

Cement and Concrete Research 30 (2000) 1465-1475

# CEMENT<sub>AND</sub> CONCRETE RESEARCH

# Basic creep of hardened cement paste A re-examination of the role of water

Basile T. Tamtsia, James J. Beaudoin\*

Institute for Research in Construction, National Research Council of Canada, Building M-20, Montreal Road, Ottawa, Ontario, Canada K1A OR6

Received 21 December 1999; accepted 7 April 2000

#### **Abstract**

A re-examination of the role which water plays in the creep process of hardened cement paste (hcp) was made using a differential thermogravimetric technique. Prior to creep experiments in a controlled humidity chamber, very thin specimens of cement paste (<1 mm) were subjected to various drying regimes including exchange of water with organic solvents for 48 h followed by a gentle drying procedure. Cement paste specimens with or without evaporable water will exhibit significant creep suggesting a mechanism involving microsliding between C-S-H sheets. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Specific basic creep; Water; Solvent exchange; D-dry and compliance rates

### 1. Introduction

Much has been written in the last several decades on the mechanisms of shrinkage and creep of hardened cement paste (hcp). However, the mechanisms of the time-dependent chemical and physical processes involved have not yet been fully explained. A historical review of the proposed creep mechanisms indicates that despite the extensive experimental data collected over many years, no universal theory has been universally accepted [1–3].

A common view is that both creep and shrinkage should be considered together and as a manifestation of the same phenomenon [1,2,4]. Creep has been defined as the time-dependent deformation of a specimen under constant external load and shrinkage as the deformation of a specimen during drying without external loading. Both are reported as strains and depend on several factors including relative humidity, load, size of specimen, and temperature [5,6]. Several researchers have reported that partial or complete extraction of the water content of the cement paste specimens always produces a decrease in the creep strain [7–9]. The specimens from which all evaporable water has been removed have been stated to exhibit no creep or permanent set [10,11].

Powers [4] argues that creep is caused by diffusion of the load-bearing water; an external load changes the free energy of adsorbed water. Feldman [12] has suggested that creep occurs through the gradual crystallization or aging of layered silicate material leading to an increase of the extent of layering. Water movement was not considered a major mechanism. Wittmann [13] suggested that in both the Powers and Feldman models, creep should be zero if the hydrated cement paste is dried out fully. However, his own measurements are conflicting as they show significant creep even of the fully dried specimen. This is contrary to the results of Mullen and Dolch [26] who reported that oven-dry specimens of cement paste exhibited no creep at all. They concluded that water movement was the dominant mechanism responsible for the creep of concrete.

Significant structural changes occur in cement pastes during the first drying [14–16]. The exchange of water in paste specimens by organic fluids with low surface tension is believed to reduce the drying stresses causing microstructural change [14]. The extent of drying before application of load influences the magnitude of creep [5,16]. Hannant [17] found that further creep and swelling are not significant when dried specimens are re-saturated with organic fluids. Re-saturation with polar molecules like methanol did exhibit some small effect on creep.

In view of the varied opinion concerning the relevance of existing types of water found in hcp, the primary objective

<sup>\*</sup> Corresponding author. Tel.: +1-613-993-6749; fax: +1-613-954-5984. *E-mail address*: jim.beaudoin@nrc.ca (J.J. Beaudoin).

of this study is to re-examine the role which water plays during the creep process. Very thin specimens and a controlled humidity cell were used in this work to eliminate the effects of moisture gradients. Solvent exchange followed by a gentle drying procedure was one method chosen to minimize cracking and other microstructural changes usually caused by normal drying. The state of water in treated and untreated specimens was characterized using a differential thermogravimetric method. Various drying regimes were used (including drying to the vapor pressure of 'dry-ice' i.e. D-drying) prior to the creep tests.

#### 2. Theory

#### 2.1. Water in hardened cement paste

The state of water in hcp has been described by many authors. A detailed description by Ishai summarizes many of the reported characteristics. In addition to chemically bound water within the tobermorite-like crystallites, he has distinguished four types of evaporable water, which are defined according to how strongly the water is bonded to the solid phase. They are:

- 1. Pore water in the capillary and gel voids, at a distance of at least 1–2 nm from the solid surfaces and therefore outside the range of the van der Waals forces of adhesion.
- 2. Water adsorbed on to the crystal surface in layers 1−2 molecules deep.
- 3. Intercrystalline adsorbed water, i.e. water confined between mutually adjoining crystal surfaces in narrow spaces two molecules wide (about 0.8 nm). This type of water, being subjected to two sets of forces, is probably more strongly bound than types 1 and 2.
- 4. Intracrystalline zeolitic water, i.e. water about one molecule thick (about 0.4 nm) embedded between the tobermorite crystallite layers. Part of this water was found to be strongly bound to the solid (chemisorbed) and defined as hydroxylic water.

