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Transient thermal strain of concrete: literature review, conditions within specimen and behaviour of individual constituents

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SYNOPSIS

This is the first of three papers presenting the results of an investigation into the effect of material and environmental factors upon the transient thermal strain behaviour of concrete during the first heat cycle under load to 600°C. A literature review of transient thermal creep of concrete is presented. It reveals inadequate understanding of this subject for temperatures above 100°C and a lack of data for conditions pertinent to the analysis of concrete structures during first-time heating. This paper also presents results of preliminary work forming background information for the analysis of the transient thermal strain behaviour of unsealed concrete specimens. The complex temperature, moisture and thermal stress conditions developing during thermal transients in concrete test specimens have, therefore, been investigated experimentally and/or theoretically. The 'structural' effects and modification of material behaviour caused by these conditions have consequently been minimized by appropriate design of experiment. Characteristics of the individual aggregate and cement paste constituents have been determined by dilatometry, DTA and TGA tests which showed aggregate thermal stability to be a critical factor.

Notation

a	radius of specimen
r	radial dimension
t	time
z	axial dimension
D	thermal diffusivity
E	modulus of elasticity
K	maximum thermal stress
R	rate of heating
T	temperature
α	coefficient of thermal expansion
σ	stress
φ	hoop dimension
ν	Poisson's ratio
ΔT	maximum radial temperature difference

Introduction

Developments within the last decade have seen a marked increase in the number of structures involving the first-time heating of mature concrete under load[1-3]. These include nuclear reactor pressure vessels, storage tanks for hot crude oil and hot water, coal gasification and liquefaction vessels and pressure vessels used in the petrochemical industries. Added to these is the 'traditional' manner in which concrete structures can be exposed to fire, hot weather or solar radiation. Maximum temperatures range from near ambient to above 1000°C, which can be attained in minutes or days and can occur by design, nature or accident.

Although the different concretes used in these diverse structures are subjected to different environmental constraints, they commonly experience a marked increase in the rate of creep during initial heating under load. The resulting 'load-induced strain' significantly exceeds that expected from any increase with temperature of basic creep and elastic strains. The additional strain 'component' has been termed 'transitional thermal creep' (ttc‡)[4]. The strain behaviour of concrete during first heating under load cannot, therefore, be predicted from constant-temperature strain measurements.

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‡The expression has been spelt out for preference, but where it occurs frequently the abbreviation has been used. Ed.

Transitional thermal creep results in relaxation and redistribution of thermal stresses, rendering the elastic stress analysis inappropriate for structures heated for the first time. At the 'material' level, transitional thermal creep accommodates, to a large extent, the thermal strain incompatibilities between aggregates and cement paste, thus helping to avoid excessive damage to the concrete, particularly at temperatures above 100°C, when unsealed cement paste experiences considerable shrinkage while the aggregate continues to expand.

Excessive tensile thermal and incompatibility stresses can develop during cooling, when transitional thermal creep is absent, which could lead to the development of cracking and serious weakening of the material. An understanding of the transient thermal strain behaviour of concrete during both the virgin heating and subsequent cooling is, therefore, necessary for the successful analysis of heated concrete structures and for the better understanding of the thermal stability of the material itself.

Despite the importance of this subject and an increasing number of relevant applications, it is as yet inadequately understood and insufficiently documented, particularly for temperatures above 100°C[5] and for nuclear reactors, where research in material properties has lagged behind the development of analytical techniques[6].

The immediate aim of this work was to determine the transient thermal strain behaviour during first heating under uniaxial compressive load of four concretes of the types used for nuclear reactor pressure vessels. The imposed boundary conditions were based on a hypothetical situation involving the thermal excursion of the concrete to the temperature of the gas coolant (about 600°C) in the core of the reactor and subsequent cooling[7].

To realize this aim, it was important to design the apparatus[8] and experiments to reduce or eliminate factors which could obscure and/or distort fundamental trends in behaviour. The measures taken included (a) minimizing axial and radial temperature differentials within the specimen and hence 'structural' effects, (b) applying a constant-gravity loading, (c) ensuring as linear a rate of heating as possible and (d) performing replicate tests, which made it possible to employ statistical techniques. Complex temperature[9], stress, moisture and pore-pressure conditions prevail within the test specimen during first heating which can significantly influence its strain behaviour. These were investigated experimentally and/or theoretically.

The test programme was designed to incorporate a range of material and environmental factors to widen the applicability of the results to the transient thermal strain behaviour of concrete in general[7], and a total of eight different concrete and cement paste mixes were, therefore, tested. The coarse aggregates were siliceous gravel, limestone, basalt and lightweight. The cement blends included OPC, OPC/pfa and SRPC/slag.

By using differential thermal analysis (DTA), thermogravimetric analysis (TGA) and dilatometry tests, the phase changes of the cement pastes and aggregates were determined. The test programme therefore allowed assessment of (a) the contributions of the individual aggregates and cement pastes, (b) the restraining effect of the aggregate and (c) the relative thermal stability of the concretes.

Since thermal strain behaviour of concrete is also influenced by the imposed environmental conditions prevailing before as well as during the heating cycle, a number of environmental factors have been investigated. These were load level (0, 10, 20 and 30% of the initial strength), rate of heating (0·2 and 1°C/min), mode of moisture pre-conditioning (moist as cast, air-dried and oven-dried at 105°C), preheating and age of concrete (1 and 9 years).

This paper is the first of three papers to be published in the *Magazine of Concrete Research*. It presents results of studies forming the background information for the analysis of the transient thermal strain results which will be the subject of the other two papers. These studies include a literature survey of transient thermal creep of sealed and unsealed concrete, followed by a discussion on the temperature, stress and moisture conditions within the test specimen during thermal transients and ending with the behaviour of the individual constituents, aggregate's and cement paste's.

Historical review

Lea and Stradling commented in their classic 1922 paper[10] that "theoretically concrete should break down at about 100 deg. C" but also noted that "actually it often increases in strength up to about 300 deg. C". It was only after the early 1960s, with the discovery of transitional thermal creep, that this apparent contradiction between theory and observation could be resolved. Ttc was probably the first reported by Johansen and Best in 1962[11], and was soon afterwards confirmed by others[12–14].

The 1970s was a period of consolidation when it became clear that transitional thermal creep could be regarded as a separate 'component' which possesses its own distinct properties. Research effort was then directed in two areas, (a) sealed concrete for temperatures below 100°C and (b) unsealed concrete for temperatures ranging from ambient up to 800°C or failure.

SEALED CONCRETE BELOW 100°C

Illston and Sanders[4,15,16] were the first to report in some detail in 1973–4 the nature and properties of this component for sealed conditions, naming it 'transitional thermal creep'. Earlier work by Hansen

and Eriksson[13] and Nishizawa and Okamura[17] although less detailed, also provided some important and original insights. Notable contributions towards the end of the 1970s came from Parrott[18–21] and Bamforth[22].

