydration and significant differences in the degree of hydration at different temperatures occur at much later ages.

Partial replacement of cement by pozzolanic materials, in particular condensed silica fume (CSF), is also claimed to affect hydration rate. The relative effect of CSF on the rates of alite and belite hydration has been investigated only for the individual cement compounds themselves rather than for cement paste, and this work indicates [3,4] that CSF has an accelerating effect on both alite and belite hydration. The accelerating effect of fume on the overall hydration of Portland cement has been studied by Huang and Feldman [5] for Portland cement - CSF blends containing 0 and 30% CSF or 30% finely ground silica sand at a water/solid [w/s] ratio of 0.6. For curing periods of up to 8 hours the amount of $Ca(OH)_2$ produced by all three mixes was the same, even though in two of the mixes, there was a 30% dilution factor. They explained this apparent anomaly in terms of accelerated hydration of OPC by the finely divided silica, which acted as a nucleating agent for $Ca(OH)_2$. They also observed a maximum in $Ca(OH)_2$ content which occurred at 7 days with 10% fume and within 1 day with 30% fume. Similar maxima have also been observed by Traetteburg [6] and Chatterji et al [7].

Traetteburg [6] determined the $Ca(OH)_2$ content and soluble silica content for a cement - 10% CSF paste of [w/s] ratio 0.5. Whereas for pure paste the $Ca(OH)_2$ content rose rapidly up to 7 days and then levelled off, for the cement blend the $Ca(OH)_2$ content reached a maximum at 6 days and then declined sharply from 7 to 15 days. This decline corresponded with a sudden increase in soluble silica content from 7 to 15 days and was interpreted as being due to rapid reaction of CSF with lime to give additional C-S-H gel. Similar results were obtained by Chatterji et al [7] on cured 80% OPC -20% CSF prisms with a w/s ratio of 0.5, although in this case the maximum in $Ca(OH)_2$ content occurred at 3 days, declined to a minimum between 7 and 14 days and then showed a further gradual increase. Detection of residual silica beyond 14 days suggested that the apparent termination of lime consumption beyond 14 days was due, not to the total consumption of the CSF, but to the formation of an inhibiting layer of reaction product around the CSF particles.

In a recent publication, Sun and Young [8] reported the results of an investigation of the reaction of CSF in hydrating cement paste by determining residual CSF using 29-Si NMR. Additions of CSF ranged from 18 - 48%. Their results are plotted in Figure 1, as percent CSF reacted against curing time. There is very rapid consumption of fume over the first

