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Deformation of NHL3.5 and CL90/PC hybrid mortars

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The time-dependent deformation of an NHL 3.5 natural hydraulic lime mortar, a 1 : 6 Portland cement mortar, two Portland cement/CL90 hybrid mortars and a CL90 lime paste, through creep and drying shrinkage under load, has been investigated. An apparatus was constructed to measure the dimensional changes of eight individual samples simultaneously. A reversible relationship between changes in specimen dimension and relative humidity has been established. The influence of load on deformation rate was found to be most significant during the first two weeks of monitoring with little effect thereafter. The rate of deformation during the first 2 weeks of sample monitoring was greatest for mortars containing a high proportion of calcium hydroxide. A linear relationship was found between strain rate and calcium hydroxide content.

1. INTRODUCTION

Lime mortars have been used in construction for thousands of years but during the last century Portland cement (PC) became the most widespread cementitious binder for most applications.[1,2] The high strength of PC combined with its relatively short setting time has allowed architects and engineers to design larger structures and reduce construction time. However, mortars manufactured using PC exhibit negligible plastic deformation under load and fail by brittle fracture. This can lead to the development of large internal stresses which are often dissipated by the formation of unsightly cracks and a reduction in overall integrity. Inappropriate use of cement during repair of historic masonry often leads to damage due to differences in strength and permeability in comparison with the original lime mortar.[3] Damage occurring in historic masonry has also been stated to be due to the large amount of soluble salts in PC.[4,5]

Greater environmental awareness of the advantages of lime mortars in terms of carbon dioxide emissions released during manufacture and the adsorption of carbon dioxide during carbonation is promoting their use. A number of different limes are available for building, differing by their strength and chemistry. Fat limes harden by carbonation but hydraulic lime mortars do so through a combination of hydration and carbonation. For hydraulic mortars, the initial hydration reaction results in the formation of a calcium–silicate–hydrate (C–S–H) structure, which provides an initial set.[6–8] Subsequently the remaining bulk calcium hydroxide then reacts with atmospheric carbon dioxide. The increase in volume associated with carbonation fills surface pores and cracks, reducing the number of diffusion paths and restricting the diffusion of carbon dioxide to greater depths.[9] In thick-walled structures this can result in the mortar located at the centre of the wall remaining uncarbonated for many years.

The ability of lime mortars to accommodate movement is considered to be a consequence of their ‘plastic’ and ‘self-healing’ properties. Cracks may provide a route, by which carbon dioxide diffuses into the mortar, reacts within the fracture and restores strength. If the mortar is fully carbonated, dissolution and re-precipitation of calcium carbonate by the movement of moisture through the structure may also contribute to strength.[10] These mechanisms are commonly associated with autogeneous healing.

The ability of lime mortars to accommodate movement can alleviate the use of expansion joints in a structure.[11] The omission of thermal expansion joints in modern construction has a number of advantages which appeal to architects and engineers. These include a reduction in cost and a more aesthetically pleasing finish. However, when omitting thermal expansion joints in a lime-built structure consideration must be given to both the mechanical and chemical behaviour of the mortar in service. Due to differences in the geometry, material properties and diffusion of moisture and carbon dioxide throughout the structure, it is highly improbable that a thick solid wall will behave in a similar way to a thin cavity wall. In the context of this paper a thick wall would be considered to have a thickness in excess of 215 mm whereas the outer skin of a typical thin cavity wall used would be 103 mm.

Dimensional changes of mortar within a structure, over time, will lead to changes in the magnitude and direction of forces both within the mortar and masonry units. The purpose of this work was to measure creep and shrinkage in lime mortars and determine whether the deformations observed are significant in a building context.

A number of studies have been undertaken by various researchers into the creep behaviour of concrete.[12–14] However, the authors are unaware of any which address lime mortars. The term creep can be used to describe the deformation of a material with time or the reduction in stress of a material under constant strain. Within this paper creep is defined as displacement attributable to the application of constant stress.