Transitional thermal creep (ttc) develops irrecoverably during, and for a few days following, first-time heating of sealed concrete under load. It appears in addition to the increase in elastic strain and basic creep (flow and delayed elastic) components with temperature[15], from which it cannot therefore be predicted.

The rate of development of ttc appears to increase slightly with increase in temperature[21] but decreases with increase in maturity or age at heating[4,18]. In one example[21], half of the ttc of mature concrete formed in 4 days at 30°C and 2 days at 55°C. The period of development is about 7–10 days for ages at heating of 35–60 days[4,11,13,17] and increases to about 20–30 days for ages of about 200–2400 days[18,23]. Ttc is, therefore, generally fully developed within a month from the start of heating.

The limiting value of ttc is a non-linear function of temperature[11,15,21,22] and, unlike its rate of development, appears to be relatively unaffected by maturity[4,15,21]. However, the total creep induced by heating under load reduces with age and delay in heating after loading[18]. Although this may be due to a reduction in the basic creep component and/or increase in strength with time, the effect of age upon ttc is still not clear. The limiting value of ttc was also found to be of approximately the same magnitude, regardless of the number of steps taken to reach final temperature[4,15,22]. A typical limiting value for the ttc of concrete given in the literature is $0{\cdot}45 \times 10^{-6}$ per N/mm^2 per °C of virgin temperature rise[21].

The limiting value of ttc and its rate of development appear to be affected by high heating rates[13,16,18,21]. It was greater during heating at 2°C/min than at the slower rate of 0·033°C/min[13] and at 4°C/min it was four times that at 0·133°C/min[18]. These results suggest that ttc and the thermal strain behaviour of concrete in general are influenced by 'structural effects' within the test specimen which are enhanced by high rates of heating. Rapid heating of concrete was found to induce substantial temporary tensile strains in the centre of unloaded test prisms due to thermal gradients[18] and ttc tended to increase when the stress state was likely to involve cracking[18]. Applying a compressive load however, tends, to prevent tensile stresses from developing in the specimen[18]. By contrast, for slow rates of heating (0·006–0·02°C/min), a lower heating rate produced more creep[24], presumably owing to contributions from time-dependent deformations. Structural effects were also probably negligible. In one study[11], however, no effect from heating rate was observed between −20°C and +20°C.

Transitional thermal creep has generally been assumed to be linearly related to applied load and the concept of specific ttc has been invoked[15,17,21,22]. However, data on the effect of stress level are lacking since it has not been directly investigated. Torsional and flexural loading have tended to yield greater strain values than compressive loading[21,25], which could be due to an additional contribution from microcracking[21].

With a single exception[12], there is agreement that ttc does not take place during cooling.[4,13,15,17,18,22,26]. It was, furthermore, appreciated from the earliest studies that ttc is irrecoverable on cooling or unloading or both[4,11,13,17–20,22,25,26]. On reheating to the same upper temperature limit, ttc is reduced[13,18,22,25] or disappears altogether[4,17] depending upon whether it is allowed to develop fully during the first cycle and upon the period at ambient temperatures between the first and second heating cycles. Ttc is also reduced or suppressed if the concrete is subjected to a temperature cycle prior to loading [15,18], which limits the potential for further phase change in the material[19]. The effect depends upon the duration at the pre-heat temperature and upon the period between the two heating cycles. Ttc does, however, re-appear on second heating if the initial temperature limit is exceeded[4,17]. For the range of temperatures not previously encountered, ttc develops as on virgin heating. If ttc has not fully developed during the first cycle, it will continue at a reducing rate on further heating[22]. An instantaneous increment, attributed to cracking, was found to occur at the end of each reheating but this reduced with number of cycles[22]. A reduction of creep with thermal cycling also occurred in flexural tests[13] except that, on further cycling, creep began to increase again until the specimen failed owing to the development of cracking.

There is evidence[13,18,22] suggesting that a 'rest' period at ambient temperature after a thermal cycle (even one week[13]) could cause a regeneration of ttc potential. An early-age temperature cycle was also found not to affect ttc several years later[22], which clearly is of practical relevance to massive structures that experience a heat-of-hydration thermal cycle. This behaviour cannot, however, be explained on the basis of current understanding except in such vague terms as self-healing or regeneration[21,22]. Therefore, although it is possible to suppress ttc completely by preheating[15], it can reappear if the initial temperature limit is exceeded or if a long rest period at ambient temperatures is allowed before reheating.

Transitional thermal creep is seated in the cement paste and the aggregate tends to restrain it[13,18]. In one case, adding aggregate has reduced ttc of cement paste by about 70%[18]. Thermal incompatibility between the aggregate and cement paste cannot, therefore, be the cause of ttc[13]. Thermal stresses were

also found not to cause ttc[13,16] which appeared even at the very slow heating rate of 0·033°C/min with negligible temperature gradients. Therefore, although the development of cracking can influence ttc at high heating rates and/or under tensile loading, it is not its cause.

Contrary to earlier thinking[13,24], it is now believed[19] that internal moisture migration is unlikely to cause ttc since the redistribution of water in the micropores of sealed cement paste on heating takes place within a few hours, as compared with a period of some 20 days for the development of ttc. Furthermore, the gel pore-capillary moisture migration is to a certain extent reversible on cooling, whilst ttc does not occur during, nor is it recovered by, cooling.

A second school of thought attributed ttc to an increase in the elastic strain on heating[25,27] which is also unlikely, since the elastic strain can experience little change when concrete is heated under load, as will be shown in the subsequent papers. Furthermore, unlike elastic strains, ttc is irrecoverable.

The mechanisms causing ttc are, therefore, still not fully understood. An advance has recently been made[19] when a correlation was found to exist between irrecoverable creep, which includes ttc and flow, and the development of silicate polymerization. Heating at 60°C increased both the proportion of polysilicate and its degree of condensation, whereas at 20°C, only an increase in the degree of condensation was detected for ages between 28 and 220 days[19]. Interestingly, both ttc and polysilicate developed in about 20 days after heating[20]. Ttc did not appear to correlate with the C/S ratio, bound water or specific surface area, all of which changed little when mature cement paste was heated. The main chemical change was the formation of polysilicate, whilst the main physical change was an increase in the volume of large pores and a decrease in the volume of small pores[20].

Therefore, although ttc may be influenced by structural effects, the evidence to date suggests that it is a 'real' material property rather than an 'apparent' structural effect as has been suggested[28]. It is primarily caused by molecular and microstructural phase changes taking place in the cement paste during virgin heating under load.

UNSEALED CONCRETE ABOVE 100°C

Transitional thermal creep also occurs irrecoverably in unsealed concrete during first heating to temperatures considerably above 100°C[29–37] and up to failure under load. For mature concrete, it is by far the largest component of the load-induced thermal strain, several times larger than the combined increases with temperature of basic creep and elastic strain[32,33].

The unsealed condition allows escape of moisture which causes the concurrent development of drying creep. A different terminology has been adopted for this condition in which the terms 'transient creep'[30] or 'transient strain'[32–34], have been used to denote the combined effects of ttc and drying creep.