From infrared and nuclear magnetic resonance analysis, Sierra [18] concluded that after D-drying, only the hydroxylic water will subsist (see Fig. 1). Water occupies and can penetrate the whole size spectrum of pores within the hcp. It is known however that a substantial volume of porosity cannot be intruded by organic liquids [1]. Several authors acknowledge the special role water can play by virtue of its small molecular size and high dipole moment, and indicate that only water can occupy the smallest pore spaces where surface forces play a dominant role [1,17]. Ruetz [7] describes evaporable water in hcp as playing an important role in creep. His studies show that creep of specimens preconditioned at different moisture contents prior to basic creep tests decreases sharply as water is removed from the sample. The smallest pores are claimed to dominate the creep characteristics and therefore, pore size distributions become important.

#### 2.2. Creep of hardened cement paste

The hcp subjected to a sustained applied load or to an environment with a lower humidity than that existing internally in the specimen exhibits strain. The resulting time-dependent deformation is generally divided into two superimposed components: *creep* (sum of basic and drying creep), which is defined as deformation under load; and *shrinkage* defined as spontaneous deformation of a specimen during drying without external loading. The magnitude of creep is believe to depend to a large extent on the water content of concrete at the time of loading and on the drying and/or wetting process while under load. The basic creep occurs under conditions of no moisture movement to or from the surrounding medium.

Several workers have studied the influence of moisture content of hcp and/or concrete upon creep, and advanced various results and hypotheses to explain its mechanism. Although the motive force for creep is an externally applied pressure, as opposed to that for shrinkage, Feldman [12] and many other workers [2,4,7,16] suggested that shrinkage resulting from loss of moisture and creep are inter-related. There are many proposed mechanisms described to explain creep. Some of these are briefly summarized below.

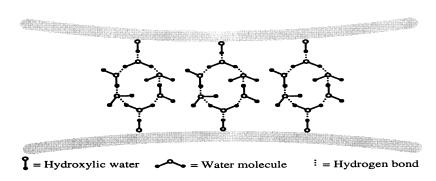


Fig. 1. C-S-H interlayer water structure after Sierra [18].

#### 2.2.1. The seepage theory

The seepage theory ascribes the volume changes due to the application of an external load to the change in the internal vapor pressure and hence, in the gel water content. It predicts water loss from the specimen during basic creep. Powers [4] describes a process where the disjoining pressure following the variations in moisture content of cement paste is exerted by the load-bearing water in areas of hindered adsorption. The external load squeezes some of this water out into areas of unhindered adsorption by a time-dependent diffusion process. Disjoining pressure gradually decreases and causes a decrease in the volume of the paste as the spacing of the particles in the gel is reduced. The removal of the load will cause a drop in the pressure in the load-bearing water. This theory predicts water loss from the specimen during basic creep. Young [1] found this in conflict with the fact that creep can occur in sealed or immersed specimens where water loss will be inhibited. It is also apparent that if creep occurs in the dry state, the seepage theory is untenable.

Powers attributes the irreversible creep (in part) to the formation of new bonds between surfaces when they are pressed together for the first time. Irreversibility results from the linear deformation of solid elements due to creep in compression during first loading. Therefore, re-application of load will not cause significant additional creep.

## 2.2.2. The interlayer theory

The 'interlayer' hypothesis proposed by Feldman and Sereda [19] is that, creep of cement paste is a manifestation of the gradual crystallization or aging of a poorly crystallized layered silicate material, accelerated by drying or stress; the adsorbed water does not play a significant role in the mechanism. Compression of C-S-H sheets under an applied or induced compressive stress and interactions between adjacent sheets cause the formation of new interlayer space. This takes place primarily at the entrances to existing interlayer regions where the layers are already in close proximity. The new interlayer spaces result from an irreversible process initiated by the elastic responses within the solid. On unloading, some of the water re-enters the interlayer space accounting for the reversible part of creep. Further, only the water in the limited regions of the interlayer entrances is affected by stress.

The gradual redistribution of water which results in a densification and ordering of C-S-H with a net increase in the layered volume is believed to be the cause of irreversible creep [12,19].

### 2.2.3. The viscous shear theory

A mechanism proposed by Ruetz [7] called the adsorption theory, suggests that creep occurs through slip between C-S-H particles in a shear process in which water acts as a lubricant. The sliding process takes place in the very thin, multi-molecular layers of adsorbed water. As the distance of any liquid molecule from the surface of a solid particle

increases, the degree of orientation of the liquid molecule will change, depending upon the interaction between the liquid molecule and the solid surface. Such an orientation of liquid molecules will be disturbed by increasing temperature until at sufficiently high temperatures the thermal inelasticity becomes so great that a viscous flow occurs. The adsorbed water molecules are oriented with respect to the surface and the effective rigidity of the film is thereby increased. Loss of water from the adsorbed films, on drying, will disturb the orientation of the films and hence, result in an increased drying creep.