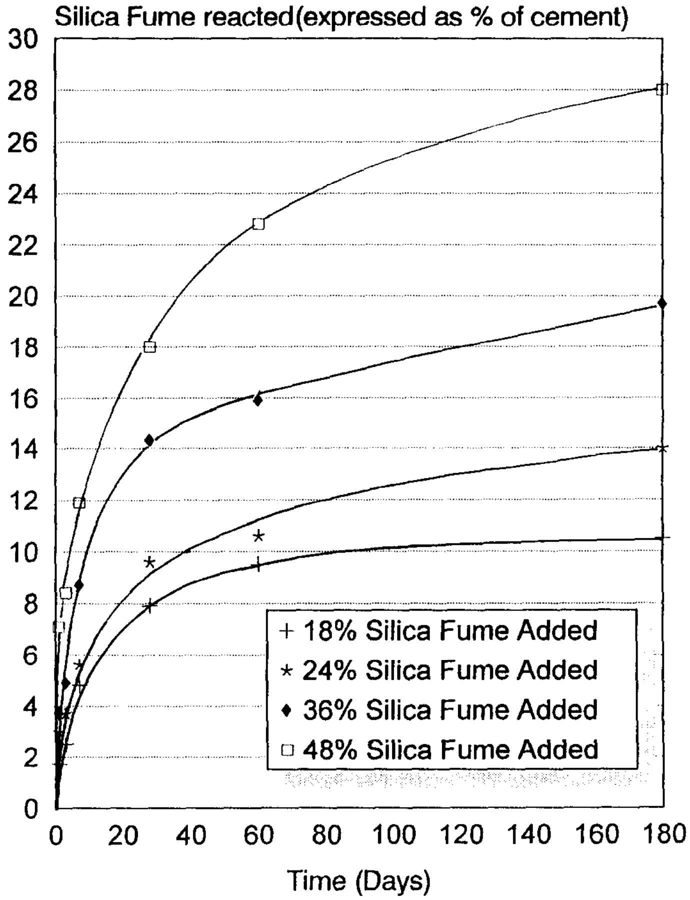

FIG. 1
CSF reacted (expressed as weight percent of total cementitious solids) versus curing time, for CSF-cement pastes with various CSF contents. Data from Sun and Young[8].

28 days and particularly within the first 7 days. However, regardless of the amount of fume added, reaction is always severely curtailed after an initial active period, and even after long curing times a large proportion of that added is still unreacted. This suggests, in agreement with Chatterji et al [7], that the principal rate controlling factor is the build up of an inhibiting layer of reaction product around each CSF particle. Also, the fact that in the very early stages of hydration, the rate of CSF consumption increases directly with CSF content, irrespective of the level of fume addition, lends support to Huang and Feldman's claim [5] that CSF additions accelerate cement hydration.

Although, in the very early stages of curing, there is clear evidence of accelerated hydration of cement by CSF, Bentz and Stutzman [9] observed in cement - CSF concrete, that cement hydration ceased between 7 and 28 days and at 28 days cement hydration was below that in the control concrete. However pozzolanic reaction, evidenced by increasing lime consumption, did continue well beyond 7 days. They also reported that CSF has little effect on the ultimate strength of cement paste but produces a significant increase in concrete strength. This increase is attributed to a much denser and more homogeneous product at the interfacial zone between the cement paste and the aggregate. This zone although denser than that formed in concrete without CSF, does contain a network of coarse pores which are formed due to dissolution of the coarse $Ca(OH)_2$ by the pozzolanic reaction.

Therefore from the literature the following conclusions can be drawn with regard to temperature change, pozzolanic activity and cement hydration in Portland cement systems.

i) In OPC paste at normal curing temperatures, a very substantial proportion of alite hydrates within the first few days and by 28 days hydration is near to completion. For belite, hydration is much slower and even at 28 days, there is still a significant proportion remaining.

ii) Temperature has a marked accelerating effect on alite hydration in cured cement paste. Within 2 days, hydration is virtually complete at 50°C whereas at 5°C only about one third has hydrated. Belite hydration in cement paste is also accelerated by temperature, but to a lesser degree and only becomes pronounced after several days of curing.

iii) CSF additions increase $Ca(OH)_2$ formation in the very early stages (8 hours) of curing and hence accelerate the hydration of the Portland cement. After this initial period, the rate of $Ca(OH)_2$ formation cannot easily be assessed because the $Ca(OH)_2$ formed is continuously removed by reaction with the CSF. However, work on CSF concrete suggests that there is little further cement hydration beyond 7 days and at 28 days, hydration is below that in normal concrete.

iv) Although $Ca(OH)_2$ consumption only becomes apparent when the $Ca(OH)_2$ content within the hydrating paste begins to decline (typically after a few days), the reaction of $Ca(OH)_2$ with CSF begins at a much earlier stage. This is evidenced by the more or less immediate decline in CSF content on commencement of curing.

v) A principal rate controlling factor in the reaction of $Ca(OH)_2$ with CSF in hydrating cement paste appears to be determined by the concentration of CSF. At extended curing times the reaction seems to be controlled by the build up of an inhibiting layer of reaction product around the CSF particles.

vi) The significant increase in concrete strength achieved by CSF additions is attributed to formation of a much denser and more homogeneous product at the interfacial zone between the cement paste and the aggregate.

The factors discussed above clearly have a profound effect on the strength development of concrete, and the complex and interdependent nature of the different variables make it difficult to predict the way in which relative strength gain will be modified when concrete containing CSF is cured at different temperatures. The aim of the current work is, therefore, to determine the manner in which these factors influence the strength development of high strength CSF concrete.