Weigler and Fischer[35–37] were probably the first to report, in 1967–68, that the thermal expansion of unsealed concrete is considerably reduced when heated for the first time under compressive load to 600°C. The nature of this reduction has been investigated in the 1970s primarily by two groups: Thelanderson[32–34] and Anderberg[33,34] in Sweden and Schneider[29–31] and Kordina[30] in Germany.

Although it is appreciated that the chemical reactions taking place during heating under load at temperatures above 100°C are time-dependent[32], it is believed that the transient creep response of concrete is 'quasi-instantaneous'[32] and, for practical purposes, it is considered to be time-invariant and a function of temperature only[30,32,33]. In this respect, it seems to differ from ttc which develops more slowly at lower temperatures. This has led to some uncertainty as to whether it should be termed transient creep[30] or strain[32]. The term creep will, however, still be adopted here, although it is appreciated that its period of development may be short.

The temperature function has been difficult to define precisely because of factors including a wide variation in the strength values of individual specimens[32,33], large radial temperature differentials (>100°C[32,33]) and non-linear rates of heating[33]. Transient strain increased non-linearly with increase in temperature, the slope being larger at higher temperatures for quartzite[32,33] and calcareous[31] concretes, but apparently levelling off at about 300°C for basalt concrete[31].

In none of the previous studies on unsealed concrete has the role of aggregate content been examined, since only concrete, and not cement paste, specimens have been used. The type of concrete examined has been primarily quartzite, except for a recent study by Schneider[31], and the role of aggregate type is still not clear. The strains during cooling have also not primarily been considered earlier in any detail.

Some studies showed an approximately linear increase in transient strain with increase in stress level[32–34]. Another[31] found no clear dependence.

Heating rate was found to be significant only at very high heating rates, as was concluded for lower temperatures. Heating rate had little influence upon transient creep for small specimens (75–80 mm dia.) within the range 0·5–5°C/min[30,33]. Larger (150 mm dia.) unloaded specimens exploded during heating at 4 and 8°C/min before the temperature at the centre of the specimen reached 200°C and when the temperature increase in the core of the specimen became stagnant, i.e. when evaporation of moisture took place[32]. The corresponding loaded specimens also failed at similar temperatures after experiencing tran-

sient creep which increased with increase in heating rate. This effect was clearly noticeable at a load equivalent to 60% of the initial cold strength, but not at 30% load. It seems, therefore, that the role of heating rate can be more pronounced at higher load levels. Such an effect was predicted theoretically[33] and related not to thermal stresses (deemed to be negligible owing to relaxation by transient creep) but rather to the stress redistribution and development of compressive stresses in the central region of the specimen caused by spatial variation in strength of the material as influenced by temperature gradients. Additionally, higher heating rates, particularly when combined with high load levels, appear to influence the total deformation[33] and the temperature at failure[32,33]. These examples serve to illustrate the importance of structural effects.

To date, the causes of transient creep at temperatures above 100°C have not been investigated. As with lower temperatures, transient creep is at the most general level likely to be correlated with concurrent phase changes in the microstructure of the cement paste.

Test specimens and materials

The specimens for the thermal strain tests were unsealed cylinders (62 mm dia × 186 mm long) with an optimum diameter small enough to minimize radial temperature differentials during thermal transients, but allowing the use of 12 mm aggregate. This restriction of maximum aggregate size instead of the 20 or 40 mm used in reactor vessel concretes was not expected to influence the qualitative trends or material comparisons. A 3:1 length:diameter ratio was chosen to reduce contributions from end effects at the central portion of the specimen. The top surfaces of the specimens were ground flat, parallel and orthogonal to their vertical axis.

TABLE 1: **Details of the five concrete mixes.**

	Mix designation				
	H	BI	BII	GI	LI
	Limestone	Basalt		Gravel	Lightweight
CONSTITUENTS (proportions by dry weight)					
Cement (OPC)	1·0	1·0	—	1·0	1·0
Cement (SRC)	—	—	0·5	—	—
pfa	0·25	—	—	—	—
Cemsave	—	—	0·5	—	—
Sand	2·3	1·8	1·8	—	—
Fine sand	—	—	—	0·65	—
Coarse sand	—	—	—	1·55	—
Fine aggregate	—	—	—	—	0·87
Medium aggregate	—	—	—	—	0·69
Coarse aggregate	3·45	2·7	2·7	2·39	0·69
Water (total)	0·555	0·45	0·45	0·60	0·80
Plasticizer	0·005	0·005	0·005	—	—
PROPERTIES					
Water/blend (effective)	0·444	0·45	0·45	0·6	0·52
Aggregate/blend	4·6	4·5	4·5	4·59	2·25
Cement content (kg/m^3)	388	415	420	365	447
Density as cast (kg/m^3)	2346	2472	2498	2259	1810
Aggregate content (% by volume)	67·8	67·3	67·2	65·0	65·3
STRENGTHS (N/mm^2)					
28 day (cube)	51·0	61·0	50·9	34·1	47*
Before test (cylinder):					
Moist	64·2	61·3	56·5	31·0	49·5
Air-dry	71·9	74·0	67·0	39·3	46·6
Oven-dry	54·9	62·1	70·4	34·8	45·6

* = interpolated

TABLE 2: **Chemical composition of cements and aggregates.**

Component	Cementitious material				Aggregate				
	OPC	SRPC	pfa	Cemsave	BI & BII basalt	BI sand	BII sand	H limestone	H sand
Lime (CaO)	63·20	63·90	8·38	43·6	9·35	14·84	12·74	52·50	4·13
Silica (SiO_2)	19·90	20·66	42·51	34·16	51·98	66·02	67·83	1·28	78·91
Alumina (Al_2O_2)	6·73	3·22	33·80	14·10	15·00	2·95	3·59	1·52	8·74
Ferric oxide (Fe_2O_3)	2·20	4·72	6·10	0·34	12·75	2·86	1·99	0·38	1·88
Magnesia (MgO)	1·30	2·16	1·71	4·68	6·56	0·50	0·38	1·54	0·84
Sulphuric anhydride (SO_3)	3·00	2·32	0·61	trace	trace	trace	—	0·07	0·07
Potassium oxide (K_2O)	0·96	0·55	0·61	0·47	0·60	0·56	—	0·01	0·07
Sodium oxide (Na_2O)	0·28	0·17	0·18	0·24	2·40	0·30	0·92	0·02	0·02
Carbonic (CO_2)	—	—	0·76	—	0·24	11·65	—	40·20	1·78
Loss-on-ignition	1·20	1·17	3·19 ex CO_2	—	—	—	10·88	—	—
Insoluble residue	0·73	0·50	—	—	—	—	—	—	—
Combined water	—	—	—	—	0·40	0·27	—	0·58	1·95
Total carbon	—	—	3·80	—	0·07	3·18	—	12·00	0·53
Total sulphur	—	—	—	1·73	trace	trace	—	—	—

Eight different concrete and cement paste mixes were tested. Details of the five concretes, and their designations, are given in Table 1. Four of these (H, BI, BII and GI) were concretes of the types used for the prestressed concrete reactor vessels (PCRVs).

