Materials and Structures/Matériaux et Constructions, Vol. **35**, April 2002, pp 134-140

SCIENTIFIC REPORTS

Drying, moisture distribution, and shrinkage of cement-based materials

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Paper received: January 2, 2001; Paper accepted: August 9, 2001

ABSTRACT

The aim of this study was to develop a new method to determine hygral diffusion, film, and shrinkage coefficients of cement-based materials. These coefficients are required for the numerical simulation of shrinkage strain and total deformation of concrete elements and structures using the finite element method. Both an experimental approach to determine the time-dependent relative humidity in the pore system of concrete and a numerical method to determine material coefficients on the basis of experimental data are described in this paper. The hygral diffusion coefficient can be expressed as function of moisture content and as function of relative humidity.

An experiment is carried out with sliced specimens measuring 150 x 100 x 3 mm. Each specimen is prepared by piling up 11 slices and sealing the outer surfaces with aluminum sheet. The distribution of relative humidity is estimated by measuring the shrinkage strain on each slice at arbitrary drying times. An inverse analysis is then used to obtain the diffusion coefficient from the measured relative humidity distribution. A numerical approach based on the weighted residual method and on a nonlinear least squares method is proposed on the basis of the experimental results.

RÉSUMÉ

L'objectif de cette étude était de développer une nouvelle méthode visant à déterminer les coefficients de diffusion hygrométrique, de pellicule et de retrait de matériaux à base de ciment. Ces coefficients sont nécessaires dans la simulation numérique de la force de retrait et de la déformation totale des éléments bétonnés et des ouvrages en utilisant la méthode de l'élément fini. Aussi bien l'approche expérimentale de détermination de la distribution de l'humidité relative dépendante du temps dans le système des pores du béton, que l'approche numérique de détermination des coefficients de matériaux sur la base des données expérimentales seront décrites dans cet article. Le coefficient de diffusion hygrométrique peut être exprimé à la fois comme fonction de la teneur en humidité et comme fonction de l'humidité relative.

Une expérience a été conduite à l'aide d'échantillons en tranches fines mesurant 150 x 100 x 3 mm. Chaque échantillon a été préparé en empilant 11 tranches, scellées sur les bords par une feuille d'aluminium. La distribution de l'humidité relative a été calculée en mesurant la force de retrait pour chaque tranche à des temps de séchage arbitraires. Une contre-analyse a été ensuite utilisée pour obtenir les coefficients de diffusion à partir d'une mesure de la distribution de l'humidité relative. Une approche numérique fondée sur une méthode de pesée résiduelle et sur la méthode des moindres carrés non linéaires a été ensuite proposée sur la base des résultats expérimentaux.

1. INTRODUCTION

Shrinkage strain develops in cement-based materials such as concrete, as moisture is lost to the environment or by self-desiccation. In case of drying shrinkage, it develops near the drying surface much quicker than in the center of a concrete element. If a concrete member were to consist of separate unrestrained elements of infinitesimal thickness, the relationship between a change in moisture content and deformation would be approximately linear [1]. In a real concrete member, however, the strain due to quicker drying near the surface as compared to the inside of the concrete produces tensile and compressive strains due to eigenstresses. In order to simulate numerically the effect of drying on the deformation of concrete, the diffusion, film, and shrinkage coefficients are required. By using the diffusion and film coefficients together, the moisture distribution in a drying sample can be obtained at any arbitrary time during drying. The unrestrained infinitesimal drying shrinkage strain is calculated by multiplying the change in moisture content by the shrinkage coefficient. Finally,

Editorial Note
Prof. Folker H. Wittmann is a RILEM Senior Member.

the total resulting deformation can be calculated according to the principle of virtual work.

To obtain the nonlinear diffusion coefficient, various methods have been proposed by different researchers [2-5]. To determine the diffusion coefficient as function of the pore equilibrium relative humidity of concrete, the relationship between moisture content at hygral equilibrium and the relative humidity, *i.e.* the sorption isotherm (which varies with ambient temperature) must be known. Experimentally derived desorption isotherms are available for some cement-based materials. However, it is not clear whether they can be generally applied. Furthermore, it takes a lot of time to determine a desorption isotherm experimentally. A method of obtaining the diffusion coefficient as a function of relative humidity directly by experiment is therefore desirable. A useful and reliable method of obtaining a diffusion coefficient as a function of relative humidity is proposed in this paper.

2. OUTLINE OF THE EXPERIMENTS

2.1 Mix proportions of mortar and concrete

In this research, normal Portland cement type-I (density: 3.12 g/cm^3) was used. The fine aggregate was river sand (density: 2.62 g/cm^3; water absorption: 1.93%). The coarse aggregate was river gravel (maximum size: 16 mm; density: 2.70 g/cm^3; water absorption: 0.62%). Mix proportions of mortar and concrete are shown in Table 1. The compressive strength of mortar and concrete at 14 days are 33.6 MPa and 36.4 MPa, respectively.