## Experimental Work

### Workability and Compaction

The partial replacement of OPC with CSF in a concrete mix can result in a number of undesirable effects which are brought about by the high water demand of CSF. These include a rapid loss of workability, incomplete compaction, a reduction in long-term strength, and in some cases, crack formation. Hjorth [10] however, has reported that the increased water demand resulting from increasing levels of CSF can be compensated for by use of superplasticisers. It has, therefore, become common practice (see Huang and Feldman [5]) to use increasing amounts of superplasticiser with increasing CSF contents to compensate for the effect of increased water demand. This is the approach adopted in the current work. Also, to minimise the effect of rapid loss of slump with time brought about by the accelerated reaction, a careful and thorough mixing procedure has been adopted.

### Constituent materials, Mix Design and Strength Testing

The constituent materials used in the work were as follows; Ordinary Portland cement (OPC) type 1, complying with BS 12: 1989 (see Table 1), CSF in the form of a stabilised slurry in which the mass ratio of CSF solids to water is 1:1. (see Table 1), fine natural sea dredged sand which complied with grade F of BS 882:1983, and 10mm single size crushed limestone aggregate.

Tests were carried out in accordance with BS812:part 2:1975 to determine the moisture content and water absorption of both fine and coarse aggregate. The results are presented in Table 2. Prior to use, the aggregates were saturated with the required amount of water in air-tight containers under constant temperature conditions for 24 hours. This ensures that the effect of any variation in moisture content is minimised.

### TABLE 1

### Physical and Chemical Properties of OPC and CSF

| | OPC | CSF |
|---|---|---|
| Particle Size (μm) | 15 | 0.15 |
| Surface Area ($m^2/kg$) | 350-500 | 15000-20000 |
| Bulk Density ($kg/m^3$ ) | 1300-1400 | 200-300 |
| Specific Gravity | 3.14 | 2.2 |
| Composition (%) | | |
| $SiO_2$ | 20.9 | 92.0 |
| $Fe_2O_3$ | 2.2 | 1.2 |
| $Al_2O_3$ | 4.5 | 0.7 |
| CaO | 64.0 | 0.2 |
| MgO | 2.3 | 0.2 |
| $Na_2O+K_2O$ | 0.9 | 2.0 |
| Impurities + LOI | 4.7 | 2.7 |

The reference mix was designed in accordance with DoE specifications [11] with the aim of producing concrete of high strength (70 $N/mm^2$). The proportions of cement : fine aggregate : coarse aggregate for the mix were 1:1.4:2.2. Several trial mixes were first prepared using different water/cement (w/c) ratios and incorporating varying amounts of CSF and super- plasticizer. The superplasticizer used was a polymeric sulphonate based liquid superplasticizer. It has a specific gravity of 1.14 at 20°C, contains no chloride and does not induce air-entrainment. With the experience gained from the trial mixes it was found feasible to produce workable concretes giving high degrees of compaction under vibration with a w/c = 0.35 and a maximum CSF content of 30%. However, to achieve this as much as 3-4% superplasticizer was required at the high fume contents. The mix proportions for the mixes investigated are given in Table 3.

### TABLE 2

### Physical Properties of Aggregates

| | Fine Aggregate | Coarse Aggregate |
|---|---|---|
| Relative density (Saturated Surface Dry) | 2.70 | 2.65 |
| Water Absorption (% of dry mass) | 2.96 | 0.34 |
| Moisture Content | 0.70 | 0 |

TABLE 3

Mix Proportions (kg per m$^3$), w/s=0.35.

| Mix No | CSF | | WATER (kg/m$^3$) | CEMENT (kg/m$^3$) | SUPER-PLASTICISER | | SLUMP (mm) |
|---|---|---|---|---|---|---|---|
| | (%) | Slurry (kg/m$^3$) | | | (%) | L/m$^3$ | |
| 1 | 0 | 0 | 165.0 | 470.0 | 0 | 0 | 15 |
| 2 | 12 | 112.8 | 112.8 | 413.6 | 2.0 | 8.2 | 74 |
| 3 | 16 | 150.4 | 89.8 | 394.8 | 2.5 | 10.3 | 110 |
| 4 | 20 | 188.0 | 71.0 | 376.0 | 3.0 | 12.4 | 141 |
| 5 | 24 | 225.6 | 52.2 | 357.2 | 3.5 | 14.4 | 156 |
| 6 | 28 | 263.2 | 33.4 | 338.4 | 4.0 | 16.5 | 170 |

