

CONCENTRATION ON THE DIFFUSION
OF CO₂ AND O₂

Y. F. HOUST

Federal Institute of Technology (EPFL), Lausanne,

Switzerland

H. SADOUKI and F. H. WITTMANN

Federal Institute of Technology (ETHZ), Zurich,

Switzerland

Abstract

Gas diffusion process through cementitious materials must be influenced, first of all, by the aggregate concentration, and, secondly, by the interfacial zones between aggregates and bulk cement paste. Indeed, an increase in the aggregate concentration reduces, on the one hand, the porous volume through which gases diffuse, and, on the other hand, augments the volume of the interfacial zones which is more porous than the bulk.

The microstructure of carbonated mortars with increasing sand concentrations has been studied by mercury intrusion porosimetry and water adsorption. The diffusion coefficients of CO₂ and O₂ through the same mortars have been measured at three different relative humidities. In addition, water vapor permeability has been measured through the same mortars at four humidity gradients.

The diffusion coefficients of CO₂ and O₂ are little influenced by water content in the range 55%-86% R.H.. It is also shown that at low sand concentration the diffusion coefficients decrease with the sand content. Above 50% sand by volume, a steep increase is observed partly due to a high volume of interfacial zones which become interconnected.

Keywords: Interfacial zones, CO₂ diffusion, O₂ diffusion, Carbonation, Microstructure, Mortar.

1 Introduction

It is well known that the porosity is of the greatest importance for the durability of cementitious materials. Aggressive substances are transported into the material in the liquid or in the gaseous state through the complex porous system. The carbonation is related to the diffusion of atmospheric carbon dioxide through cementitious materials. Its unfavorable consequences on the durability of reinforced concrete have been already largely discussed. According to Tuvit (1982), the service life of a structure, from the point of view of reinforcement corrosion, can be subdivided into an initiation stage and a propagation stage. The carbonation of the concrete cover takes place during the initiation stage and the steel corrosion during the propagation stage. The corrosion rate, which is practically zero during the initiation stage, is generally limited by the rate of diffusion of oxygen, specially in water saturated concrete. From the surface of a carbonating concrete, one can observe, after a certain time, a fully carbonated concrete part, a reaction front, where carbonation is taking place, and the bulk of non-carbonated concrete. The carbonation proceeds by the displacement of the reaction front. In this simple model, carbon dioxide diffuses through carbonated material. During the propagation stage, oxygen diffuses also through carbonated material. That is why diffusion coefficients of carbon dioxide and oxygen have to be determined through carbonated material. Water, which is produced by the carbonation reaction, diffuses from the reaction front to the surface, where the moisture is lower. The rates of carbon dioxide

Étant donné que, pour les autres échantillons ayant des trous de dimensions inférieures, on retrouve une bonne linéarité, on peut prétendre que seule la zone de transition provoque une hausse de diffusivité. A partir des équations de ces droites, en posant que Ai égale At, les résultats indiquent que la zone de transition aurait une diffusivité environ de 12 à 15 fois supérieure à celle de la pâte. Cependant, dans le cas où l'épaisseur de la zone de transition serait de 10 µm, plutôt que de 100 µm, on aurait une diffusivité environ de 120 à 150 fois supérieure à celle de la pâte, ce qui ne serait pas négligeable. Dans un milieu poreux à température constante, le coefficient de diffusion apparent du Cl⁻ est fonction de l'interaction de ces ions avec le milieu et de la nature des pores de ce milieu. Même s'il n'existe pas de relation bien établie entre la perméabilité et la diffusivité, il n'en demeure pas moins que pour un même matériau, plus la diffusivité est élevée plus la perméabilité est élevée, étant donné que seule la nature des pores en est responsable. Cela indique que la zone de transition serait un chemin préférentiel à l'écoulement et qu'elle pourrait devenir un lieu favorable à la détérioration de la fissure injectée.

6 Conclusion

Les techniques d'analyse utilisant la migration des ions chlore par diffusion ou par champ électrique, sur des échantillons de roche injectée dont, pour une même section, la quantité de pâte reste toujours la même et où seuls le nombre et l'étendue des contacts roche-coulis changent, permettent de caractériser la diffusivité de la zone de transition par rapport au reste de la pâte. De plus, il semble que ces techniques permettent d'observer les effets du retrait endogène de la pâte de ciment à l'intérieur d'une fissure injectée.