Ishai [3] suggests that, whereas the diffusion of inter- and intracrystalline adsorbed water is the principal creep mechanism even in immersed bodies, this moisture movement is not connected with the drying shrinkage that occurs in the normal range of relative humidity. The creep process, which takes place in the inter- and intracrystalline spaces and the respective adsorbed water, is different both in type and origin. The applied load causes a decrease in those interparticle spaces within the range of the physical forces reducing the energy level of the system. The process is therefore irreversible.

#### 2.2.4. The thermal activation theory

According to Wittmann, diffusion of water does not play a major role in the creep mechanism. He does consider, however, that water plays an indirect role through its effect on disjoining pressure which, in turn, weakens interparticle bonds. After the strength of these forces is reduced, the particles slide apart with respect to each other and creep is therefore increased. The hypothesis is that time-dependent strains are the result of thermally activated processes that can be described by the rate process theory. Creep strains will originate through deformation of a microvolume of paste, designated as a 'creep center'. This area will undergo deformation to a lower energy state if external energy is applied to the material by means of either load or temperature.

The irreversibility of thermally activated creep is ascribed to the deformation of creep centers in their effort to achieve a lower energy state. Creep centers are viewed as areas of slip between adjacent particles of C-S-H.

### 2.2.5. Other theories

Various other mechanisms have been invoked to account for creep phenomena in cement paste. These include microcracking, which may even occur at high relative humidity as a result of shrinkage stress developed near non-shrinking CH crystals [3,20]. Recrystallization under load [12,21] (sometimes referred to as aging) reflected by the changes in surface area measurement at different degrees of reaction and different loading and drying conditions and slip between paste and aggregate [3] are other possible considerations.

More recently, another creep mechanism was proposed by Bazant et al. [22] referred to as the microprestress-

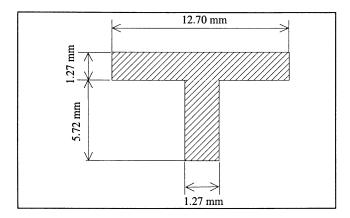


Fig. 2. Cross-section of 'T-shaped' specimen used for creep tests.

solidification theory which appears to be an extension by the authors of the solidification theory. The microprestress is generated by the disjoining pressure of the hindered adsorbed water in the micropores and by very large and highly localized volume changes caused by hydration or drying.

Bentur et al. [16] suggested that irreversible creep might be related to aging due to silicate polymerization of the C-S-H, induced by loading or drying.

## 3. Experimental program

# 3.1. Material characterization

Portland cement paste was prepared by vacuum-mixing with de-aired distilled water and type 10 Portland cement. The water-cement ratio was 0.50. The hcp was moist-cured for 30 years. Samples were sealed and continuously stored in a rubber membrane containing a small excess of saturated lime water at  $24 \pm 2$  °C. Relatively young cement paste was also produced to obtain early age properties. The determination of compressive strength on these was conducted using small cylindrical specimens, 1.27-cm diameter and 2.54-cm height. In this case, the cement paste was mixed using de-aired distilled water with a standard mixer for 3 min and vibrated for 1 min when casting. Once cast, specimens were stored in a moisture room at  $24 \pm 2$  °C. The samples were demoulded after 28 days and an average value of 34.5 MPa compressive strength with a standard deviation of 4.6% was obtained. The evaporable water content was determined using thermal analysis methods in a controlled environment. The thermal analysis equipment was housed in an environmental chamber.

Reagent grade anhydrous methanol and isopropanol were used in an attempt to remove water from the specimens with a minimum of disturbance to their microstructure.

## 3.2. Specimen preparation

Thin 'T-shaped' specimens, 25.4-mm long were made with hydrated type 10 Portland cement paste having a

water—cement ratio of 0.50. They were used for creep tests. The cross-sectional dimensions of these specimens are shown in Fig. 2. They were prepared from small 30-mm diameter and 25.4-mm long cylindrical samples cut with a diamond saw from cylinders 80-mm long. The pastes were made using vacuum-mixing and were cast in hollow cylinders. The samples were slowly rotated during hardening, to produce a homogeneous product. The samples were left in the mould for 2 days and then removed with a hydraulic press. After demoulding, samples were continuously exposed to lime-saturated water during storage for 30 years and during cutting to produce the thin 'T-shaped' specimens.