The CSF content is given as the mass of CSF solids expressed as a percentage of the mass of OPC in the reference mix. Thus the cementitious content (OPC & CSF) of the different mixes was maintained at a constant value of 470 kg/m$^3$. In all the concrete mixes incorporating CSF, the water/cementitious solids ratio (w/s) is based on the total cementitious materials, i.e. OPC & CSF solids. In calculating the w/s ratio allowance was made for the water included in the CSF slurry. Water, CSF slurry and superplasticizer were first thoroughly mixed together and then poured onto the coarse aggregate in the pan mixer. After mixing for 20 seconds the fine aggregate was introduced and mixed in for a further period of 20 seconds. This was followed by the cement which was mixed in until a total mixing time of 2 minutes was achieved. The mixes were immediately cast into 100mm cube moulds and compacted on a vibrating table. After 24 hours the demoulded cubes were cured in water at temperatures of 20°C and 50°C for periods from 4 days to 91 days and were then tested in compression to BS1881:1983.

## Results and Discussion

The cube compressive strengths are given in Table 4. Each strength value is the average of three tests (individual values varied to within ±5% of the mean).

### Concrete cured at 20°C

Figure 2 shows the relative strength of the CSF concrete, as a function of curing time, at 20°C curing and at different CSF contents. The relative strength is the ratio of the

### TABLE 4

**Cube Compressive Strengths for CSF Concrete Cured at 20°C and 50°C.**

| Mix No | CSF (%) | 28-Days Density | Temp (°C) | Compressive Strength, (N/mm$^2$) | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | 4-Days | 7-Days | 14-Days | 28-Days | 91-Days |
| 1 | 0 | 2440 | 20 | 60.2 | 66.3 | 72.9 | 76.7 | 87.7 |
| 2 | 12 | 2430 | | 58.4 | 66.3 | 80.6 | 86.7 | 88.2 |
| 3 | 16 | 2405 | | 57.5 | 69.1 | 80.1 | 91.5 | 94.1 |
| 4 | 20 | 2425 | | 49.6 | 62.1 | 78.1 | 84.4 | 95.5 |
| 5 | 24 | 2410 | | 54.5 | 65.5 | 79.2 | 84.2 | 102.7 |
| 6 | 28 | 2405 | | 65.0 | 76.8 | 92.1 | 99.7 | 119.8 |
| 1 | 0 | 2445 | 50 | 61.7 | 68.8 | 70.0 | 74.0 | 82.4 |
| 2 | 12 | 2420 | | 72.6 | 77.0 | 77.9 | 80.4 | 79.2 |
| 3 | 16 | 2390 | | 83.9 | 83.8 | 83.7 | 82.4 | 89.0 |
| 4 | 20 | 2415 | | 87.2 | 88.8 | 93.8 | 90.7 | 95.5 |
| 5 | 24 | 2405 | | 94.8 | 95.8 | 100.4 | 98.6 | 101.7 |
| 6 | 28 | 2410 | | 99.6 | 103.1 | 108.7 | 110.5 | 110.3 |

compressive strength at a given curing period to the compressive strength of the reference mix at the same curing period and curing temperature. Clear trends are evident as the CSF content increases (Figures 2a to 2c). At all fume contents investigated, relative strength was found to increase rapidly within the first three weeks of curing. However, beyond three weeks at low fume contents (12% and 16%) relative strength subsequently declined, at intermediate fume contents (20%) it remained constant, and at high fume contents (24% and 28%) it continued to increase. In addition in the early stages of curing and particularly at the intermediate fume contents relative strength was below 100% ( i.e. the fume concrete was weaker than the reference mix).