Les résultats obtenus indiquent, pour un coulis de ciment de type 10 (E/C = 0,50) injecté dans une roche granitique, que la zone de transition, par rapport au reste de la pâte de ciment, possède une diffusivité plus grande. Dans le cas où celle-ci aurait une épaisseur de 100 µm, la diffusivité de cette zone serait environ de 12 à 15 fois supérieure à celle de la pâte de ciment.

De plus, étant donné que les résultats sont obtenus à partir de la même pâte de ciment, la zone de transition possède également une plus forte perméabilité que le reste de la pâte de ciment.

7 Références

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and oxygen diffusion are dependent on numerous factors like porosity, pore-size distribution, water, cement and aggregate content, curing, time, as well as on the environment: temperature, CO₂ and O₂ concentrations, relative humidity, etc.

The bulk of the experimental data available cannot be understood and interpreted fully because so far there is no comprehensive general theoretical concept of the mechanisms involved in carbonation and corrosion of reinforced concrete. Houst, Roelstra and Wittmann (1983) have developed a numerical model to describe the diffusion of carbon dioxide and oxygen through the concrete structure and the drying process. All parameters of the model can be linked with technological data. However, some of these parameters were unknown. This was true in the case of the influence of moisture content on gas diffusion through porous materials. In a first step, Houst (1991) has determined the influence of the microstructure and moisture content on the diffusion coefficients of CO₂ and O₂ through hydrated and carbonated cement paste. For reasons of time to get an equilibrium with moisture and also of time of measurement of gas diffusion, it was not possible to determine these parameters on representative samples of concrete. That is why we have determined the influence of fine aggregates on the microstructure and on diffusion coefficients. A composite material model is under development to describe the results.

2 Gas diffusion, microstructure and modelling

2.1 Gas diffusion through porous system

The one-dimensional diffusion of a gas can be described phenomenologically by Fick's first law:

$$J = -D \frac{dc}{dx} \quad (1)$$

where J is the flux of the gas, dc/dx the gradient of concentration and D the diffusion coefficient.

Equation (1) defines the diffusion coefficient of two gases diffusing into one another without hindrance. If the diffusion is hindered by a porous medium, the gases must diffuse through the pores of the material. The distance to cross the material is longer than that in unhindered diffusion. The tortuosity is defined as the distance to cross the material divided by its thickness. Thus only an effective diffusion coefficient can be determined for a specimen having the representative thickness :

$$J = -D_{eff} \frac{\Delta c}{d} \quad (2)$$

where d stands for the thickness of the representative porous material. The representative thickness is defined as the minimum thickness for which D_{eff} becomes independent of d.

In reality, diffusion processes in capillaries are complex and it is possible to distinguish at least three different transport mechanisms. The Knudsen diffusion begins to take place as soon as the pores dimensions get smaller than the mean free path of the gas molecules. In large pores, where the mean free path of the gas molecules is smaller than the pore diameter, normal gas diffusion predominates. In addition to the two mentioned mechanisms, surface diffusion also can occur at the same time. These mechanisms have been described in more detail by Houst (1991) who gives also references.

2.2 The cement-paste/aggregate Interface

At least for transport mechanisms, concrete cannot be considered to be a simple composite material consisting of aggregates and hydrated cement paste (hcp). Farran (1956) has demonstrated in some cases the presence of an interfacial zone up to 40 µm

wide between aggregates and cement paste. The existence of this interfacial zone is currently due to the bleeding and depends on the reactivity of the aggregate. It was partly assumed that interfacial zones represent only a small volume in concrete until a study by Diamond et al (1982), who observed that the mean spacing between aggregates in concrete is only 75 to 100 µm. With an interfacial zone of about 40 µm, the most of the paste is included in interfacial zones. Therefore, only a small volume of the bulk hcp can be present in concrete. The concentrations of compounds such as calcium hydroxide and ettringite are larger near the aggregate. The existence of microcracks in the interfacial zone due to drying shrinkage is also observed.

The image analysis of backscattered electron measurements has allowed Scrivener and Pratt (1987), Scrivener and Gartner (1988), and Scrivener et al (1988) to determine the variation of porosity with distance from a piece of aggregate. They observed a dramatic increase of porosity as the interface is being approached. The porosity, which is very high (30%) at 2 µm from the aggregate, drops to a relatively constant level of about 10% after 30 µm. The amount of anhydrous cement grains is very low at the interface and increases almost linearly up to a distance of about 30 µm from the interface. According to Scrivener et al (1988) the particle size distribution of the cement determines the width of the interfacial zone.