### 3.3. Specimen pre-treatment

Some of the thin water-saturated 'T-shaped' specimens used for this experiment were D-dried (drying to the vapor pressure of dry-ice at  $-78^{\circ}$ C) or dried at 42% relative humidity prior to re-saturation with synthetic pore solution. Some specimens were immersed in organic solvents for 48 h then dried under vacuum at 37°C for 24 h (Table 1). The equivalent to the D-dried state refers to drying under vacuum at 105°C for 3 h [19]. It has been argued that methanol and/or isopropanol exchange reduce stresses that cause microstructural changes in normal drying as surface tension forces are much smaller when the organic solvent is removed [14,27].

Table 1 Summary of the sample pre-treatment regimes

Initial state	Final test state		
Reference	Saturated (first state)		
D-dried	Re-saturated (second state)		
Dried at 42% RH	Re-saturated		
Saturated	D-dried		
Saturated	Vacuum-dried at 37°C for 24 h		
Methanol immersion	Vacuum-dried at 37°C for 24 h		
Isopropanol immersion	Vacuum-dried at 37°C for 24 h		

The creep deformation measurements began a few minutes after the load application. The initial elastic strain of the 'T-shaped' specimens was determined from load—strain plots where the load was increased incrementally to give a value of applied stress equal to 11.5 MPa. This corresponds to a stress/strength ratio of about 0.3 for the hcp.

During the creep tests on D-dried specimens, the creep cells were kept dry by placing a magnesium perclorate agent in the cells and the moist-air in the cells was removed by flushing with nitrogen (Fig. 3).

#### 3.4. Creep apparatus

The creep experiments involved mounting the 'T-shaped' specimens on a miniature fixed frame linking the specimens to the load cell and to the modified Tuckerman optical extensometers used for the length change measurements. The creep frames were subsequently placed in cells containing drying agent for all experiments conducted at the dry state. Length change was monitored using the modified Tuckerman optical extensometers with an accuracy of one micro-strain. A schematic diagram of the controlled environment for measuring creep and/or drying shrinkage of 'T-shaped' specimens is presented in Fig. 3.

#### 4. Results and discussion

The hcp 'T-shaped' specimens were pre-conditioned as described in Table 1 prior to loading. The stress/strength ratio was 0.3 corresponding to an applied stress of 11.5 MPa.

The general characteristics of a differential thermogravimetric analysis (TGA) curve (cement paste) obtained under equilibrium conditions are presented in Fig. 4. The first endothermal peak is attributed to bulk water (this includes capillary and micropore) and the second peak to interlayer water [28]. The results of TGA carried out on re-saturated specimens (in addition to reference specimens, saturated or equilibrated to 42% RH) after the treatment previously described (Table 1) are presented in Fig. 5 in terms of the derivative of the mass loss vs. time (expressed in %/min) plotted against temperature. The methanol exchange curve is similar to the saturated case (first state) in the temperature region above 75°C suggesting that after re-saturation, water enters both the pores and interlayer space. Capillary water appears to be the more predominant type of water with methanol treatment. The reference curve for paste dried at 42% RH has a more pronounced peak at 105°C suggesting that it contains a large amount of interlayer water. The saturated reference has both peaks. The other re-saturation curves all have (on close examination) evidence of more

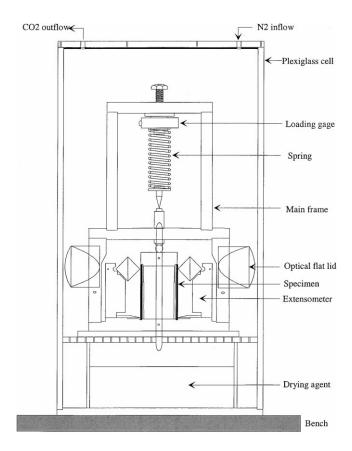


Fig. 3. Schematic diagram of the controlled environment for measuring deformation of miniature 'T-shaped' Portland cement paste specimens.

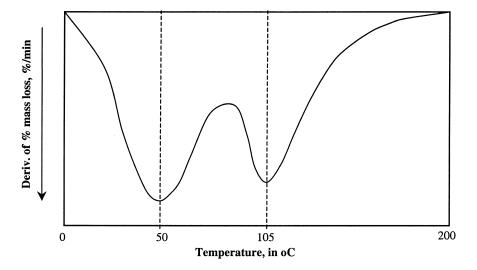


Fig. 4. Ideal curve of derivative mass change of saturated hydrated cement paste.

than one peak and exhibit a broad continuous response over the entire temperature range. The second peak is significantly broader and occurs at a much lower temperature suggesting that the structure has been altered and it is a much slower process to remove the interlayer water even though the removal starts at a lower temperature.