Evidence from previous work has suggested that the build up of a layer of reaction product around the fume particles, is the principal rate controlling factor in the reaction of fume with lime. For this type of mechanism, the formation of an inhibiting layer of reaction product would be expected to be very rapid at low fume content whereas, for high fume contents (where surface area is much greater) the time taken for complete formation of this inhibiting layer would be much greater. In relation to the current observations (Figure 2), at low fume contents (12% and 16%), relative strength declines after 28 days. It is proposed that for these specimens reaction of lime with fume is seriously retarded beyond 28 days and hydration is near to completion whereas for the reference mix, hydration is at a less advanced stage and strength is still showing significant increases. For high fume contents (24% and 28%), at 28 days the inhibiting layer of reaction product around the fume particles will not be fully developed. Therefore continued reaction of fume with lime will result in further strength enhancement relative to that of the reference mix.

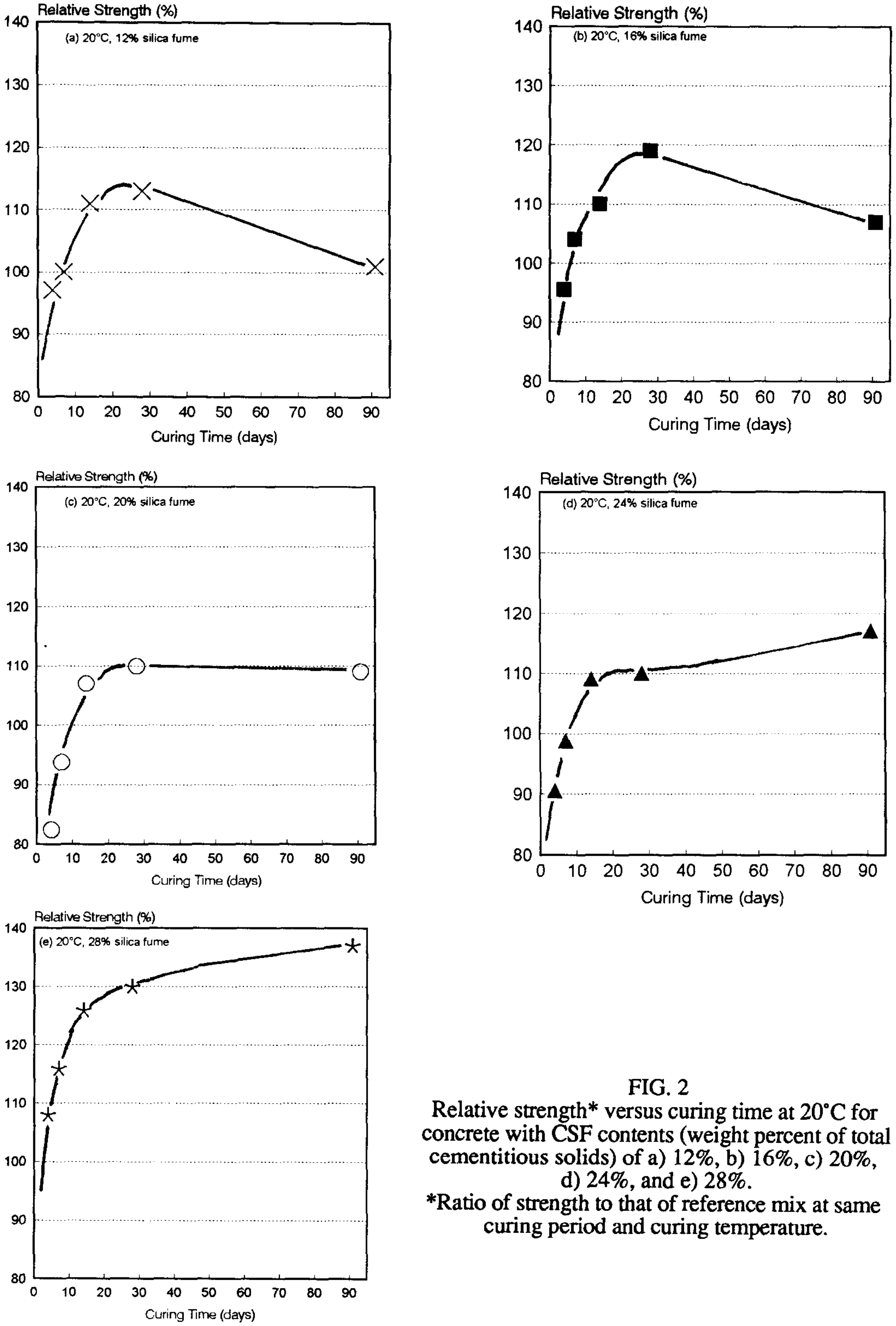

FIG. 2
Relative strength* versus curing time at 20°C for concrete with CSF contents (weight percent of total cementitious solids) of a) 12%, b) 16%, c) 20%, d) 24%, and e) 28%.
*Ratio of strength to that of reference mix at same curing period and curing temperature.