Previous research into concrete has identified shrinkage as an additional process which occurs simultaneously with creep. It is common practice to consider these two phenomena to be additive and this approach is suitable for many practical applications where these two processes occur together. However this approach should be treated with caution as it has been established that the effect of shrinkage in concrete is to increase the magnitude of creep.[1] There is mounting evidence that shrinkage and creep occur by quite different mechanisms.[14]

A number of theories describing creep have been proposed; however, it is probably justified to say that as they currently stand none is capable of explaining all of the observations made. This may be because the creep response of a cement or concrete material is a function of a number of mechanisms operating simultaneously. If this is indeed the case the contribution of each process to the total deformation measured may be a function of a number of specimen and environmental factors. The dominance of different mechanisms would account for varying behaviour.

From a phenomenological standpoint a number of broad mechanisms for creep in concrete can be distinguished. These include: mechanical deformation theory; viscous flow; plastic flow; seepage of gel water; delayed elasticity; and microcracking. Evaporation and the subsequent loss of surface adsorbed water from calcium silicate hydrate (C–S–H) and hydrostatic tension from small capillaries are the mechanisms ascribed to drying shrinkage.[15]

Whereas fat lime mixes rely on carbonation the natural hydraulic limes contain a siliceous phase that is normally characterised by the presence of dicalcium silicate. This also appears in Portland cement-based mixes, although the primary reaction for these materials is driven by the more reactive tricalcium silicate, which is essentially absent in natural hydraulic limes. Certain mechanistic properties associated with Portland cements may, however, feature in the behaviour of natural hydraulic limes. The addition of fat lime would reduce the silicate content in the mortar and could modify the creep behaviour as demonstrated by the experimental results.

2. EXPERIMENTAL METHOD

2.1. Raw materials and sample mix designs

All mortars were prepared using quartz-based Croxden sand as the aggregate with the particle size distribution depicted in Figure 1. An energy-dispersive X-ray analysis carried out on a Hitachi S-2300 scanning electron microscope indicated the presence of silicon, oxygen, iron, potassium, calcium, sulfur and titanium, in order of decreasing signal intensity (Figure 2). Five different mix designs were used (see Table 1). The materials CL90, NHL3.5 and PC were supplied by Castle Cement, Clitheroe, Lancashire, UK. Sufficient water was added to provide a mix flow between 180 and 200 mm using a slump test.[15]

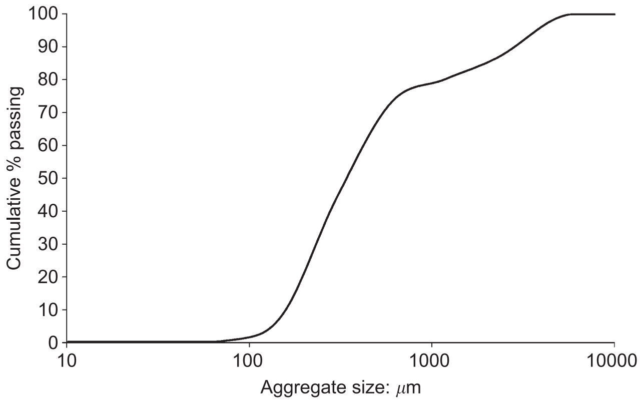

Figure 1. Croxden sand particle size distribution

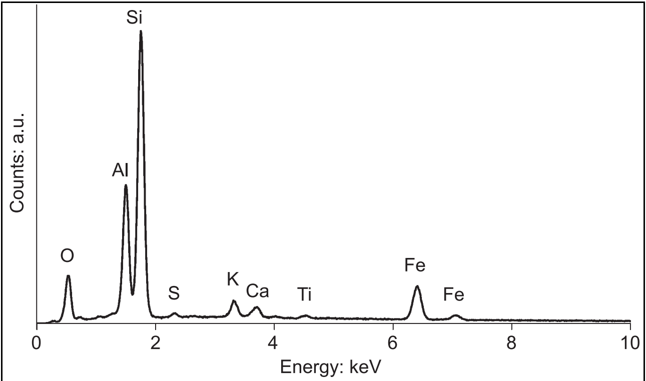

Figure 2. Energy dispersive x-ray analysis from the surface of a Croxden sand grain showing the elemental composition