The high porosity of the interfacial zone must have a great influence on transport mechanisms such as gas diffusion. An increase of the aggregate content tends to bring interfacial volumes closer together and they become interconnected. This creates continuous channels of high porosity.

It is well known that carbonation reduces the porosity, the specific surface area of hcp and concrete, and, therefore, also gas diffusivity. Houst (1991) has found that the lower the porosity of non-carbonated hcp, the higher the decrease of porosity after carbonation. For example, non-carbonated hcp's have porosities of 24.1%, 32.3% and 48%, after carbonation the porosities drop to 13.0%, 23.5% and 42.3%. In the same paper, it is shown that diffusion coefficients of O₂ and CO₂ through carbonated hcp are much less influenced by relative humidity or water content than expected and found for non-carbonated cementitious materials. It seemed us very important to study the influence of the carbonation on materials with interfacial zones, like mortars, with the aim of determining microstructural changes due to carbonation on carbonated mortars and gas diffusion coefficients.

3 Experimental

3.1 Materials

For the determination of diffusion coefficient in our experimental set-up, we needed samples thin enough to be completely carbonated and equilibrated at different relative humidities in a relatively short time. For this purpose, we prepared Portland cement mortars with a constant water-cement ratio (W/C = 0.6) and three cement-sand ratios (C/S = 0.25, 0.43, 0.67). The cement used was a rapid hardening Swiss Portland cement and the sand was composed fully of quartz grains. The particle-size distributions of the cement and the sand, determined by means of a laser granulometer (Malvern Mastersizer 1002), is given in figure 1. This fine sand was chosen in order to have a large volume of interfacial zones and at the same time to avoid having to cut a large number of aggregates and so modify considerably the properties of interfaces.

After 28 days of curing in a tight cylindrical mold of 160 mm diameter and 320 mm high, discs of mortar of 3 to 5 mm thick were sawn. Then, the specimens have been equilibrated at 76% R.H., a part of them have been artificially carbonated at 76% R.H. in an atmosphere of 80% to 90% CO₂. After a complete carbonation checked by the phenolphthalein test, samples were equilibrated at different relative humidities before CO₂ and O₂ diffusion and H₂O permeability measurements. For mercury intrusion porosimetry and water adsorption, we used pieces of the same discs as those used for diffusion measurements. For the determination of total porosity by water absorption, we used discs of about 30 mm thick.

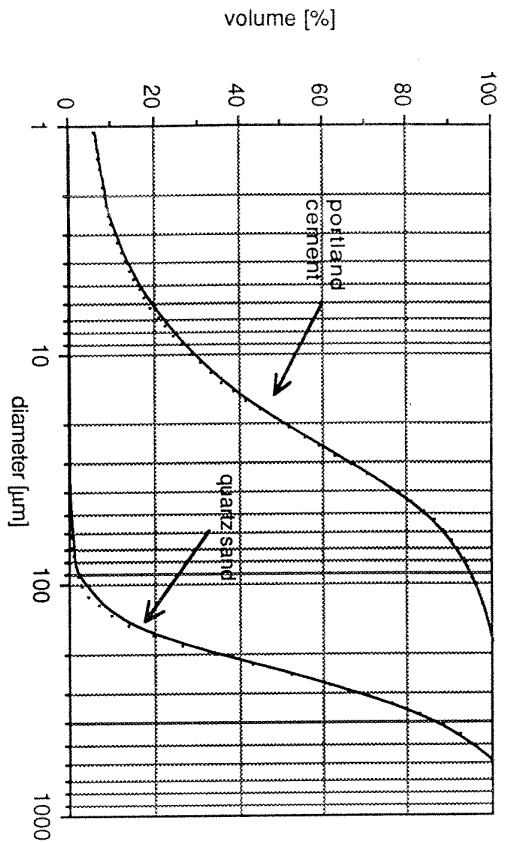


Fig. 1. Particle size distribution of quartz sand and portland cement

3.2 Mercury intrusion porosimetry (MIP)

A standard porosimeter (Micromeritics Autopore 9200) which allows the application of pressures up to 415 MPa has been used. This corresponds to a minimum radius of 1.7 nm. After having introduced a number of simplifying assumptions, the radius r of pores, which can be penetrated by mercury at pressure P , can be related by equation (3):

$$r = -\frac{2 \sigma \cos \theta}{P} \quad (3)$$

where σ stands for the surface tension of mercury and θ for the mortar-mercury contact angle. A value of 135° for θ was used in this study. The pore-size measurements have been carried out on small pieces of mortar (3 to 5 mm) pre-dried at 70°C .