2.2 Geometry of specimens

A pile of sliced specimens, which consists of 11 slices with the following dimension 150 x 100 x 3 mm, was used for the determination of moisture distributions. For comparison, solid specimens of 150 x 100 x 33 mm were also used in this series of experiments. All surfaces, except the two parallel drying surfaces 150 x 100 mm, were sealed with aluminum sheet. Two solid specimens and nine sliced specimens were put into the climate room with relative humidity of 45% at 20°C. Drying began at an age of 14 days. At 0.5, 3, 7, 14, 28, 42, 56, 70, and 98 days after the start of drying, the aluminum sheet was removed from one sliced specimen in order to obtain the moisture loss of each slice. After the moisture loss was measured, the specimen was discarded.

Fig. 1 shows a sliced specimen used for the investigation of shrinkage strains. A pile of sliced specimens consists of 11 slices (150 x 100 x 3 mm).The gauge points are located on the top and bottom surfaces along two circular lines. Row B (see Fig. 1) is on an arc centered with respect to the gauge point in the 6th (middle) layer in row A. The gauge points in row A are similarly centered with respect to the 6th slice of row B. The gauge points are made of 3 x 3 x 10 mm brass and they have a 2 mm hole in their center. Four surfaces of specimen were sealed with aluminum sheet. The shrinkage strains were measured in the climate controlled rooms with relative humidity of 45%, 60%, and 75%, respectively. Drying began after the specimens were cured in water for 7 days and subsequently in a chamber at 100% relative humidity for 7 days. Length change in the longitudinal direction was measured using a linear gauge with a minimum division of 1/1,000 mm.

Table 1 – Mix proportions of mortar and concrete

Mixture	ϕ_{max} (mm)	W/C (%)	s/a (%)	Weight per unit volume (kg/m^3) W	C	S	G 4~8	G 8~16
Mortar	4	50	100	240	480	1536	–	–
Concrete	16	50	50	200	400	854	352	528

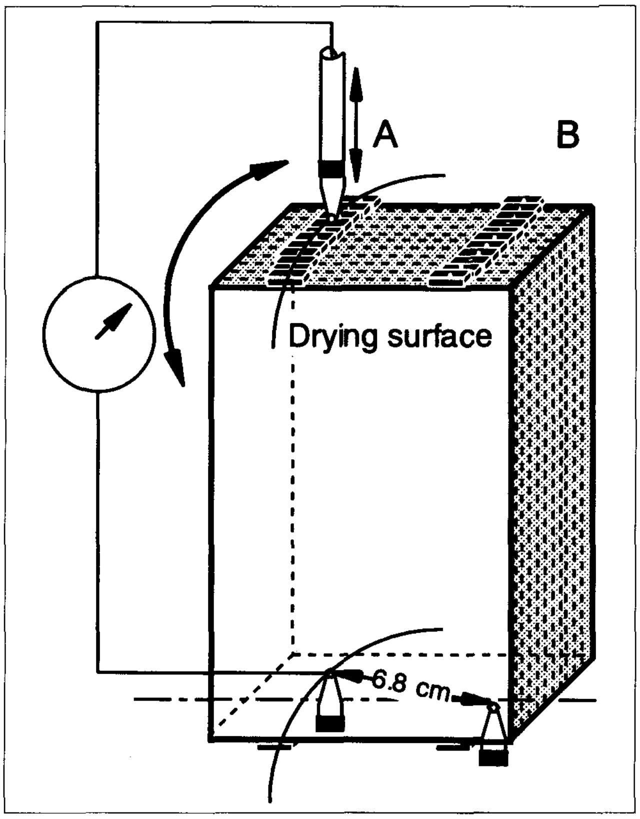

Fig. 1 – Sliced specimen for measuring shrinkage strains.