One unexpected feature of the current results (see Table 4), is that for 20°C curing at short curing periods (4 days and 7 days), there is an initial decline and then a subsequent increase in strength as the CSF content increases. The decline in strength can be explained as an initial dilution effect by the CSF at early ages. The addition of fume also has an accelerating effect on the hydration rate of the cement, and the fume itself reacts with lime to produce additional strength enhancing gel. Hence there are three interdependent variables influencing strength development and the overall result will therefore be governed by those that predominate in a specific mix at a particular time. In addition although great care was exercised to ensure full compaction for each mix, there will inevitably be a small variation between mixes which introduces a further complicating factor. However, it should be noted that specimens from the same mixes cured at 50°C did not show this effect which suggests that the effect is not due to variation in compaction.

## Concrete cured at 50°C

Figure 3 shows the relative strength of CSF concrete as a function of curing time for 50°C curing. At four days relative strength is well above 100% and systematically increases with increase in CSF content at each of the curing periods monitored. At very early curing periods (i.e. hours rather than days) where strength was not monitored relative strength would be expected to be lower due to the diluting effect of the CSF. This is indicated by the dashed part of the curves in Figure 3. The curves also show that beyond 4 days relative strength sharply declines with increase in curing time. This might be expected because the increase in temperature to 50°C will not only accelerate cement hydration for the CSF concrete but will also accelerate the lime-CSF reaction. This is supported by the data in Table 4 which show enhanced early strength at four days and minimal strength increase (10%) beyond 4 days. For the reference mix however without fume addition, the strength increased by 34% between 4 and 91 days. An interesting feature of the curves in Figure 3, is the development of a distinct minimum in the relative strength curing time plots at approximately seven days and a maximum between 14 and 21 days. This could be explained as a result of early termination of the cement hydration in the CSF concrete, a phenomenon observed by Bentz and Stutzmann[9]. However, substantial analytical work would be required to confirm this suggestion.

Surprisingly (see Table 4), increase in temperature has very little effect on the strength of the reference mix. Thus acceleration in hydration rate by increasing the curing temperature does not appear to enhance strength. In fact the long term strengths for 50°C cured concrete tend to be a little below those for 20°C cured concrete. Bland et al [12] have recently shown that although elevated curing temperatures give accelerated cement hydration, they can also lead to microcracking in which deposits of Portlandite and ettringite are concentrated in and around the cracks. Also, a number of authors have reported that Portlandite deposits in cured concrete are much coarser in concrete cured at 40°C [13,14] and 50°C [15] than that cured at 20°C. This modification in microstructure can result in loss of strength, a feature supported by the present work.