The aggregates were crushed dolomitic limestone, crushed quartz dolerite basalt, natural weathered Thames River siliceous gravel and lightweight (sintered pulverized-fuel ash – Lytag). The fine aggregate was natural siliceous sand (Zone 2) in all concretes except for the BII and lightweight mixes, which were made with calcareous quartz sand (Zone 2) and Lytag fines respectively.

Two types of cement were used, ordinary Portland (OPC) and sulphate-resisting Portland (SRPC). Cement replacements included pulverized-fuel ash (pfa) and ground granulated slag (Cemsave).

The three cement pastes (CPl, CP2 and CP3) were OPC, OPC/pfa (25% pfa), and SRPC/Cemsave (50% Cemsave) respectively, with water/cement ratios of 0·3, 0·3 and 0·32 respectively.

The chemical composition of the various cements, cement replacements, aggregates and sands used in the mixes is given in Table 2.

Outline of preliminary tests

CONDITIONS WITHIN SPECIMEN

The temperature distribution in the specimen during heating was determined by using 10 chromel–alumel thermocouples cast within both gravel and lightweight concrete specimens representing upper and lower bounds of thermal diffusivities. Other parameters investigated included initial moisture content, preheating and rate of heating[9].

Weight loss and rate of weight loss measurements were made on the concrete and cement paste specimens as used in the strain tests. They were heated at 0·2 and 1°C/min, from three initial moisture conditions (moist, air-dry and oven-dried at 105°C), in a single-zone furnace mounted over a Mettler P11 balance. Weight readings were recorded to 0·01 g. Temperature was monitored on the centre surface of the specimen by a single-fine-wire thermocouple so as not to influence the weight readings.

BEHAVIOUR OF INDIVIDUAL CONSTITUENTS

Dilatometry tests were performed on individually cut samples (20–30 mm rectangular blocks) of aggregate and cement paste during constant heating and subsequent cooling at a rate of 2°C/min in rough vacuum (~0·1 mm Hg) using a Netzsch 402E quartz tube dilatometer.

Differential thermal (DTA) and thermogravimetric (TGA) analyses were also performed on individual samples of aggregate and cement paste during heating at 10 and 1·5°C/min respectively, in nitrogen using a Stanton Redcroft apparatus.

Conditions within specimen and structural effects

Structural effects are defined here as those induced by significant spatial variation in stress, strain, moisture content, pore pressure, material properties etc., within the specimen caused by large temperature gradients during rapid heating or cooling and which distort its thermal strain behaviour. The effect of differential thermal movement between the aggregate and cement paste is regarded here as essentially material, rather than structural, since it is to a large extent dependent upon the choice of constituents.

Three examples of structural effects have been cited in the literature. (1) Rapid heating was found to induce substantial temporary tensile strains in the centre of unloaded specimens[18] which, as shown below, are due to thermal stresses. (2) By contrast, a build-up of compressive stresses in the centre of loaded specimens was predicted for quartzite concrete above 300°C, when it experienced a sharp decline in strength[33]. Spatial variation in strength resulting from large temperature gradients caused a transfer of load from the weaker hotter surface region to the inner cooler and higher strength central zone. (3) Under extreme conditions, explosion of the specimen occurred[32], when the temperature increase in the core of the specimen became stagnant, i.e. when moisture evaporation took place.

TEMPERATURE DISTRIBUTION

Analytical solution of the Fourier transient heat conduction equation[9] for a specimen of radius a and thermal diffusivity D during heating at a constant rate R, assuming infinite length and invariant material properties, indicates that the radial temperature distribution $T(r,t)$ developed, beyond the initial transients, at time t is:

$$T(r,t)=R\left(t-\frac{a^2-r^2}{4D}\right) \quad (1)$$

The maximum radial temperature difference ΔT $[=T(a,t)-T(0,t)]$ therefore is:

$$\Delta T=Ra^2/4D \quad (2)$$

Radial temperature gradients for a given material can therefore be minimized by reducing the rate of heating and particularly the radius of the specimen. Both these measures were implemented in this work.

A finite difference solution[9] of the Fourier equation confirmed these relationships for a specimen of finite length. The temperature distribution at the centre of the specimen remained unaffected by end heating, provided the length/diameter ratio was greater than 1 to 1·5 during heating at 1°C/min.

An experimental investigation[9] confirmed these trends and showed that the effect of variation in material properties (thermal diffusivity) is to cause two peaks in the ΔT versus surface temperature relationship at about 160°C and 550°C corresponding to the processes of moisture loss and dissociation of $Ca(OH)_2$ respectively. The first peak was understandably removed by initial oven-drying at 105°C, whilst preheating the specimen at 600°C stabilized ΔT throughout the test temperature range. Peak values of ΔT for gravel and lightweight concrete specimens, representing upper and lower bounds of thermal diffusivities, did not exceed 24°C and 12°C respectively, during heating of 60 mm dia. specimens at 1°C/min. The peak ΔT at 0·2°C/min would be a fifth of these values and hence negligible.

THERMAL STRESS DISTRIBUTION

During heating

Although thermal stresses are relaxed by transient creep during virgin heating, they can still be significant[18]. On the basis of deductions from the literature for sealed specimens heated to temperatures below 100°C[13,18], thermal stresses may occur at rates of heating in excess of 2–4°C/min (cf. maximum of 1°C/min in this work). The situation will be less severe in unsealed specimens since drying-shrinkage strain gradients develop in the opposite direction to those of thermal expansion. Thermal stresses were estimated[33] to be negligible in unsealed specimens during heating above 100°C at rates even higher than 1°C/min because of the magnitude and rapid rate of development of transient creep.

An upper bound of thermal stresses during heating can be obtained from an elastic thermal stress analysis for the specimen[7]. An analytical solution, for an infinitely long specimen, shows that the axial thermal stress distribution $\sigma_z(r)$ at radial distance r, resulting from the temperatures given in equation 1, and expressed in terms of the modulus of elasticity E Poisson's ratio ν and coefficient of thermal expansion α is:

$$\sigma_z(r)=\frac{\alpha ER}{8D(1-\nu)}(a^2-2r^2) \quad (3)$$

which is zero at $r=a/\sqrt{2}$ and maximum tensile at the central axis ($r=0$):

$$\sigma_z(0)=\frac{\alpha ERa^2}{8D(1-\nu)}=\frac{\alpha E\Delta T}{2(1-\nu)}=K \quad (4)$$

and hence directly proportional to the maximum radial temperature difference ΔT $(=Ra^2/4D)$. Likewise, it can be shown that all maximum axial, radial and hoop stresses are fractions of K (Table 3), whilst shear stresses are zero throughout the cross-section. Thermal stresses can, therefore, also be minimized by