3. MOISTURE DISTRIBUTION

3.1 Effect of gaps in sliced specimens on moisture transfer

The diffusion coefficient of vapor transfer in air is approximately 218 mm^2/day at 20°C. This is about 50 or 100 times faster than in mortar or concrete [6]. This means that the transfer of vapor through the air is much easier than through mortar or concrete. However, the air layer between slices may act as an obstacle to moisture

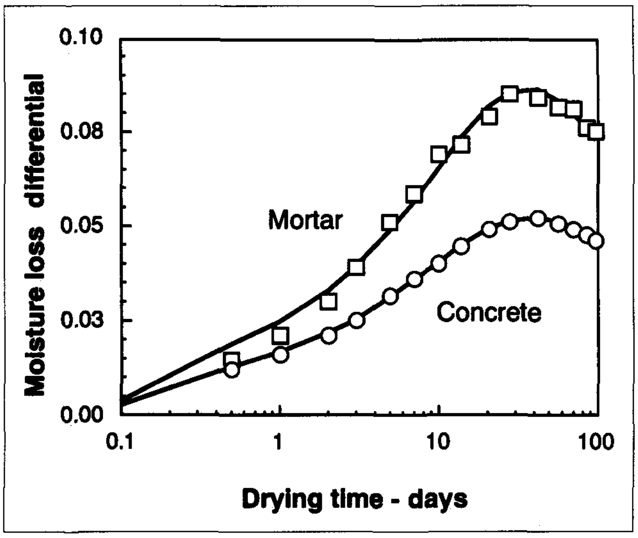

Fig. 2 – Moisture loss difference between solid and sliced specimen.

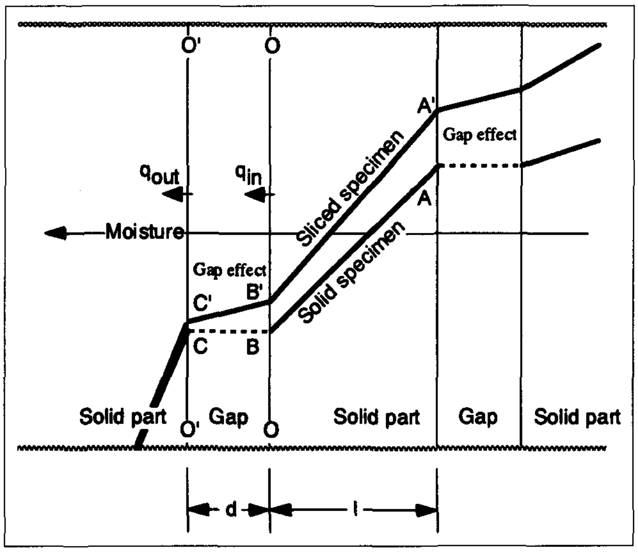

Fig. 3 – Effect of gaps on moisture transfer.

transfer in the sliced specimens in the high humidity region if the gap is considerable. In this study, the moisture distribution obtained from a pile of sliced specimens is used as a substitute for that of the solid specimen in order to obtain the diffusion coefficients. Before this substitution is applied, it has to be confirmed that the effect of gaps on the moisture flow in a pile of sliced specimen is small or negligible.

Fig. 2 shows the difference in moisture loss between the sliced and solid specimens. The difference in moisture loss is related to the saturated moisture content. The moisture loss difference finally disappears. The results clearly show the solid specimens loose moisture faster than the sliced specimens. This is due to the moisture transport disturbed by air gaps between slices. This can be explained as shown in Fig. 3. The lines A'B'C' and ABC express the assumed moisture distribution in sliced and solid specimens, respectively. When q_{in} and q_{out} are the moisture flows per unit time from surface O-O into an air gap and from an air gap into surface O'-O', respectively, q_{out} minus q_{in} is equal to the moisture change in the gap formed by OO and O'O'.

$$q_{out} - q_{in} = \frac{1}{2}\frac{\partial(\omega_{B'} + \omega_{C'})}{\partial t} \times d \tag{1}$$

where, ω_B' and ω_C' are moisture contents at the position B' and C', respectively; and d is the thickness of the gap between two slices. If the gap has zero thickness, q_{out} must be equal to q_{in}; that means, the moisture contents at surfaces O-O and O'-O' are the same. Whereas, when d cannot be ignored, the slope of moisture increases in the gap. With the slope of moisture in the gap and the diffusion coefficient D_{air} of air, the moisture flow q_{out} can be expressed by Equation (2).

$$q_{out} = D_{air}\frac{\omega_{B'} - \omega_{C'}}{d} = q_{in} + \frac{1}{2}\frac{\partial(\omega_{B'} + \omega_{C'})}{\partial t} \times d \tag{2}$$

Since q_{out} is bigger than q_{in}, ω_B' must be bigger than ω_C'. The wider the gap d, the bigger the difference between ω_B' and ω_C'. Hence, the sliced specimen loses its moisture slower than the solid specimen (q. e. d.).

Fig. 4 shows the moisture distribution of sliced and solid specimens. Open circles and squares are the experimental moisture loss distributions obtained experimentally from sliced specimens. The solid circles and squares are the estimated moisture distributions of solid specimens using ΔC, which is the moisture loss difference between a solid specimen and a pile of sliced specimens shown in Fig. 2. In order to obtain the moisture loss distribution of solid specimen, the following hypothesis is used: the gradients of moisture distribution at the drying surfaces of sliced and solid specimens are considered to be equal.