It is suggested that during the drying to 42% RH, part of interlayer water diffuses from small to coarser space within the specimen. This view is supported by the fact that the second peak of the saturated (first state) sample, which is at about 105°C, significantly decreases during the drying process and does not reappear after re-saturation with pore solution. This suggests that micropores cannot be

totally refilled after re-saturation under vacuum. D-drying, drying at 37°C under vacuum for 24 h or drying at 37°C after isopropanol exchange, all followed by re-saturation with pore solution, appear to affect considerably the microstructure of the specimen by increasing the temperature required for the removal of water in the micro-spaces. An exception appears to be drying at 37°C after methanol exchange followed by re-saturation with pore solution which results in a slight modification of the micropore system, as the corresponding DTGA curve around 105°C has a shape similar to that of the original specimen (saturated). The use of organic solvents to remove water from the specimen results in a slight reduction of the total

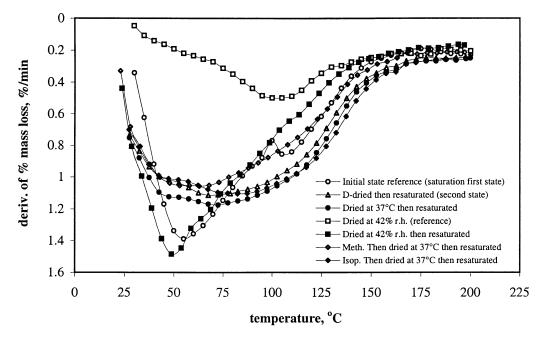
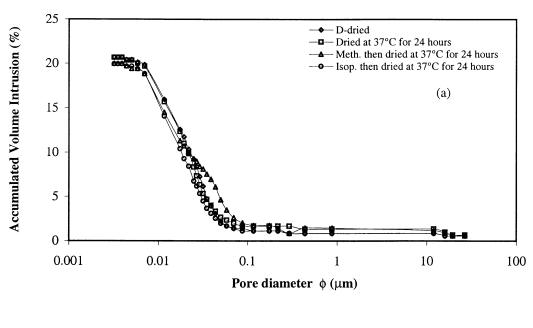


Fig. 5. Derivative mass change of hydrated cement paste (w/c = 0.5) after re-saturation with pore solution subsequent to different drying pre-treatments.



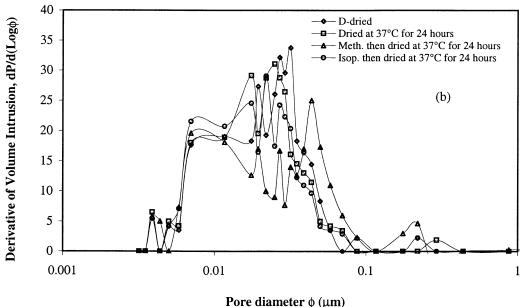


Fig. 6. Cumulative (a) and differential (b) pore size distribution of hydrated cement paste (w/c = 0.5) after different drying pre-treatments.

porosity as shown in Fig. 6. The methanol exchange prior to drying produces a larger percentage of micropores than does the isopropanol exchange.

The magnitude of the basic creep compliance of samples kept moist while under load (Fig. 7) is dependent on the drying history. Samples dried at 42% RH or D-dried and then re-saturated have significantly greater creep at 25 days than the saturated reference specimen. These compliance values are smaller than or even similar to that of D-dried samples (Fig. 8) for which the corresponding magnitude is significant and about  $180~\mu\text{e}/\text{MPa}$  at 30 days. These results confirm some previous findings relating the basic creep to the water content of hcp specimens

prior to loading [7–9]. However, contrary to the hypothesis that no creep results from fully dried hcp specimens [13], the D-dried sample exhibits a significant amount of basic creep. The amount of creep of dry specimens can be very large if the specimens are immersed in organic solvents prior to the drying process (Fig. 8). This suggests that the hydroxylic water molecules remaining on the crystallite after D-drying as well as their orientation may contribute to the creep. The creep taking place in hcp is believed to be mainly due to microsliding between adjacent sheets of C-S-H (Fig. 9) and/or due to a change in orientation of hydroxylic water strongly held on the crystallite surfaces of D-dried paste. The removal of water

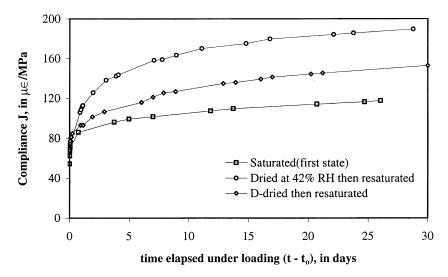


Fig. 7. Compliance of hydrated cement paste (w/c = 0.5) after re-saturation from different drying pre-treatments.

by a solvent prior to vacuum-drying may reduce the microstructural changes taking place in normal drying and therefore, increase the creep capacity of hcp as indicated by the results in Fig. 8 and the corresponding high surface area [24]. The available creep sites may then be greater in number resulting in an increase in the magnitude of basic creep.