2.2. Sample preparation procedure

The raw materials were stirred in a Hobart mixer for 10 min. The mould was coated with release oil prior to casting cylinders 18 mm in diameter and 36 mm in length. These samples were of suitable dimensions for creep and compressive testing. To reduce the presence of air pockets within the specimens and to ensure an even distribution of lime mix within the mould, each mould was half filled and vibrated for 1 min before filling to just below the top and continuing vibration for a further 1 min. A thin layer of high alumina cement was then applied to the top of each specimen and smoothed to ensure that the surface was flat and parallel with the bottom surface. The specimens were then left under the laboratory conditions of 25°C and 50% relative humidity to harden. To ease the extraction of the specimens, the mould had a removable bottom and was split along the specimen length. After 1 day, the bottom of the mould was removed. Samples were removed from the mould for insertion into the creep rig, under the conditions detailed in Table 2. An additional set of CL90/PC mixtures were tested after a period of between 9 and 14 days.

2.3. Compressive testing

The compressive strength of each mix was determined by testing between seven and 10 specimens using a Zwick/Roell testing machine with 10 kN load cell. Each specimen was mounted between two parallel stainless steel platens. The top platen was allowed to pivot on a ball joint to accommodate specimen surface irregularities. Loading was applied at a constant stress rate of 0·079 N mm^{-2} s^{-1} until the ultimate value was reached. The average compressive strength of samples from each batch studied was determined prior to inserting samples from the same batch into the creep rig. The average compressive strength obtained was used as a guide to allow samples to be loaded to a

Mix design	NHL3.5 (natural hydraulic lime)		CL90 (Calcium Lime)		PC (Portland Cement)		Croxden sand	
	Volume: cm^3	Weight: g	Volume: cm^3	Weight: g	Volume: cm^3	Weight: g	Volume: cm^3	Weight: g
1 : 2 NHL3.5	1	1	–	–	–	–	2	4·68
CL90	–	–	1	1	–	–	–	–
1 : 2 : 9 PC	–	–	2	1	1	1	9	1·4
1 : 1 : 6 PC	–	–	1	1	1	1·9	6	2·3
1 : 6 PC	–	–	–	–	1	1	6	1·4

Table 1. Specimen mix designs (by volume)

Mix design	Post mixing hardening period: days	Compressive failure stress		Young's modulus: MPa	Number of samples tested in creep rig
		Mean, *x*: MPa	Coefficient of variation: %		
1 : 2 NHL3.5	4	0·93	15	205	4 + 4 unloaded
Pure CL90	7	1·37	19	213	8
1 : 2 : 9 PC	7	1·87	22	217	8
1 : 1 : 6 PC	7	2·43	12	372	8
1 : 6 PC	7	2·81	15	545	8

Table 2. Details of NHL3.5, CL90 and PC samples used to calculate deformation rates

value as close to the compressive failure stress as possible. However, for practical reasons this was not always possible. Additional samples were also tested in compression at 33, 95, 185 and 365 days.

2.4. Creep rig

A creep rig containing eight individual locations for simultaneous sample monitoring was constructed as shown in Figure 3, where a labelled photograph of the end sample location on the creep rig is illustrated. Displacements were logged using a PC interfaced with Solartron Metrology linear displacement transducers with a resolution of 50nm via an Orbit measurement system interface. Readings were recorded at intervals of 10 s.

The pivot points within the creep rig were required to operate under a high load in conjunction with the exceptionally low rotational displacements of the arm from sample movement. To ensure that the force remained constant with displacement an Omegadyne Inc LCM703-250 load cell was placed in series with a small block of lead, approximately 10 × 10 × 10 mm, inserted into one of the sample locations. A PC with an interface and data-logging software monitored force and resulting displacement applied over a 10-day period. The force remained constant with changing displacement.

Following an initial post-mixing period of hardening, samples were inserted into the creep rig and a load substantially below the compressive failure stress applied. For the much weaker CL90 samples, however, only 47% of the compressive failure stress could be applied.

3. RESULTS

3.1. Long-term strength development

Figure 4 shows the strength development of 1 : 2 NHL3.5, 1 : 6 PC and hybrid mortar mixes up to 365 days.