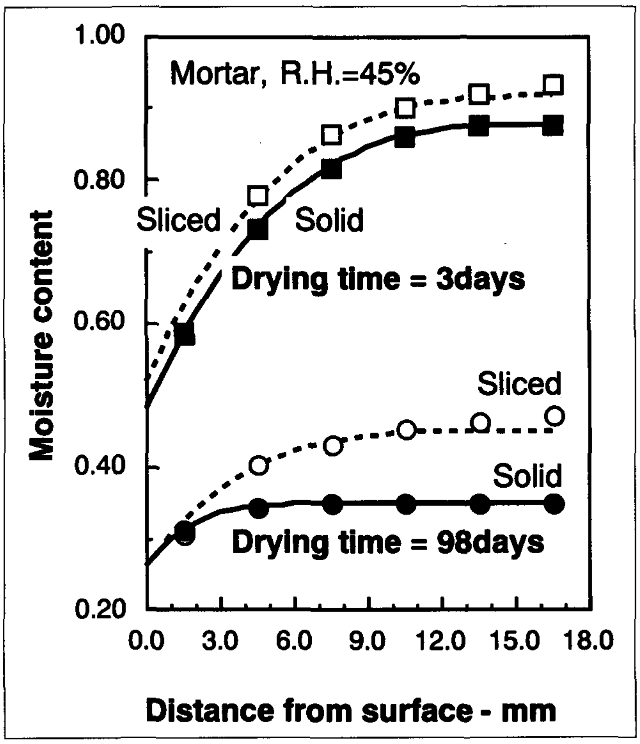

Fig. 4 – Moisture distribution in sliced and solid specimens.

Under this hypothesis, the difference in moisture content at the center of each specimen becomes maximum. By comparison of two diffusion coefficients obtained from the moisture distributions of solid and a pile of sliced specimen, the effect of gap on the moisture transport will be confirmed to be small in the next section.

3.2 Diffusion coefficient as function of moisture content

Equation (3) is the diffusion equation in one dimension.

$$\frac{\partial\omega(x,t)}{\partial t} = \frac{\partial}{\partial x}\left(D(\omega)\times\frac{\partial\omega(x,t)}{\partial x}\right) \tag{3}$$

where, $\omega(x,t)$ is the moisture content; $D(\omega)$ is the diffusion coefficient which is a function of moisture content $\omega(x,t)$; and x and t are distance in the direction of specimen thickness and drying time, respectively. Equation (3) must be satisfied everywhere in the specimen. Equation (4) holds with respect to an arbitrary function $F(x,t)$.

$$\int_{x_{surface}}^{x_{center}} F(x,t)\left\{\frac{\partial}{\partial x}\left(D(\omega)\times\frac{\partial\omega(x,t)}{\partial x}\right) - \frac{\partial\omega(x,t)}{\partial t}\right\}dx = 0 \tag{4}$$

By means of partial integration, Equation (5) is derived:

$$\begin{aligned}&\int_{x_{surface}}^{x_{center}} D\times\frac{\partial F}{\partial x}\times\frac{\partial\omega}{\partial x}dx \\ &= F(x_{surface},t)\times q_t - \int_{x_{surface}}^{x_{center}} F\times\frac{\partial\omega}{\partial t}dx\end{aligned} \tag{5}$$

where, q_t is the moisture content which passes through a unit drying surface area per unit time. Now, $\omega(x,t)$ is chosen for the arbitrary function $F(x,t)$. Equation (5) is rewritten as Equation (6):

$$\begin{aligned}&\int_{x_{surface}}^{x_{center}} D\times\left(\frac{\partial\omega}{\partial x}\right)^2 dx \\ &= \omega(x_{surface},t)\times q_t - \int_{x_{surface}}^{x_{center}} \omega\times\frac{\partial\omega}{\partial t}dx\end{aligned} \tag{6}$$

Furthermore, when $\omega(x,t)$ is expressed by the moisture content of each layer $\omega_1(t)$, $\omega_2(t)$, ..., $\omega_i(t)$, ... and $\omega_6(t)$, Equation (6) can be rewritten as Equation (7):

$$\begin{aligned}&\sum_{i=1}^{6} D(\omega_i)\times\left(\frac{d\omega_i}{dx}\right)^2\times l_i \\ &= \omega(x_{surface},t)\sum_{i=1}^{6}\frac{d\omega_i}{dt}\times l_i - \sum_{i=1}^{6}\omega_i\times\frac{d\omega_i}{dt}\times l_i\end{aligned} \tag{7}$$