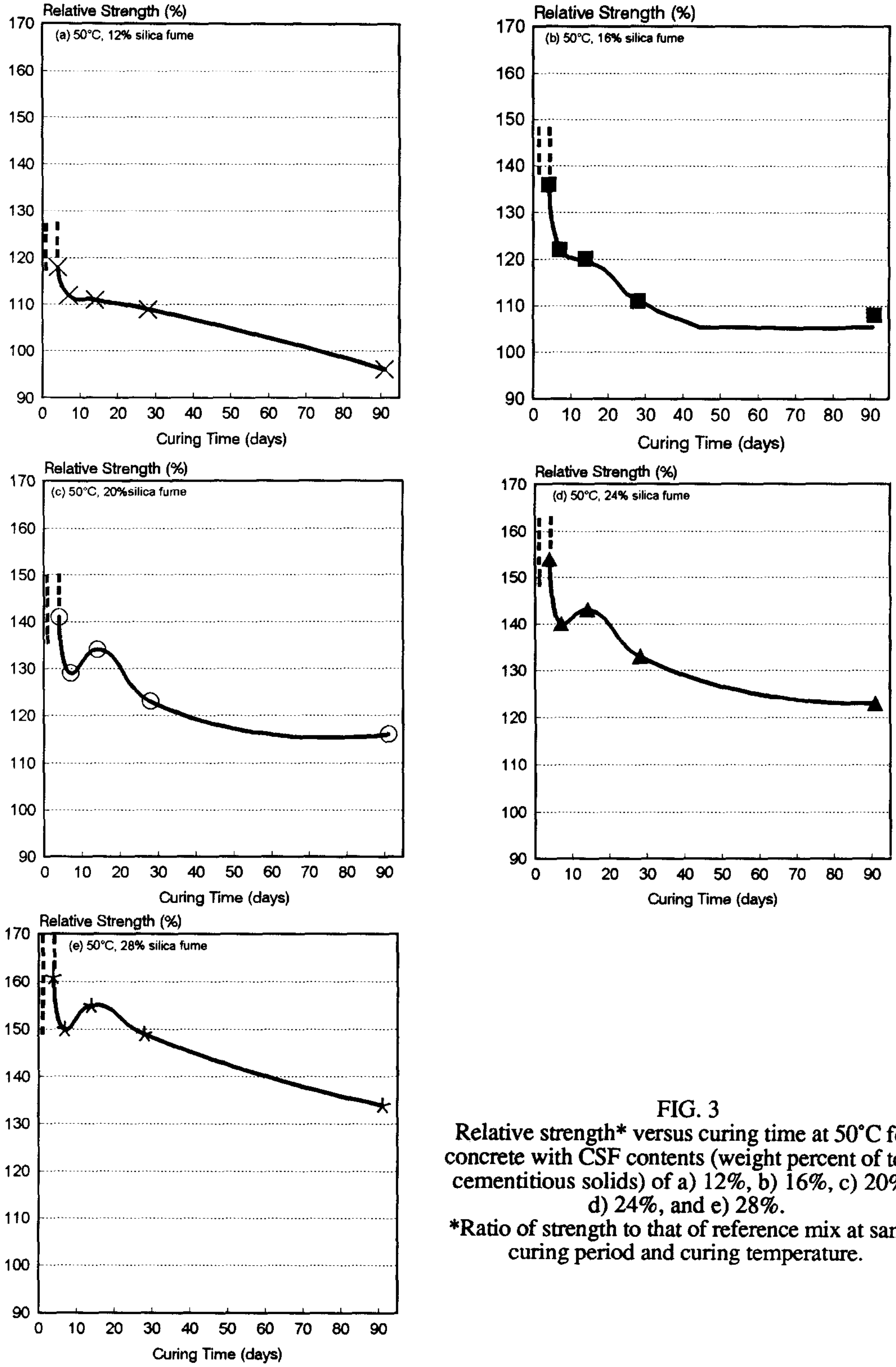

FIG. 3
Relative strength* versus curing time at 50°C for concrete with CSF contents (weight percent of total cementitious solids) of a) 12%, b) 16%, c) 20%, d) 24%, and e) 28%.
*Ratio of strength to that of reference mix at same curing period and curing temperature.