3.3 Water adsorption isotherm

The samples (pieces of 3 to 5 mm), dried at 105°C , were first placed in desiccators where different relative humidities are obtained by saturated salt solutions. After the equilibrium was reached, the water uptake was measured by weighing. The water content as a function of the relative humidity allowed us to draw the adsorption isotherms. All adsorption experiments were carried out at 18°C on non-carbonated and carbonated mortar. The results of carbonated samples are not available at the time of writing this paper.

3.4 Water permeability

The wet cup method has been used to determine the water vapor permeability at 18°C . Four humidity gradients (97%-86% R.H., 86%-76% R.H., 76%-55% R.H. and 55%-33% R.H.) have been obtained by saturated salt solution and have served for the measurements on two discs of each mortar: non-carbonated and carbonated.

3.5 Diffusion of O_2 and CO_2

We have developed equipment which allows us the simultaneous determination of diffusion coefficients of O_2 and CO_2 as a function of the relative humidity, by a non-

steady state method. The heart of the apparatus is a measuring cell which is divided into two rooms by the porous disc of the mortar. At the beginning of the experience, the pre-conditioned gas mixture (2% CO_2 , 20% O_2 and 78% N_2) enters one of the room whilst the other one contains pure nitrogen. Then, the gas mixture diffuses through the mortar into the room filled with N_2 where O_2 and CO_2 are monitored by gas analyzers during one or one and a half day. The diffusion coefficients are deduced from gas concentration-time curves for O_2 concentrations of 2% to 12% and for CO_2 concentrations of 0.2% to 1%. Before and during the measurements, all gases are moistured with water vapor. All measurements were carried out between 20°C and 25°C on fully carbonated mortar discs.

The method of determination of the diffusion coefficients is described in more detail in a publication by Houst and Wittmann (1986).

4. Results and discussion

4.1 Microstructure

The pore-size distribution of the non-carbonated and carbonated mortars are shown in figure 2. Each curve of this figure is the average of 3 to 4 runs. The porosity of pores with radii $< 300 \mu\text{m}$, measured by MIP, and the total porosity are given in table 1. The total porosity of non-carbonated mortars varies between 28.6% and 23%, whilst porosity of pores of radii $< 300 \mu\text{m}$ is quite identical. The total porosity is hardly lowered by the carbonation for mortars of C/S = 0.25 and 0.43. But, that of the mortar

Table 1. Main microstructural properties of the non-carbonated (nc) and carbonated (c) mortars

C/S	Total porosity ¹		Porosity ²		BET sp. surf.		Sp. surf. micro- and meso-pores ⁴	
	[% vol./vol.]		[% vol./vol.]		area[m ² /g]		meso-pores ⁴ [m ² /g]	
	nc	c	nc	c	nc	c	nc	c
0.25	25.4	24.6	15.5	13.4	26.6	14.7	21.2	11.0
0.43	23.6	23.1	14.9	14.9	42.0	19.7	35.2	14.8
0.67	28.6	18.4	15.3	14.9	53.6	23.4	48.5	20.3

¹ Measured by water adsorption under vacuum

² Measured by mercury intrusion porosimetry

³ Calculated from water adsorption isotherm data between 9% and 44 % R.H.

⁴ Calculated from water adsorption isotherm data between 11.2% and 96.4 % R.H.

of C/S = 0.67 is largely reduced. The pore-size distribution is modified: the volume of pores with radii $> 0.03 \mu\text{m}$ is increased for the two mortars with higher cement contents and is decreased for pores with lower radius. The same modification is observed for the mortar of lower cement content, but the limit is about $1 \mu\text{m}$. The pore-size distribution of macro- and micro-pores, computed from adsorption isotherms according to Pierce (1953), is given in figure 3. These results confirm those obtained by MIP. The BET specific surface areas have been also computed from the adsorption isotherms (table 1). The carbonation causes a drastic decrease of specific surface area which is at least divided by 2. This is not surprising as finer pores have higher specific surfaces. The increase in volume of pores larger than $300 \mu\text{m}$ can be explained by their being filled with carbonation products and therefore not taken into account by MIP. According to these results as the carbonation causes only a little drop of total porosity for mortars of C/S = 0.25 and 0.43 and as specially the volume of finer pores is decreased, the transport properties of these carbonated mortars should not be considerably modified. But, it should not be the case for the mortar of C/S = 0.67, which has a significant drop of porosity.