The diffusion process may possibly be responsible for drying shrinkage or swelling of cement paste. The analogy suggesting a microdiffusion process in hcp under applied stresses as responsible for the early age creep is however questionable particularly in the case of basic creep. Such microdiffusion, if any, would support the seepage theory. This has been seriously questioned by Young because of the fact that creep can occur in sealed or immersed hcp speci-

mens where water will not diffuse out of the loaded sample (1). The water of type (3) and/or (4) described previously (Ishai) may facilitate sliding of C-S-H sheets one over the other by acting as lubricant [7,21]. The sliding taking place may be followed by the changing orientation of intra- and intercrystallite water as the surface of the C-S-H sheets approach each other resulting in further layering or aging as suggested by Feldman [12].

The results indicate that organic solvents used to remove water from the cement paste sample result in significant basic creep after vacuum-drying at 37°C. The methanol-exchanged specimens exhibit less specific basic creep than isopropanol-replaced specimens. This may be explained by the fact that after the water exchange process, some of the organic solvent remains physically

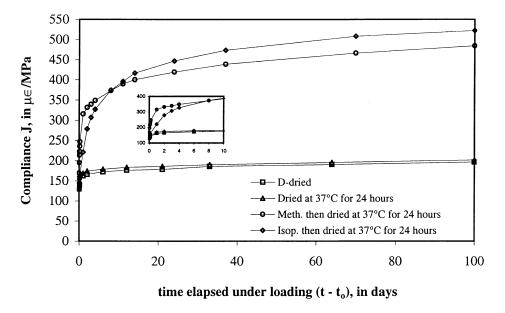


Fig. 8. Compliance of hydrated cement paste (w/c = 0.5) after different drying pre-treatments.

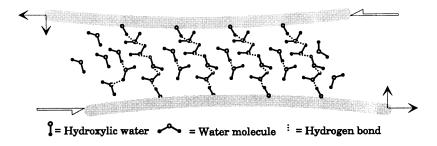


Fig. 9. Sliding between C-S-H sheets resulting in breaking and reconstitution of hydrogen bonds.

adsorbed on the surface of the C-S-H sheets and is not totally removed after 24 h of vacuum-drying at 37°C or by other procedures based on evacuation or heating at temperatures below those at which reactions yielding carbonate begin to occur [24]. The creep result can also be explained by the formation of a new complex from the reaction between the solvent and cement paste. Methanol has been shown to react with CH and C-S-H [14,23]. Such physically adsorbed films and/or complexes may weaken the C-S-H surface resulting in an increase in the sliding capacity between the sheets.

Plots of the compliance (total deformation per unit stress) vs. time (log-log scale) are linear. The parameters obtained from regression analysis depend on the water content of the sample and/or the pre-treatment history. The curves describing the compliance rate are power functions and can be expressed as follows:

$$\frac{\mathrm{d}J(t,t_{\mathrm{o}})}{\mathrm{d}t} = \alpha(t-t_{\mathrm{o}})^{\beta} \tag{1}$$

where,  $J(t,t_o)$  is the compliance of hcp at age t loaded at  $t_o$ ,  $\alpha$  and  $\beta$  are constants, and  $(t-t_o)$  is the time elapsed under loading. The constant  $\beta$  is dimensionless whereas the constant  $\alpha$  has units of  $\mu \in MPa/day^{1+\beta}$ .

It is apparent from Fig. 10 and the parameters  $\alpha$  and  $\beta$  obtained from regression analysis (see Table 2) that the rate of change of the total deformation per unit stress (compliance) obtained by experiment follows the trend expressed by Eq. (1). Curves similar to the one in Fig. 10 (for saturated hydrated cement paste) were obtained for all the pre-treatment regimes.

In each case, the power function parameters were determined with a correlation coefficient greater than 0.90. This has implications for models of long-term creep developed from early age data. The creep equation (2) can be obtained by simple integration of Eq. (1).

$$J(t, t_{o}) = \frac{\alpha}{1+\beta} (t - t_{o})^{1+\beta}.$$
 (2)

Experiments by Ulm et al. [25] on concrete showed the same trend of compliance rate with time except that in Eq. (1), the parameter  $\beta$  was chosen to be equal to -1 and the parameter  $\alpha$  was the only variable. The range of variation for the  $\beta$  parameter in this study is small, i.e. 0.71-0.94. The assumption that  $\alpha$  is the only variable seems reasonable. The variation in  $\alpha$  (Table 2) indicates that methanoland isopropanol-exchanged specimens behave quite differ-

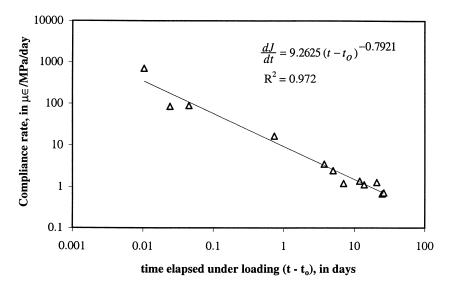


Fig. 10. Compliance rate of saturated hydrated cement paste (w/c = 0.5).