3.2. Effect of relative humidity on deformation

The effect of relative humidity was monitored over a period of 10 days for a 90-day-old sample of NHL3.5 (Figure 5). Perturbations in relative humidity correlated with changes in the observed deformation, suggesting a relationship between these parameters. An average strain rate of $1{\cdot}5 \times 10^{-6}$ mm per 1% change in

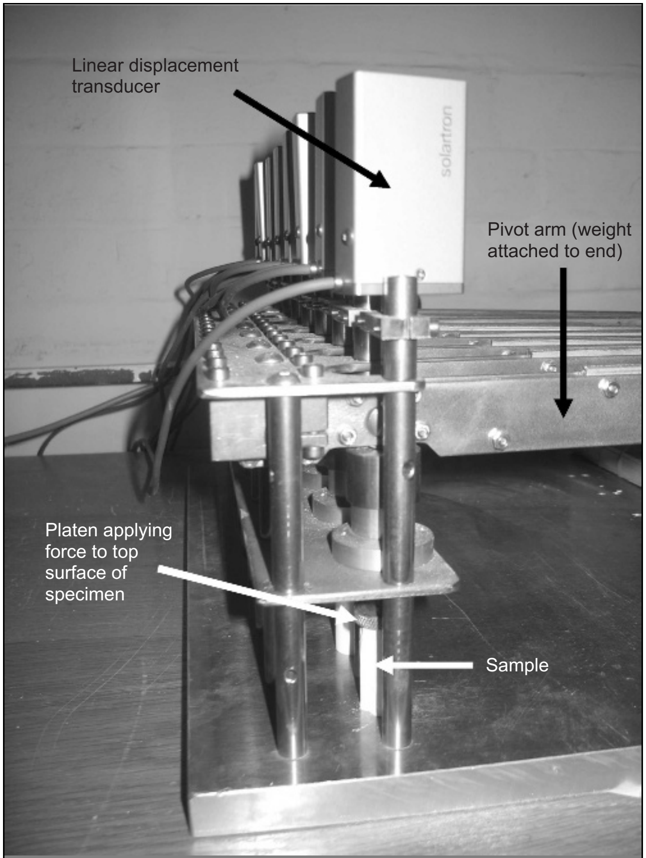

Figure 3. Photograph of rig used to monitor shrinkage and creep

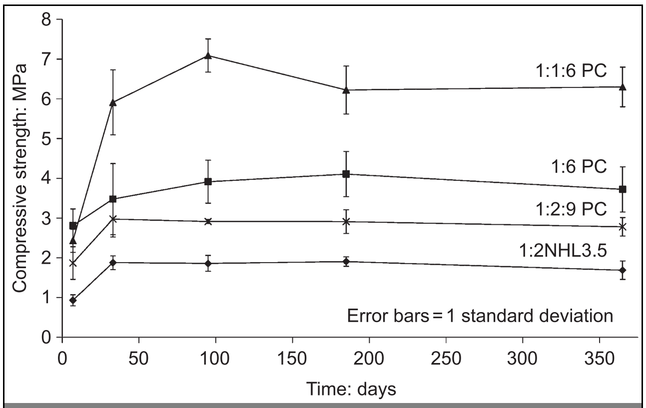

Figure 4. Compressive strength development of mortars over a 365-day period

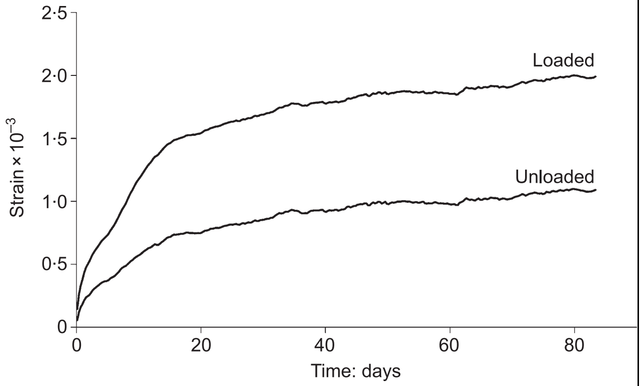

Figure 6. Deformation rates observed in specimens manufactured from 1 : 2 NHL3.5

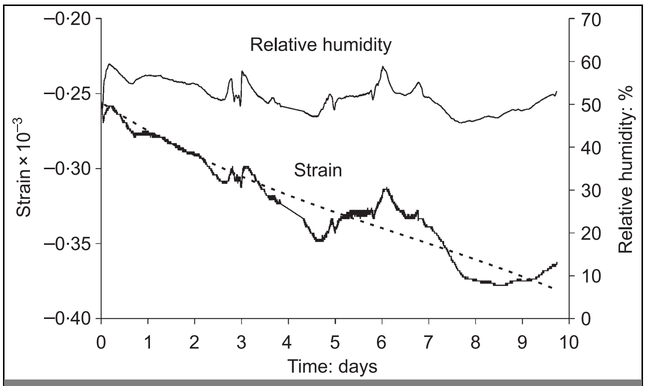

Figure 5. Deformation and relative humidity of a 90-day-old sample of NHL3.5 over a period of 10 days

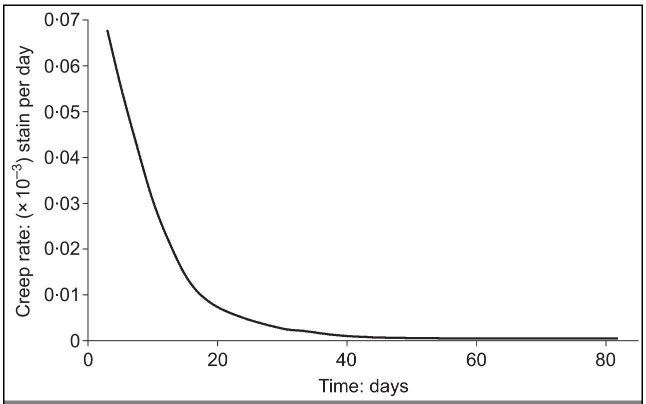

Figure 7. Creep rate observed in 1 : 2 NHL3.5 specimen

relative humidity at 23°C was found with a coefficient of variation of 12%.

3.3. Time-related deformation of NHL3.5

The deformation of 1 : 2 NHL3.5 to sand mortar was monitored over a period of 80 days. Following an initial 14-day period when there was a relatively large change, the deformation rate was observed to reach a steady value. An experiment was conducted comparing loaded and unloaded samples. These curves are shown in Figure 6, each being an average of four individual samples. Once again after approximately 2 weeks the deformation decreased and remained stable. The deformation behaviour of a specimen aged for over 90 days, subjected to a higher load, exhibited a strain rate that was comparable with the fresh samples after 2 weeks. Figure 7 shows a plot of creep rate against time calculated by subtracting the unloaded rate of deformation from that under loading.