TABLE 3: **Calculated thermal stresses (positive = tensile) in cylindrical specimen during heating at constant rate without relaxation by creep.**

Type of stress	Magnitude of stress		Radial position of zero stress
	$r=0$	$r=a$	
Axial (σ_z)	K*	$-K$	$r=\pm a/\sqrt{2}$
Radial (σ_r)	$K/2$	$-K$	$r=\pm a/\sqrt{3}$
Hoop (σ_φ)	$K/2$	0	$r=\pm a$

*$K=\alpha E\Delta T/2(1-\nu)$

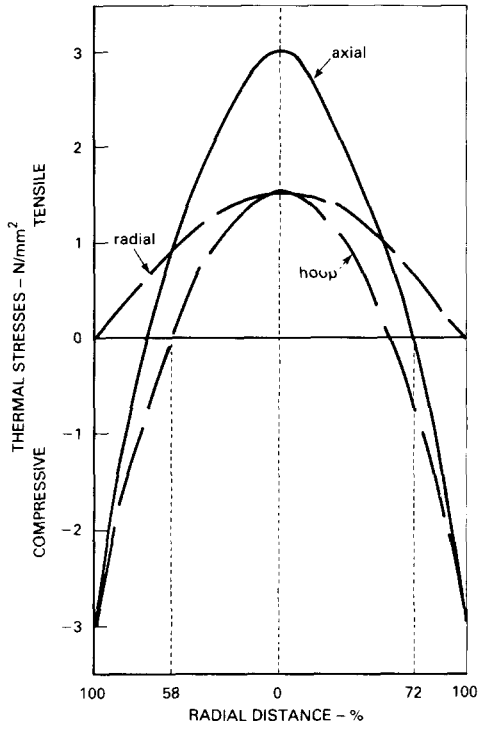


Figure 1: Calculated thermal stress profiles in the central cross-section of gravel concrete specimen (60 mm dia.) during heating at 1°C/min, without relaxation by creep.

reducing the rate of heating and particularly the radius of the specimen.

Maximum tensile stresses occur at the specimen's centre during heating (as reported empirically[18]), the axial component being the largest and twice the value of both the radial and the hoop stresses. The stresses at the specimen's surface are, on the other hand, compressive ($= -K$) except for the radial stress which, as expected, is zero (Figure 1). A finite element elastic stress analysis[7] confirmed these relationships for a specimen of length/diameter ratio of 3:1 heated on all sides (Figure 2).

During cooling and reheating

Thermal (and incompatibility) stresses assume greater importance during cooling and reheating because of the absence of transient creep. The elastic analysis given above is more applicable here except that the stresses during cooling would be reversed. In other words, tensile stresses can develop near the surface of the specimen during rapid cooling and could lead to cracking which will be confined to the surface region. In contrast, tensile stresses due to thermal incompatibility may occur throughout the specimen. Published data[38] suggest that the residual strength of concrete specimens similar to those used in the current tests is unlikely to be affected for rates of cooling below 2°C/min.

MOISTURE CONDITION AND PORE PRESSURE

The weight loss of a specimen during first-time heating to 500–600°C was taken to represent the amount of moisture escaping from its surface. A typical result for rate of weight loss per unit surface area of the specimen is shown in Figure 3 for limestone concrete heated at 1°C/min.

The cement paste lost moisture which was initially retained in various forms ranging from the free capillary, physically adsorbed, 'interlayer' and chemically bound water in the CSH and $Ca(OH)_2$. The aggregates used did not decompose below 600°C; any contribution to weight reduction came from moisture present in the aggregate pores and was negligible except for the lightweight aggregate.

Complex interrelated heat and mass transfers

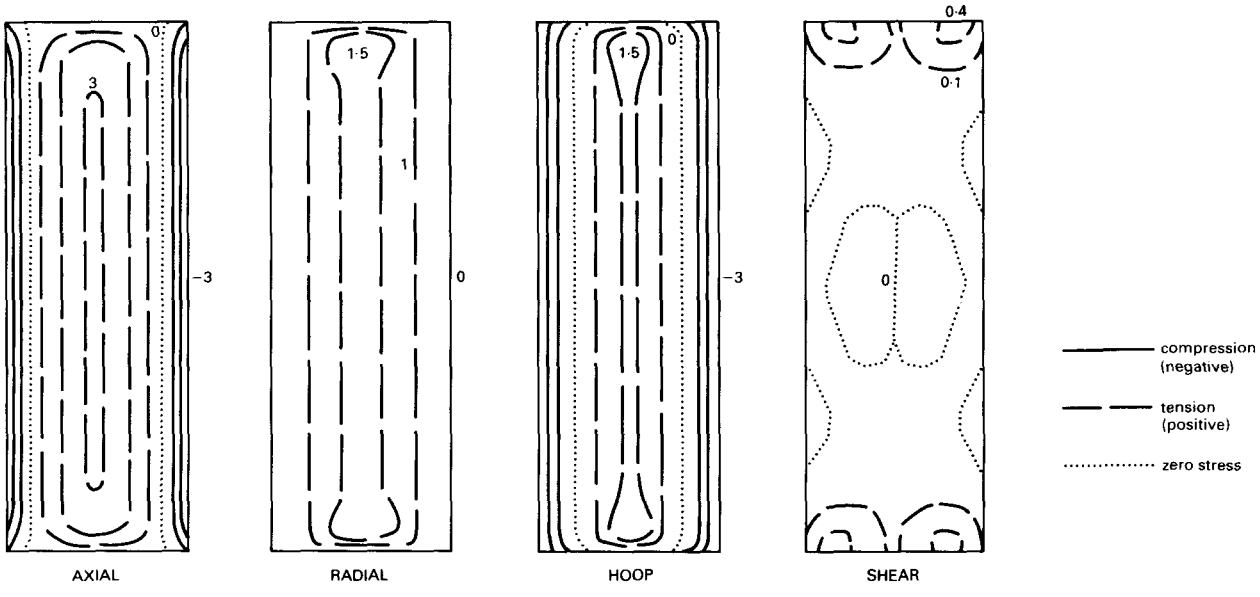


Figure 2: Calculated thermal stress contours (N/mm²) in gravel concrete specimen (60 mm dia. × 180 mm) during heating at 1°C/min, without relaxation by creep.

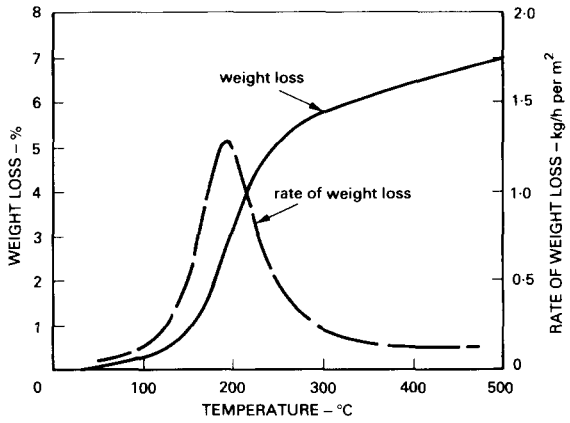


Figure 3: Weight loss of limestone concrete specimen measured during heating at 1°C/min from the initially moist condition.