Table 2 Parameters  $\alpha$  and  $\beta$  obtained from regression analysis

Pre-treatment regimes	α	β	Correlation coefficient
Saturated (first state)	14.38	-0.86	0.98
Re-saturated (second state)	11.47	-0.72	0.97
Re-saturated from 42% RH	13.36	-0.71	0.92
D-dried	7.96	-0.89	0.94
Vacuum-dried at 37°C for 24 h	6.96	-0.86	0.93
Methanol vacuum-dried at 37°C	42.00	-0.94	0.98
Isopropanol vacuum-dried at 37°C	43.96	-0.80	0.95

ently from the others. Ulm's results showed that the specific creep rate was independent of loading time and degree of hydration. An important observation supporting this was the co-linearity of the log creep rate—log time curves. It was also demonstrated that the long-term aging effect was characterized by a decrease of the creep rate that was proportional to the inverse of the material age. This would seem to suggest that the rate-determining mechanism is associated with the behavior of C-S-H sheets themselves; perhaps involving a slipping and sliding process (see Fig. 9). Despite the similarity between the compliance rates of both cement paste and concrete samples, more experimental data are needed in order to consider modeling the long-term creep of concrete specimens from early creep data collected on hcp samples.

The creep rate is more pronounced for the methanol-treated specimens at early ages after loading. Isopropanol-treated specimens creep more than methanol-treated specimens but have a similar creep rate after a few weeks. Both these solvents appear to alter the structure of cement paste.

### 5. Conclusions

- 1. Hardened cement paste specimens that have been fully dried (if there is no moisture exchange with the surrounding medium) creep as much or more than saturated reference specimens.
- Cement paste dried to an intermediate humidity equal to 42% RH and re-saturated creeps significantly more than saturated cement paste that has not been dried. This is attributed to the pore coarsening effect due to drying and possible increase in creep sites due to increased layering of C-S-H.
- 3. The effect of D-drying and/or vacuum-drying at 37°C on basic creep is similar and significant.
- 4. Exchanging water-saturated paste samples with methanol and isopropanol for 48 h followed by vacuum-drying at 37°C will increase the basic creep of hardened cement paste relative to D-drying.
- 5. Re-saturation of D-dried cement paste specimens will increase the basic creep relative to the creep of initially water-saturated specimens.

- 6. Solvent exchange (methanol and isopropanol) for 48 h followed by vacuum-drying at 37°C has a smaller effect on the microstructure of cement paste than normal drying or vacuum-drying at 37°C for 24 h as indicated by their lower total porosity.
- 7. Specific basic creep can be expressed as a power function the parameters of which will depend on the water content of the samples and/or on the drying condition and pre-treatment history.
- 8. A re-examination of the role of water suggests that creep of hardened cement paste specimens is primarily due to microsliding between adjacent particles of C-S-H which additionally is affected by layering or aggregation of C-S-H sheets.

## Acknowledgments

The authors wish to acknowledge the Natural Sciences and Engineering Research Council of Canada (NSERC) for its support of this research. The authors also wish to express their gratitude to Messrs. Robert E. Myers and Gordon Chan for their technical help at National Research Council Institute for Research in Construction where the laboratory work was carried out. Thanks to Dr. Franz Josef Ulm, associate professor at MIT, for his helpful discussions.