3.4. Effect of calcium hydroxide content in cement hybrid mortars

The effect of the hydraulic component within the mortar was investigated by comparing CL90 and PC samples with hybrid mortars containing two combinations of CL90 and PC (Table 1). The difference in deformation rate before and after approximately 14 days followed the same trend as that of the NHL3.5 samples described previously.

A plot of mean strain rate against calcium hydroxide content by weight for each mix is shown in Figure 8. The calcium hydroxide concentration was determined from X-ray fluorescence (XRF) data using a test method equivalent to the wet chemical method described in BS EN 196-2.[16] This data was then used to calculate the percentage calcium hydroxide content in each of the mix designs tested. The samples tested after a period of between 9 and 14 days showed little increase in compressive strength but a markedly decreased strain rate.

4. DISCUSSION

The strength development of the mortar mixes up to 365 days are shown in Figure 4, indicating that after approximately 40 days no significant increase in compressive strength was observed. The data for the 1 : 2 NHL3.5 mortar over a period of 80 days and the CL90/PC mixes over 40 days show that the observed deformation curves for the mortars can be divided into a number of stages (Figure 9). During the first stage, a very high

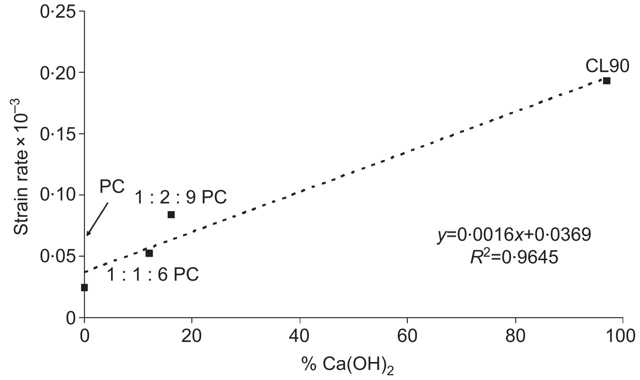

Figure 8. Relationship between strain rate and proportion of calcium hydroxide content by weight