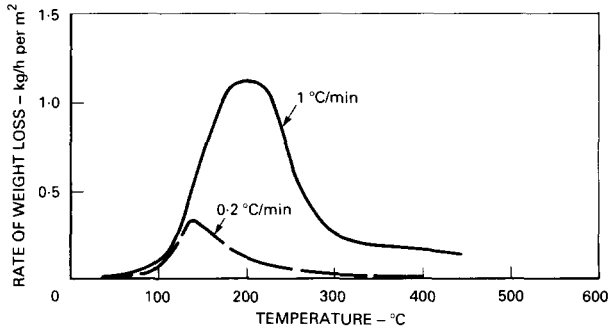


Figure 5: Effect of rate of heating upon the rate of weight loss of basalt II concrete specimens measured during heating from the initially moist condition.

occurred within the specimen during heating, the moisture flux being dependent upon both temperature and moisture gradients and probably best explained in terms of pore pressures. The temperature gradient can initially drive some moisture inwards, causing moisture and pore pressure build-up in the central region at the same time as free capillary water escapes from the surface layer. The pore pressure developed within the specimen at 1°C/min was estimated from published data to be less than 0·2 N/mm^2[7]. This is less than about 7% of the tensile strength of the concretes tested and would be even lower at 0·2°C/min.

Three-zones can be envisaged to exist within the specimen during heating above 100°C: an external dry zone followed by an intermediate evaporation zone and an inner moist, possibly water-clogged zone. At temperatures above 250–300°C, the dry zone engulfed the whole specimen as indicated by Figure 4. The specimen, however, continued to lose weight due to escape of CSH-bound water, dissociation of $Ca(OH)_2$ above 400°C etc.

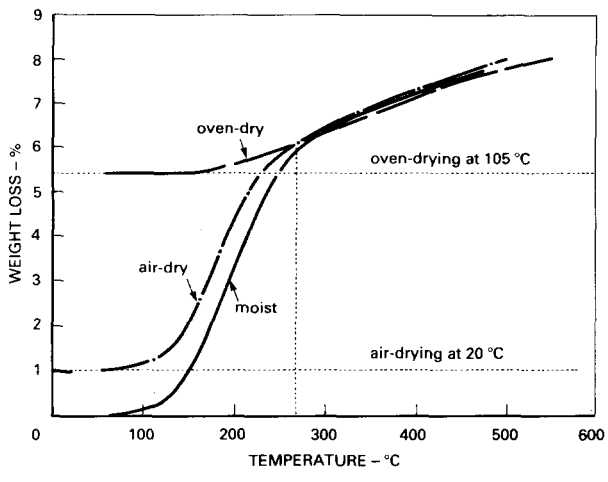


Figure 4: Effect of initial moisture condition upon the weight loss of basalt II concrete specimens measured during heating at 1°C/min.

It was found that only 3% of the evaporable water content escaped during initial heating between 20°C and 100°C at 1°C/min. Even at 0·2°C/min only 9% had escaped. Therefore, over 90% of the evaporable water is still retained when the specimen reaches 100°C, suggesting that hydrothermal (autoclave) conditions may exist during first heating even in small unsealed specimens. This may cause a transformation of the CSH into a weaker or stronger hydrate, depending upon the CaO/SiO_2 ratio.

The sharp increase in the rate of moisture loss above 100°C for all mixes confirmed that the permeability of concrete increases markedly above that temperature, as reported recently by other workers[39,40]. The rate of moisture loss during slow heating reached a peak at about 135–160°C (Figure 5), whilst during fast heating, the peak was three times greater and occurred at higher temperatures (175–200°C).

However, the total moisture lost for any given temperature was greater during slow heating (Figure 6) because of the five-fold increase in time to reach the temperature. Also the specimens remain under hydrothermal conditions for a longer time during slow heating.

On further heating to above 250–300°C, the rate of moisture loss declined sharply, becoming independent of the initial moisture content (Figure 4) and also similar for both rates of heating (Figure 6). Additionally, during transient heating above about 250°C, the total amount lost for any given temperature during the transient also approached the maximum loss attained in constant-temperature tests (Figure 6).

The dissociation of $Ca(OH)_2$ was not clearly evident, but in normal concretes, this causes a weight loss of only about 1·5% (less with pfa or slag concretes) and this is spread over a range of temperatures.

The temperature at the peak of moisture loss rate was similar for the cement paste and concrete specimens since, for uncracked specimens, moisture migration is governed by the permeability of the cement paste matrix. Furthermore, no correlation was found between the water/cement ratio and the peak

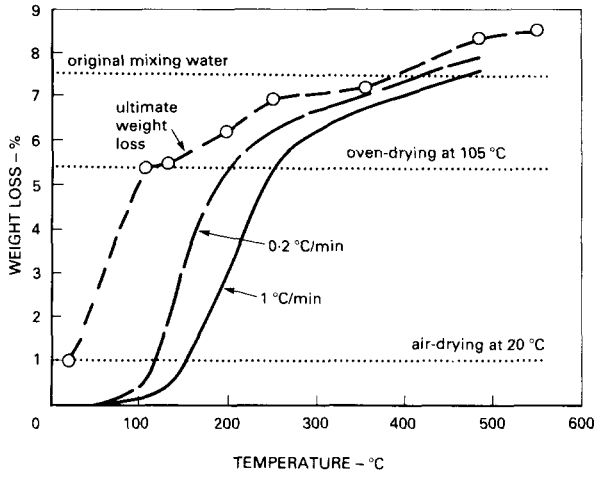


Figure 6: Weight loss at two rates of heating compared with ultimate weight loss (Basalt II concrete, initially moist condition).

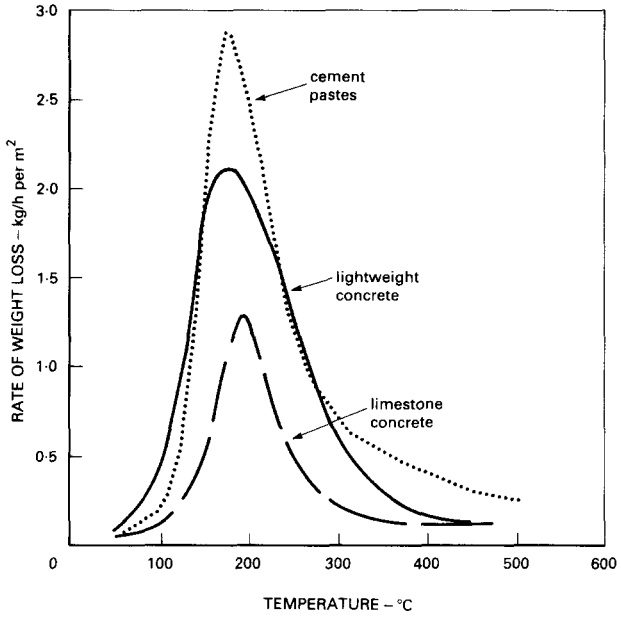


Figure 7: Effect of type of mix upon the rate of weight loss of specimens measured during heating at 1°C/min from the initially moist condition.

temperature, since the discontinuity of the capillary pore structure ensured that moisture migration takes place through the gel pore system.