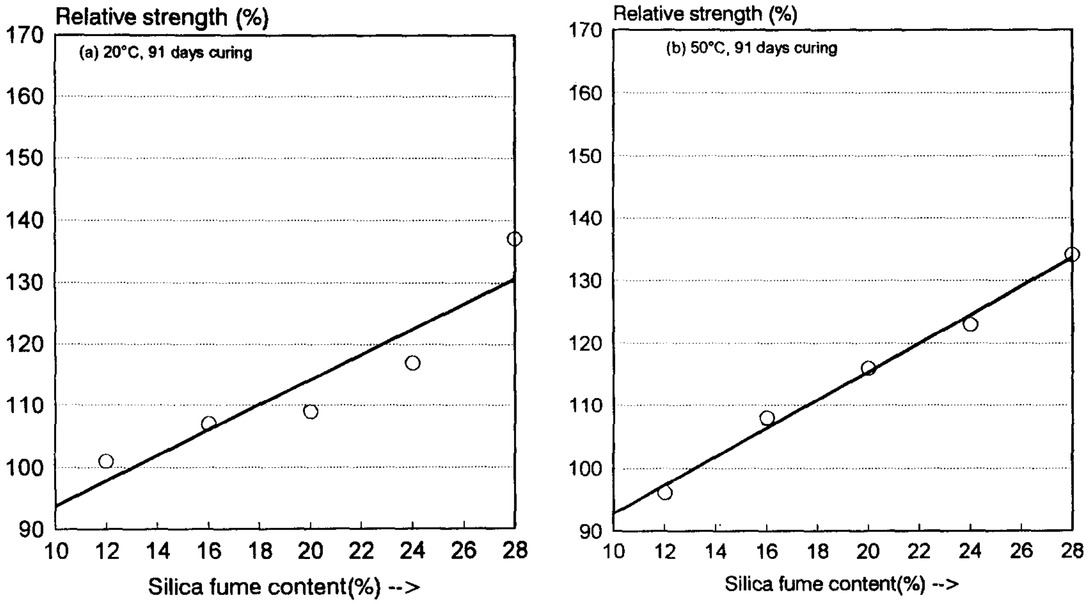

FIG. 4
Relative strength* at 91 days versus CSF content for concrete cured at a) 20°C b) 50°C
*ratio of strength to that of reference mix at same curing period and curing temperature

Current observations also show that the short term strengths of CSF concrete cured at 50°C are considerably in excess of those cured at 20°C. This substantial gain in strength (see Table 4) is undoubtedly a result of the increased reaction rate of fume with lime which will rapidly consume Portlandite crystals and accelerate deposition of C-S-H gel. After longer curing periods when reaction at the lower temperature is much more advanced there is, as might be expected, a much smaller difference in strength of concretes cured at the two different temperatures. In addition the ultimate strengths (91 days) of the CSF concretes are significantly greater than those for the control mix. Also the relative strengths (see Figure 4) show a continuous increase with increase in CSF content, and the rate of increase is similar at the two curing temperatures. These results add support to the work of Bentz and Stutzman [9] who attribute the increase in concrete strength to formation of a denser and more homogeneous interfacial zone as a result of consumption of coarse $Ca(OH)_2$ crystals by reaction with the CSF.

## Conclusions

The following conclusions can be drawn from the current work.

I) When curing at 20°C the relative strength (ratio of strength to that of reference mix at same curing period and curing temperature) of CSF concrete increases rapidly within the first 21 days. Beyond 21 days, relative strength declines at low fume contents but continues to increase at high fume contents. This behaviour can be explained in relation to the time taken for an inhibiting layer of reaction product to form around CSF particles thus terminating the lime-fume reaction. The time taken for this to occur, increases with increasing fume content.

ii) The increased hydration rate of the cement combined with the increased reaction rate of the CSF with lime when the curing temperature is raised to 50°C results in a very marked increase in relative strength at very early ages. The near completion of these reactions within a few days leads to an overall decline in relative strength with curing time. However, a minimum develops in the relative - strength curing time curves with increased CSF content and it is suggested that this minimum results from the termination of cement hydration in the CSF concrete.

iii) At early ages curing temperature has little effect on the strength of the control concrete and at extended curing periods strengths of 50°C cured concrete tend to be less than those of 20°C cured concrete. It is suggested that this loss in strength is due to coarsening of calcium hydroxide crystals particularly at interfacial zones, which is an effect reported by a number of workers. Curing temperature does however have a very substantial effect on the strength of CSF concrete at early ages principally as a result of the increased rate of reaction of CSF with calcium hydroxide, although as might be expected, the ultimate strengths are very similar. Also there is a continuous increase in relative strength with CSF content at both curing temperatures and the magnitude of these increases are similar at both temperatures. This suggests that the CSF is performing the same function at both temperatures. It is suggested that this function is the formation of increasingly dense interfacial zones resulting from consumption of $Ca(OH)_2$ crystals by reaction with CSF.

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