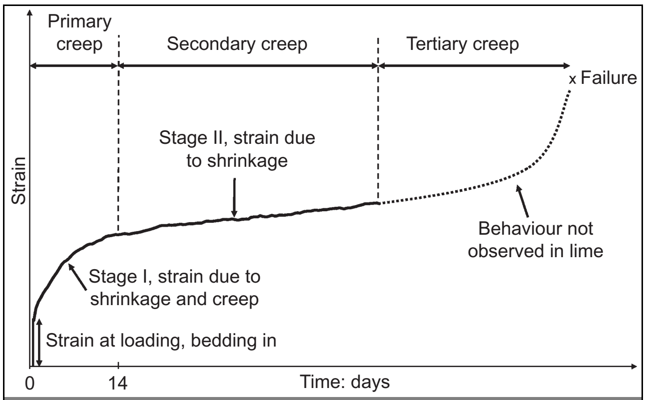

Figure 9. General form of strain–time curve for material subject to creep

rate of deformation occurred within 2 days following loading. This is believed to be associated with bedding-in of the sample as asperities on the sample surface were flattened by the smooth loading platens. Following the initial bedding-in period a deformation in the form of two essentially linear stages identified as primary and secondary creep was observed. In these experiments tertiary creep, a phenomenon used to describe the point to failure, was not observed and would have been unlikely to occur where there was no increase in stress. Moreover the observed deformation took the general form normally recorded for strain–time curves used to describe materials subject to creep.[17–19] It should be noted that the absence of tertiary creep does not imply that the samples cannot fail by brittle fracture.

A number of attempts have been made by previous researchers to assign a basic expression describing the deformation behaviour in concrete to predict strain without the need to carry out long-term tests.[17]

Two general treatments have been used to describe creep in composite materials containing a cement matrix and aggregate phase: those which assume that equilibrium will be reached at a limiting value, and those which assume that the process of deformation increases indefinitely. The most common expressions are those which assume a limiting value since for practical purposes, in ordinary structures, most of the creep occurs within 4 to 5 years.

Creep with a limiting value is described using expressions containing exponential and hyperbolic functions whereas those which increase indefinitely generally contain power and logarithmic functions.[17] All these expressions contain a varying number of constants or coefficients, some of which are empirical and others quasi-rational. The form of the deformation curves found from one mathematical treatment to the next highlights the variation in mechanical behaviour observed in cement structures suggesting that changes in mix parameters and environmental conditions have a marked affect on the overall behaviour.

Here we have investigated the application of these equations to the behaviour of lime mortars after periods of hardening exceeding 40 days. Attempts have been made to apply each of the four expressions described above to data sets (40 days and above) in this study. The highest correlation coefficients ($R^2 = 0{\cdot}94$ to 0·98) were obtained from the logarithmic Equation 1, where ε is creep strain, t is time and A and B are constants. The associated probability that these coefficients were obtained by chance is less than 0·01 indicating a very high level of significance. Values of A and B for logarithmic equations fitted to each of the mortar designs examined are given in Table 3.

$$\epsilon = A \ln t + B \qquad (1)$$

It is not possible, within the scope of this paper, to assign constants A and B to a specific mechanism or parameter within the material; however, the good fit may imply that deformation in lime mortars does not have a limiting value. Despite the relatively high correlation coefficients obtained for the logarithmic expression some deviation during the initial period of rapid deformation in the first 1 to 2 weeks occurred. This suggests that a more complex expression is required to describe the mechanism of early deformation.