where, $d\omega_i/dx$ is the gradient of moisture distribution at the center of each layer. l_i is the thickness of each layer. If the diffusion coefficient is expressed by an exponential equation, that is,

$$D(\omega) = a\times e^{b(1-\omega)} \tag{8}$$

then, Equation (7) can be rewritten as Equation (9):

$$\begin{aligned}&a\sum_{t=0}^{t=98}\sum_{i=1}^{6} e^{b(1-\omega_i)}\times\left(\frac{d\omega_i}{dx}\right)^2\times l_i \\ &= \sum_{t=0}^{t=98}\left(\omega(x_{surface},t)\sum_{i=1}^{6}\frac{d\omega_i}{dt}\times l_i - \sum_{i=1}^{6}\omega_i\times\frac{d\omega_i}{dt}\times l_i\right)\end{aligned} \tag{9}$$

Equation (9) expresses that Equation (7) must be satisfied at any drying time. Now, the moisture distribution has been determined from the experimental data. By using a least square method, constants a and b can be determined. The modified Marquart method is adopted in this study. The diffusion coefficients D_{sliced} and D_{solid} of concrete obtained by using the experimental results for sliced specimens and using the assumed moisture distributions of solid specimens are as follows:

$$D_{sliced} = 6.47e^{-3.23(1-\omega)} \tag{10}$$

$$D_{solid} = 9.15e^{-3.35(1-\omega)} \tag{11}$$

The smaller the diffusion coefficient, the slower the moisture loss. It is evident from a comparison of Equation (10) and Equation (11) that the diffusion coefficient of sliced specimens is smaller than that of solid specimens. The diffusion coefficient of sliced specimens as described by Equation (10) is an effective coefficient, which takes the influence of gaps into consideration. It is obvious that the proposed numerical method precisely reflects the fact that moisture transfer in a pile of sliced specimen is slower than in a solid specimen.

Fig. 5 shows the average moisture loss of three cylindrical specimens with different diameters for concrete. The empty circles, lozenges, and squares are the experimental results obtained from cylinders with diameters 50, 80, and 150 mm, respectively. The height of these cylinders is 100, 100, and 150 mm, respectively. These results were obtained in an environmental 45% relative humidity. The top and the bottom surfaces of the cylin-

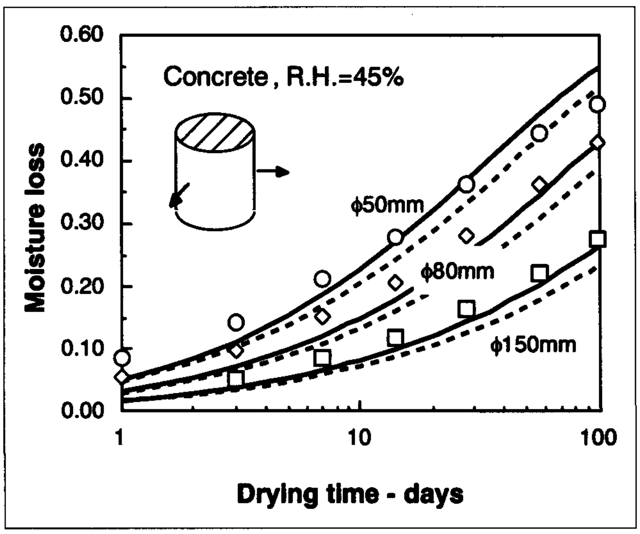

Fig. 5 – Moisture loss of cylinders with different diameter; Solid line: Equation (11), dashed line: Equation (10).

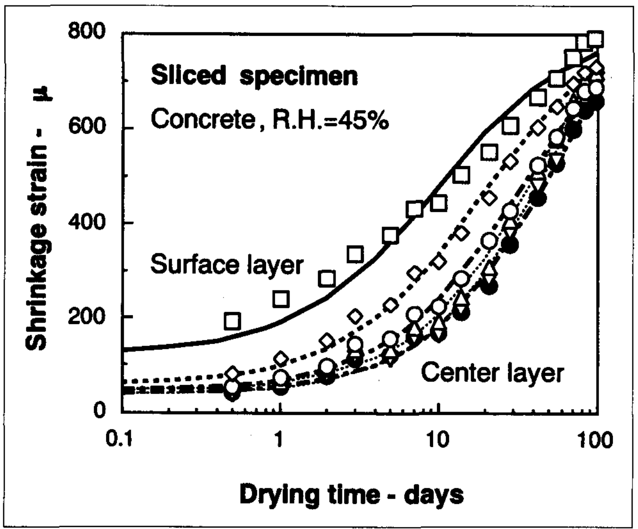

Fig. 6 – Shrinkage strain measured at each layer of a sliced specimen.

ders were sealed with resin in order to prevent moisture transfer. The curves were calculated by the finite element method. The dashed line was calculated with the diffusion coefficient D_{sliced} as expressed by Equation (10). The solid line was calculated with the diffusion coefficient D_{solid} as expressed by Equation (11). It is clear from this figure that the difference between the calculated values with D_{sliced} and D_{solid} is very small and that the calculated curves fit the experimental data reasonably well. It follows that the effect of gaps between slices on the moisture transfer can be considered to be small.