#### References

- J.F. Young, assessment of the influence of microstructure on timedependent deformations of hardened cement paste, in Proceedings of First Austrialian Conference On Engineering Materials, Sidney, (1974) 3-28.
- [2] X. Yunping, H.M. Jennings, Relationships between microstructure and creep and shrinkage of cement paste, Materials Science of Concrete, Vol. III, The American Ceramic Society, Westerville, OH, 1992, pp. 37–63.
- [3] O. Ishai, The time-dependent deformational behaviour of cement paste, mortar and concrete, Proceedings of International Conference On the Structure of Concrete, Cement and Concrete Association, London, England, (1968) 345–364.
- [4] T.C. Powers, Mechanisms of shrinkage and reversible creep of hardened portland cement paste, Proceedings of International Conference On the Structure of Concrete, Cement and Concrete Association, London, England, (1968) 319–344.
- [5] A.M. Neville, W.H. Dilger, J.J. Brooks, Creep of Plain and Structural Concrete, Construction Press, London, 1983, p.361.
- [6] I. Soroka, Portland Cement Paste and Concrete, MacMillan, London, 1979, pp. 46–72 (Chap. 3).
- [7] W. Ruetz, A hypothesis for the creep of hardened cement paste and the influence of simultaneous shrinkage, Proceedings of International Conference On the Structure of Concrete, Cement and Concrete Association, London, England, (1968) 365–387.
- [8] A.M. Neville, The measurement of creep of mortar under fully controlled conditions, Mag Concr Res, 9 (25) (1957) 9–12.
- [9] F.H. Wittmann, Influence of moisture content on the creep of hardened cement, Reol Acta, 9 (2) (1970) 282–287.
- [10] N.H. Brown, B.B. Hope, The creep of hydrated cement paste, Ccm Concr Res, 6 (4) (1976) 475–486.
- [11] J. Glucklich, O. Ishai, Creep mechanism in cement mortar, J Am Concr Inst, 59 (7) (1962) 923–948.

- [12] R.F. Feldman, Mechanism of creep of hydrated portland cement paste, Cem Concr Res, 2 (5) (1972) 509-520.
- [13] F.H. Wittmann, Discussion of Some Factors Influencing Creep of Concrete, State Institute of Technical Research, Report Series III, Vol. 167, Finland.
- [14] R.L. Day, Reaction between methanol and Portland cement paste, Cem Concr Res, 11 (3) (1981) 341–359.
- [15] L.J. Parrott, J.F. Young, Shrinkage and swelling of two hydrated alite pastes, in: F.H. Wittmann (Ed.), Fundamental Research on Creep and Shrinkage of Concrete, Martinus Nijhoff Publishers, The Hague, 1982, pp. 35–48.
- [16] A. Bentur, R.L. Berger Jr., F.V. Lawrence, N.B. Milestone, S. Young, J.F. Young, Creep and drying shrinkage of calcium silicates pastes: III. A hypothesis of irreversible strains, Cem Concr Res, 9 (1) (1979) 83–96.
- [17] D.J. Hannant, An experimental investigation of the creep mechanism in concrete, Symposium on Creep of Concrete, Part 1 ACI Special Pub., Detroit, 1964, pp. 331–340.
- [18] R. Sierra, Répartition des différentes formes d'eau dans la structure des pâtes pures de C<sub>3</sub>S et de Ciment Portland, Proc. 7th Int. Congress on the Chemistry of Cement, Paris, France, Vol. 3, (1980) 201–206.
- [19] R.F. Feldman, P.J. Sereda, A model for hydrated portland cement paste as deduced from sorption-length change and mechanical properties, Mater Struct, 1 (6) (1968) 509-520.

- [20] F.O. Slate, B.L. Meyers, Some physical processes involved in creep of concrete, Proc. Civil Eng. Mat. Conf., Part 1 Wiley-Interscience, London, 1971, pp. 769–773.
- [21] I. Ali, C. Kesler, Mechanism of creep in concrete, Symposium on Creep of Concrete, Detroit, ACI Publication SP-9, Vol. 1, (1964) 35–57.
- [22] Z.P. Bazant, A.B. Hauggard, S. Baweja, F.J. Ulm, Microprestress—solidification theory for concrete creep: I. Aging and drying effects, J Eng Mech, 123 (11) (1997) 1188–1194.
- [23] J.J. Beaudoin, Validity of using methanol for studying the microstructure of cement paste, Mater Struct, 20 (115) (1987) 27–31.
- [24] H.F.W. Taylor, A.B. Turner, Reactions of tricalcium silicate paste with organic liquids, Cem Concr Res, 17 (4) (1987) 613–623.
- [25] F.J. Ulm, F. Le Maou, C. Boulay, Creep and shrinkage coupling: new review of some evidence, Rev Fr Génie Civ, 3 (3-4) (1999) 21-37.
- [26] W.G. Mullen, W.L. Dolch, Creep of Portland Cement Paste, Proceeding of the American Society of Testing Materials, Vol. 64, (1964) 1146–1171.
- [27] R.F. Feldman, A Hypothesis for the Creep of Hardened Cement Paste and the Influence of Simultaneous Shrinkage, Proceeding 5th International Symposium Of Chemistry of Cement, Part III Tokyo, Vol. III, (1968) 53–66.
- [28] R.F. Feldman, V.S. Ramachandran, Differentiation of interlayer and adsorbed water in hydrated Portland cement by thermal analysis, Cem Concr Res, 1 (6) (1971) 607–620.