For the analysis a straight line relation was adopted as an approximation for the creep rate in stages I and II. The mean, coefficient of variation and difference between the creep rate of the loaded and unloaded samples of NHL3.5 are shown in Table 4. The results indicated that in stage I the creep rate

	Coefficients from logarithmic equation	
Mix design	*A*	*B*
1 : 2 NHL3.5	0·39	0·34
1 : 2 NHL3.5 unloaded	0·22	0·11
Pure CL90	0·27	0·32
1 : 2 : 9 PC	0·23	0·25
1 : 1 : 6 PC	0·21	0·28
1 : 6 PC	0·14	0·11

Table 3. Coefficients *A* and *B* from exponential equations fitted to creep/shrinkage data

	Mean × 10^{-3}	Coefficient of variation	Difference (loaded − unloaded) × 10^{-3}
Stage I			
Loaded	80·6	12·9	42·7
Unloaded	37·9	8·8	
Stage II			
Loaded	6·0	12·2	1·43
Unloaded	4·6	14·7	

Table 4. Deformation rates during stages I and II for 1 : 2 NHL3.5 samples

was an order of magnitude higher than that observed in stage II.

Creep is defined as the time-dependent deformation of a specimen under load therefore the difference between the strain rates in the loaded and unloaded samples represents the creep rate. This is illustrated in Fig. 7. The creep rate was, however, observed to decrease rapidly with time during stage I, and was essentially zero during stage II suggesting that the deformation exhibited during the latter stage is due to a shrinkage process.

It is commonly accepted that hydraulic lime mortars harden by a combination of hydration and carbonation, the hydration being much faster than carbonation.[9] It is therefore assumed that during the initial few weeks the hydraulic component is an important factor in determining creep.

This was investigated using mix designs manufactured from combinations of CL90 and PC to have different hydraulic characteristics. This approach provided a convenient way to produce mixes with known hydraulic content which were also commonly used in the construction industry. The alternative approach of using a range of limes with different hydrolicity (2, 3·5, 5) was not undertaken considering that these classifications were defined by 28 day mortar strengths and encompassed very broad, overlapping, strength ranges. In Figure 8 the percentage of calcium hydroxide determined from XRF data for each specimen is plotted against the mean strain rate recorded during stage I. A gradient of 0·0016 was recorded with a correlation coefficient (R^2) of 0·96. The associated probability that these coefficients were obtained by chance is less than 0·05 indicating a high level of significance. The reduced recorded deformation rate observed in samples with high proportions of PC is a likely consequence of the formation of a rigid structure of calcium silicates which is less prone to creep than the calcium hydroxide and carbonate structures formed in CL90 lime. These results indicate therefore that higher creep rates can be associated with mortars containing higher proportions of calcium hydroxide.

Previous researchers have identified the influence of aggregate content and shape on compressive strength.[5,20] The different aggregate content present in these mixes is shown in Table 1. In the case of mortars that harden by carbonation there is a trade-off between the aggregate producing a more open pore structure which promotes carbonation and a reduction in strength associated with porosity. Stresses are also transmitted through the network of touching aggregate particles. In this study the CL90 paste did not contain any aggregate which may influence carbonation and thus the creep rate recorded.

The similar behaviour observed in the natural hydraulic and hybrid mortars instils confidence in drawing comparisons between mortars containing different hydraulic components.

In similar tests on samples with a post-mix hardening period of 9 and 14 days, a reduced stage I strain rate was observed suggesting that as samples are aged they become less susceptible to creep. This is consistent with greater hydration and carbonation and is in agreement with work of Troxell.[21]

5. CONCLUSIONS

(*a*) Time-dependent deformation was observed in NHL3.5 lime mortars. This was attributed to a creep mechanism which was load dependent and a shrinkage mechanism which was independent of load. The load-dependent creep component was most significant during the 14 days after insertion in the rig.

(*b*) A reversible relationship between sample dimension and relative humidity was observed in a 1 : 2 NHL 3.5 lime mortar.

(*c*) The high creep rate observed from NHL3.5 specimens during the first 2 weeks was not exhibited in samples over 90 days old.

(*d*) Samples manufactured from CL90 and PC exhibited a lower load-dependent stage I creep on ageing.

(*e*) Drying shrinkage rates observed after initial curing when hydration would be expected to dominate were similar for all samples tested.

(*f*) Higher creep rates were observed in specimens with high calcium hydroxide content. A linear relationship between the deformation (creep and shrinkage) rate and calcium hydroxide content was observed.

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