4. SHRINKAGE STRAIN AND RELATIVE HUMIDITY DISTRIBUTION

4.1 Relative humidity in mortar and concrete

Fig. 6 shows the measured shrinkage strains of each layer of a pile of sliced specimens. Squares, lozenges, circles, triangles, inverted triangles, and solid circles are the data obtained from the drying surface layer, the second layer, and so forth to the center layer. The curves shown in this figure were obtained by regression analysis using Equation (12):

$$Shrinkage\ \ strain = c_0 + \frac{c_2 \times t}{c_1 + t} \tag{12}$$

where, t stands for the drying times, and c_0, c_1 and c_2 are unknown parameters to be determined by the least square method. As the shrinkage strain of very thin specimens develops approximately linearly with changes in relative humidity, the relative humidity of the sliced specimens can be expressed using c_1 or c_0 and c_2, as shown in Equation (13):

$$\begin{aligned} Relative\ \ humidity\ \ of\ \ specimens &= 1 - \frac{(100 - R.H.) \times t}{c_1 + t} \\ &= 1 - (100 - R.H.) \times \frac{shrinkage\ \ strain - c_0}{c_2} \end{aligned} \tag{13}$$

where $R.H.$ is the relative humidity in the surrounding atmosphere. Equation (13) indicates that the relative humidity of each thin slice of a specimen is 100% when drying time is equal to zero and that the relative humidity in all specimens is equivalent to that of the surrounding atmosphere when the shrinkage strain reaches its ultimate value; that is, c_0 plus c_2.

4.2 Diffusion coefficient as a function of relative humidity

Substituting the moisture content term ω_i in Equation (9) by the relative humidity h_i term for cement-based materials, Equation (9) may be rewritten as follows:

$$\begin{aligned} & a \sum_{t=0}^{t=98} \sum_{i=1}^{6} e^{b(1-h_i)} \times \left(\frac{dh_i}{dx}\right)^2 \times l_i \\ & = \sum_{t=0}^{t=98} \left(h(x_{surface}, t) \sum_{i=1}^{6} \frac{dh_i}{dt} \times l_i - \sum_{i=1}^{6} h_i \times \frac{dh_i}{dt} \times l_i \right) \end{aligned} \tag{14}$$

The diffusion coefficients of mortar and concrete have been determined using Equation (14). Results are given in Equations (15) and (16):

$$\text{Mortar:} \quad D(h) = 26.3e^{-7.21(1-h)} \tag{15}$$

$$\text{Concrete:} \quad D(h) = 22.0e^{-8.26(1-h)} \tag{16}$$

4.3 Film coefficient

The total relative humidity Q is defined by Equation (17):

$$Q = \sum_{i=1}^{11} (1 - h_i) \times l_i \tag{17}$$

The film coefficient H_F expresses the relationship between q_t and ($h_{surface}$-h_a) where q_t is the time differentiation of $Q/2$; $h_{surface}$ is the relative humidity on the drying surface; and h_a is the relative humidity of the surrounding atmosphere.

$$H_F = \frac{q_t}{h_{surface} - h_a} \tag{18}$$

$$q_t = \frac{1}{2} \times \frac{dQ}{dt} \tag{19}$$

Fig. 7 shows the relationship between q_t and ($h_{surface}$-h_a) in the case of mortar. As is evident from this figure, the effect of the relative humidity of the surrounding atmosphere on the film coefficient is very small. For mortar and concrete used in this study, the film coefficients H_F are 0.773 and 0.541 mm/day, respectively.