The peak rate of moisture loss was, however, influenced by factors which determine the total amount of water present initially in the mix. These include the cement paste content, water/cement ratio, initial moisture content and porosity of the aggregate. The peak rate of moisture loss therefore increased successively for the limestone concrete, lightweight concrete and pure cement paste (Figure 7).

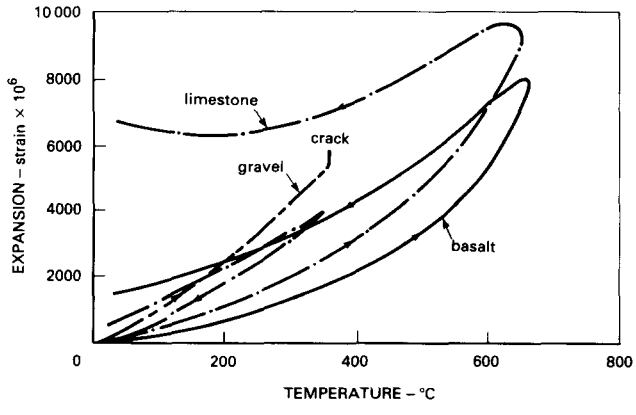
Behaviour of the individual constituents

DILATOMETRY RESULTS

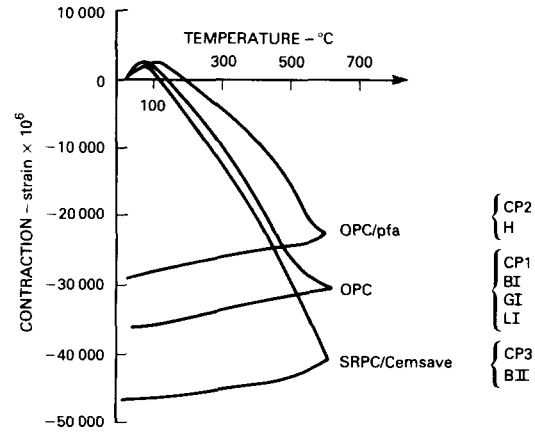
Dilatometry of the aggregates indicated that thermal stability progressively increased for the gravel, limestone, basalt and lightweight aggregates (Figure 8).

The expansive cracking of the gravel aggregate at about 350°C renders it unsuitable for use in concrete destined for heating above that temperature. Damage, though less severe, of the limestone and basalt aggregates is evident from their residual expansions, the former being less stable and experiencing a residual expansion after heating to 350°C. Lightweight aggregate was expected to be dimensionally stable even at 600°C, as it is manufactured above 1000°C.

The dilatometry tests on the cement pastes (Figure 8) showed that their free thermal strain was significantly influenced by the type of cement and cement replacement used. This was due to differences in thermal deformations of the various anhydrous and



(a) Aggregates



(b) Cement pastes

Figure 8: Free thermal strain of small rectangular blocks (20–30 mm) of aggregate and cement pastes (initially moist) measured during first heat cycle (2°C/min) to 600°C.

hydrated constituents[41] present in different proportions in each blend. Following initial expansion, the cement pastes underwent considerable shrinkage above 100°C, as they lost evaporable water and dehydration of the various hydrates proceeded at different temperatures. Shrinkage reached a minimum at about 500–600°C when (1) the dehydration processes were past their peak rates, (2) thermal expansion of unhydrated cement grains continued and (3) microcracking was enhanced[41]. Thermal shrinkage increases in order for the OPC/pfa, OPC and SRPC/Cemsave mixes. Thermal movement on subsequent cooling was at least an order of magnitude less for all the pastes.

It is not surprising when comparing the thermal expansion of the aggregate with the shrinkage of the cement paste above 100°C that previous scientists have been concerned about this thermal incompatibility and its effect upon the thermal resistance of the composite concrete. However, the cement paste will be shown in the next paper to exhibit significant transient creep which can accommodate this incompatibility to a great extent. The thermal stability of the aggregate is probably the more crucial factor during first heating.

Another important feature of the results is that the bulk of change took place during first heating. The expansion of both aggregates and cement pastes during the second cycle of heating was reversible[7].

DTA RESULTS

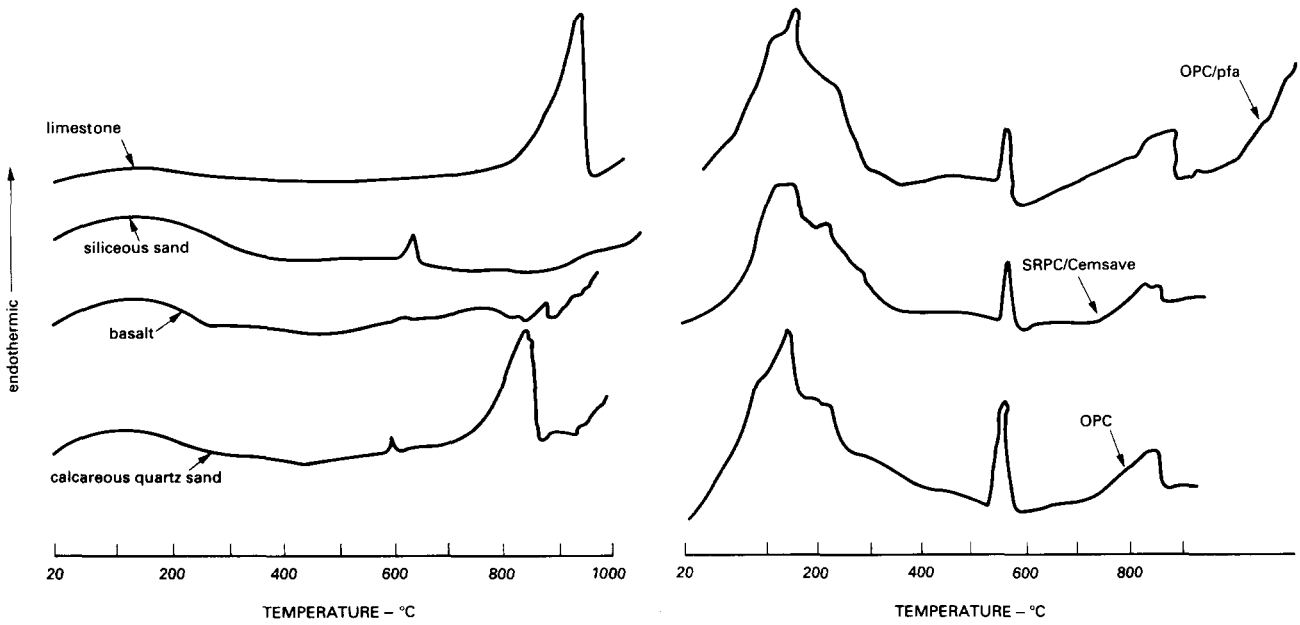
The three cement pastes exhibited similar over-all DTA trends (Figure 9) with endothermic peaks at about 150°C and 550°C due to moisture loss and dissociation of $Ca(OH)_2$ respectively. The role of both pfa and Cemsave in reducing the amount of $Ca(OH)_2$ formed during hydration is evident in the smaller endothermic peak areas of the cement blends. It should be noted that the DTA peak temperatures are functions of heating rate and do not represent the temperature at the start of the reaction, which for $Ca(OH)_2$ may be about 400°C.