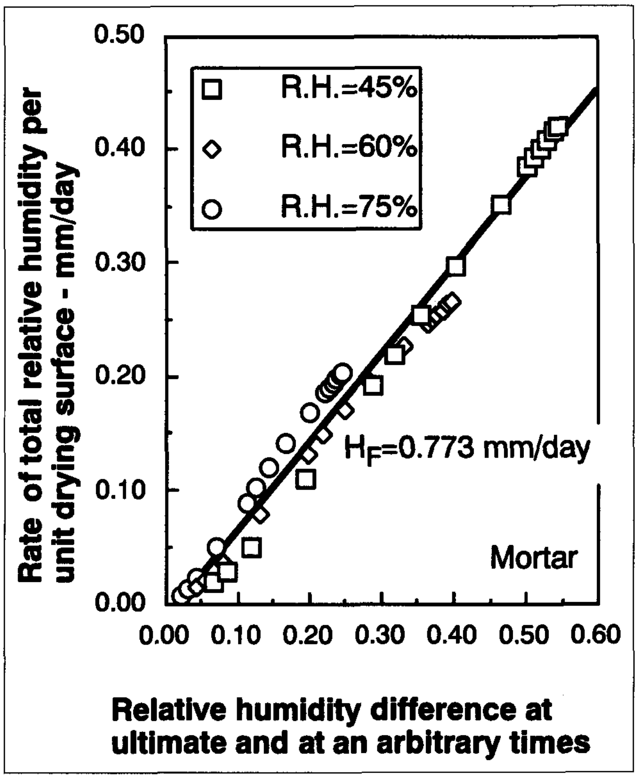

Fig. 7 – Film coefficient of mortar.

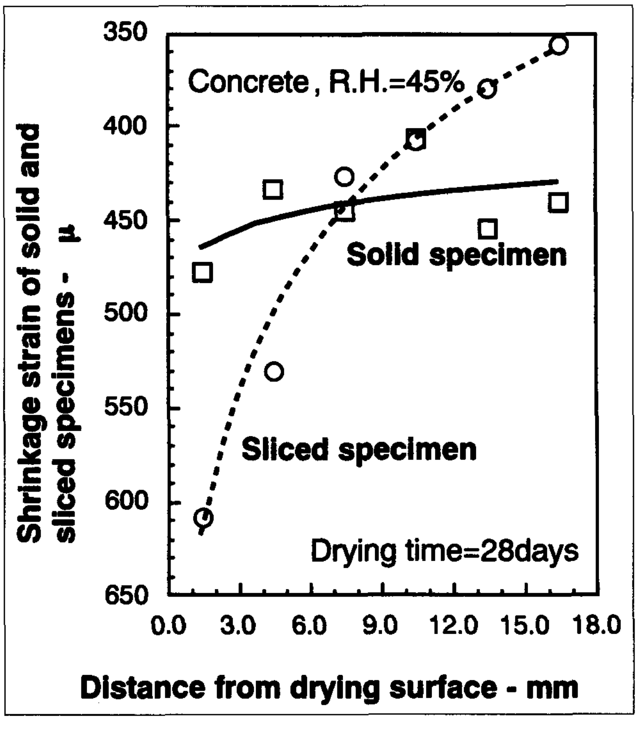

Fig. 8 – Shrinkage strain of piled sliced and solid specimens.

5. DEFORMATION DUE TO DRYING

5.1 Shrinkage coefficient

The shrinkage coefficient expresses the change in strain as a function of moisture or relative humidity change. If a specimen is thin enough so that the moisture distribution can be regarded to be approximately constant, the shrinkage coefficient may be obtained directly. However, the deformation of a very thin specimen may be strongly affected by carbonation. A method of obtaining the shrinkage coefficient without the effect of carbon dioxide needs to be established [7]. In a more precise analysis, the relationship given in Equation (13) can be used if the effect of carbonation, creep and crack formation due to eigenstresses are also calculated and included in the total deformation. Such a calculation may be much complicated. The shrinkage coefficient, which includes the effect of carbonation, eigenstresses and so on, is proposed in this session.

Fig. 8 shows the shrinkage strain measured on solid and sliced specimens. The deformation of sliced specimens due to drying increases almost proportionally to the relative humidity change because each layer is unrestrained. On the other hand, the deformation of solid specimens is nearly independent of the position in the drying sample. Eigenstresses are built up which modify the deformation to ensure compatibility. It is clear that Bernoulli's hypothesis, which states that plane sections remain plane, holds for the drying deformation of solid specimens in case they are long enough and the curvature of the end faces can be neglected. The deformation due to shrinkage strain of solid specimens can be calculated by Equation (20) when Bernoulli's hypothesis holds.

$$\varepsilon_i(t) = \frac{l}{L} \times \sum_{i=1}^{11} Sh_i(t) \tag{20}$$

where $Sh_i(t)$ is the shrinkage strain corresponding to the i-th layer. $\varepsilon_i(t)$ is the shrinkage strain measured at position i in a solid specimen. As l = 3 mm and L = 33 mm, l/L = 1/11. Equation (20) means that the deformation of this size of solid specimen due to drying is independent of the position of measurement.

When the relationship between shrinkage strain and relative humidity of a specimen is expressed by a power law in order to take into consideration as a limit approximation, the effects of carbonation and creep due to eigenstresses, Equation (21) can be derived:

$$\sum_{t=0}^{t=98} \varepsilon_{avg.}(t) = \frac{1}{11} \times a \sum_{t=0}^{t=98} \sum_{i=1}^{11} \left(1 - h_i(t)\right)^b \tag{21}$$

where, $\varepsilon_{avg.}(t)$ is the average deformation measured in a solid specimen. a and b are unknown parameters to be determined by the least square method. The relationship between shrinkage strain and relative humidity of mortar and concrete obtained from Equation (21) can be summarized by Equations (22) and (23), respectively.

$$\text{Mortar:} \quad Sh(h) = 2{,}170\left(1 - h(t)\right)^{0.918} \tag{22}$$

$$\text{Concrete:} \quad Sh(h) = 1{,}370\left(1 - h(t)\right)^{0.749} \tag{23}$$

5.2 Verification of the proposed method

Figs. 9 and 10 show the deformation of cylinders due to drying for the mortar and concrete mixes (one cylin-

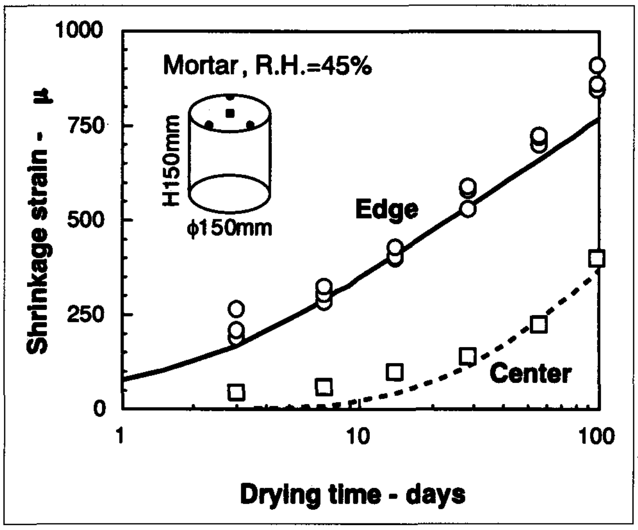

Fig. 9 – Shrinkage strain of cylinders (Mortar).

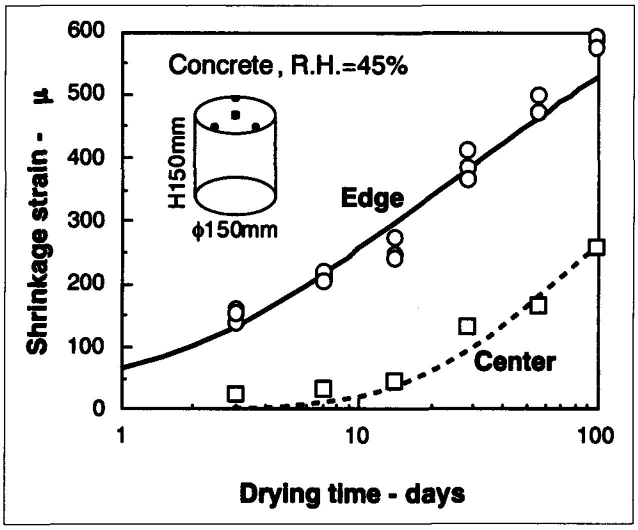

Fig. 10 – Shrinkage strain of cylinders (Concrete).

der only). Circles and squares represent the experimental data measured at the center and the edge of the cylindrical specimens, respectively. The diameter and height of the specimens were both 150 mm. The top and bottom of the specimens were sealed with resin. These data were measured at 45% relative humidity. The curves in these figures were calculated by the finite element method using the diffusion, film, and shrinkage coefficients described in the previous sections. The results show that the calculated values fit the experimental data very well, and demonstrate the validity and applicability of the proposed method.

6. CONCLUSIONS

An experimental approach and a numerical method are proposed to obtain diffusion, film, and shrinkage coefficients of cement-based materials. One advantage of the proposed method is the direct determination of the diffusion coefficient as function of relative humidity for cement-based materials without the need to measure desorption isotherms. The results obtained from experiments with sliced specimens confirm that the influence of gaps between slices on the transfer of moisture is small. Therefore, diffusion coefficient can be determined as function of relative humidity using a pile of sliced specimens without necessary to consider the effect of gaps. A new numerical method to obtain diffusion coefficients from the moisture distribution or relative humidity distribution is proposed. This method is based on the weighted residual method combined with a nonlinear least square method. The shrinkage coefficient was obtained from solid specimens. Bernoulli's hypothesis is satisfied if the drying specimens are long enough. It is shown that the relationship between shrinkage strain and relative humidity of solid specimens can be expressed by a power law. Diffusion, film, and shrinkage coefficients have been determined for mortar and concrete. The results demonstrate the applicability of the method proposed in this contribution.

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