DTA of the aggregates (Figure 9) shows them to be thermally stable in nitrogen up to the test temperature of 600°C except for endothermic peaks in the natural siliceous and calcareous quartz sands due to the α-β inversion of quartz at 573°C. The basalt aggregate did not undergo phase change up to at least 800°C and the peaks produced by the limestone aggregate and calcareous quartz sand at higher temperatures were due to decarbonation. The dimensional instability demonstrated in the dilatometry tests for the basalt and limestone aggregates may, therefore, have been of physical origin.

TGA RESULTS

Unlike shrinkage, the TGA weight loss of the three cement pastes (Figure 10) appears to be approximately similar for temperatures up to about 550°C, which suggests that the shrinkage–weight-loss relationship depends upon the cement blend. Above 550°C, the OPC/pfa mix experienced the greatest weight loss. As expected, the OPC paste underwent slightly more weight loss between 400°C and 500°C than the cement blend pastes, but this is not easily discernible in Figure 10.

The TGA profile of cement paste (Figure 10) is more comparable with the 'ultimate' than the 'tran-



(a) Aggregates *(b) Cement pastes*

Figure 9: DTA of (a) Aggregates and (b) cement pastes during heating at 10°C/min in nitrogen (measured on a time base).

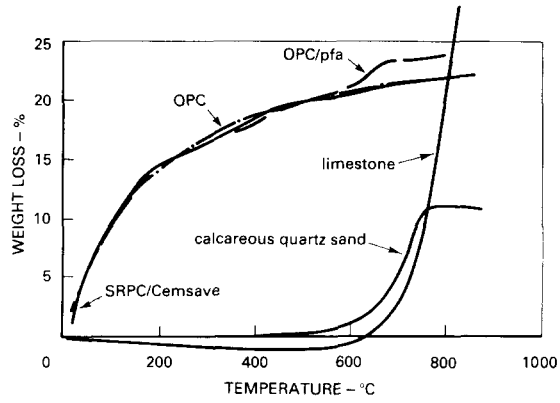


Figure 10: TGA of aggregates and cement pastes during heating at 1·5°C/min in nitrogen.

sient' weight loss profile of the larger test specimens (Figure 6). This demonstrates the influence of size of specimen upon the rate of moisture loss during heating.

The basalt aggregate and natural siliceous sand did not experience weight change during heating to 800°C. The weight loss accompanying decarbonation of the limestone aggregate and calcareous sand above 600°C is shown in Figure 10.

Summary and conclusions

The literature survey revealed the lack of data on the transient thermal strain behaviour of concrete above 100°C. Tests performed to date had been mainly on quartzite concrete and no tests had been performed on cement paste. Heating rates used were more relevant to fire applications and resulted in large temperature differentials within test specimens. These introduced structural effects which made correlations difficult between the measured strains and concrete temperature.

CONDITIONS WITHIN SPECIMEN AND STRUCTURAL EFFECTS

The complex temperature, stress and moisture conditions within the specimen have been investigated and radial temperature differences and structural effects minimized by reducing the specimen radius to 31 mm and the heating rate to <1°C/min.

Temperature distribution

The maximum radial temperature difference in the central region of a cylindrical specimen, with a length/diameter ratio greater than about 1·5, was found to be linearly related to the rate of heating and inversely related to the thermal diffusivity, and a function of the square of the radius:

$$\Delta T = Rr^2/4D$$

Two peaks in the radial temperature difference occurred and were due to moisture loss (150°C) and dissociation of $Ca(OH)_2$ (550°C). Peak values of ΔT for gravel and lightweight concrete specimens, representing upper and lower bounds of thermal diffusivities, do not exceed 24°C and 12°C respectively during heating at 1°C/min and would be a fifth of these values at 0·2°C/min.

Thermal stress distribution

Upper bounds of thermal stresses during heating were obtained from an elastic stress analysis for the specimen. An analytical solution, confirmed by a finite element study, showed that thermal stresses, which are tensile at the centre of the specimen during heating, are proportional to K and ΔT:

$$K = \frac{\alpha E R a^2}{8D(1-\nu)} = \frac{\alpha E \Delta T}{2(1-\nu)}$$

Thermal stresses are therefore also minimized by reducing the rate of heating and particularly the radius of the specimen. However, stresses are relaxed by transient creep during first heating, but not during cooling when they are reversed.

Moisture condition and pore pressure

Hydrothermal conditions may exist even in the small unsealed specimens, since over 90% of evaporable water remains when the temperature reaches 100°C during heating at 0·2–1°C/min., but pore pressures are estimated to be less than 0·2 N/mm^2. Rate of moisture loss increases markedly above 100°C and peaks at about 150–200°C regardless of type of mix. For saturated specimens, maximum rate of moisture loss depends upon the initial moisture content and hence increases in the order: normal-weight, lightweight and cement paste. Total moisture loss is higher during slow heating, but both the rate, and temperature at peak, of moisture loss are reduced. Above 250°C, weight loss is independent of initial moisture content.

BEHAVIOUR OF INDIVIDUAL CONSTITUENTS

Thermal stability of the aggregate progressively increased for: gravel, limestone, basalt and lightweight aggregates. Gravel expanded as it cracked above 350°C, whereas limestone and basalt produced residual expansion.

The large shrinkage of cement pastes above 100°C progressively increased for: OPC/pfa, OPC and SRPC/slag blends, but thermal movement on cooling of all pastes was an order of magnitude less. Cement pastes made with pfa or slag contained less $Ca(OH)_2$, which results in reduced DTA endothermic peaks. DTA endotherms of quartz and carbonate aggregates were caused by quartz inversion at 573°C and by decarbonation above 600°C respectively.

TGAs of different cement blends appeared similar up to 550°C, suggesting that shrinkage–weight-loss relationship depends upon cement blend. Above 550°C, OPC/pfa loses most weight. The TGA profile was more comparable to 'ultimate' than 'transient' weight loss of large specimens, demonstrating the importance of size of specimen.

No change was indicated by DTA or TGA in the basalt aggregate or natural siliceous sand up to 800°C, but decarbonation of other aggregates caused weight loss above 600°C.

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