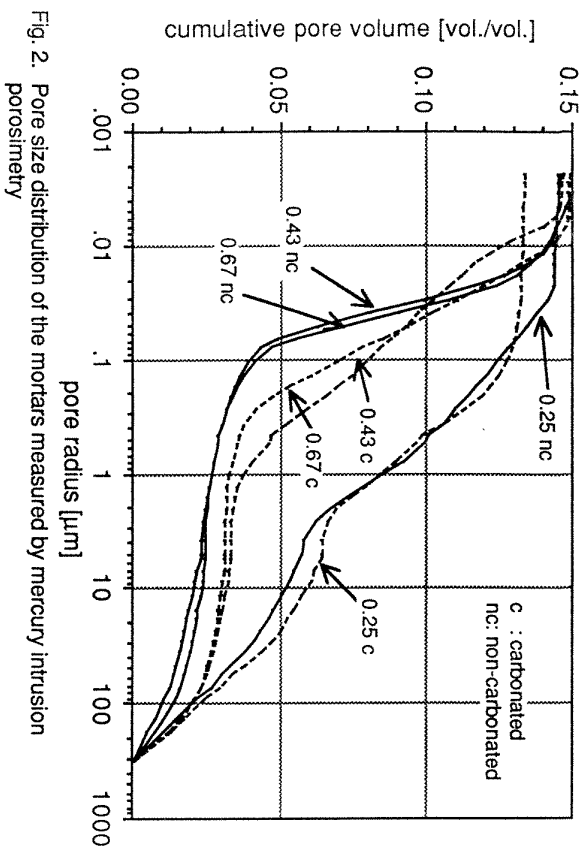


Fig. 2. Pore size distribution of the mortars measured by mercury intrusion porosimetry

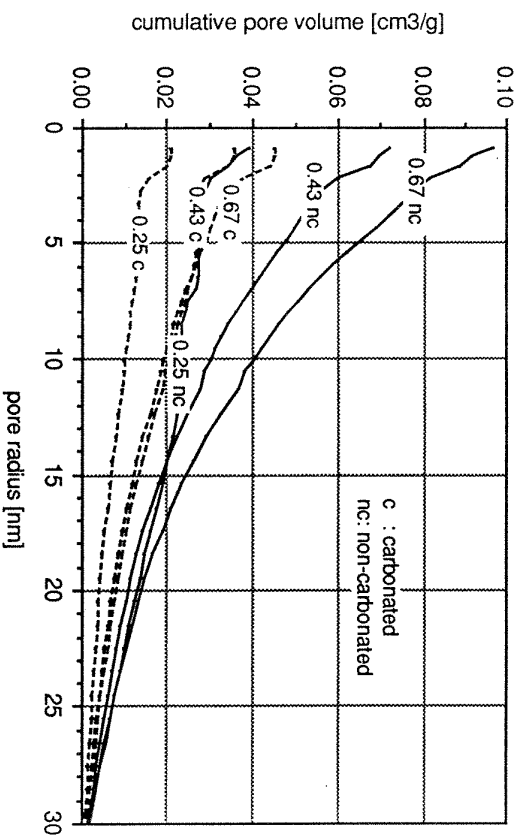


Fig. 3. Meso- and micro-pore size distribution calculated from adsorption isotherms data of the mortars (0.25, 0.43, 0.67 are C/S)

The difference between total porosity, measured by water absorption under vacuum, and porosity, measured by MIP is about 10% to 13%. This difference is essentially due to the presence of air pores, entrained by the fine aggregate. The volume of the interfacial zones has been computed on the basis of the particle size distribution of the

sand and by assuming spherical particles, for interfacial zones from 30 µm to 10 µm thick. The corresponding volumes are 63.6% (C/S=0.25) and 11.9% (C/S=0.67). By assuming an interfacial zone of 20 µm thick, the interfacial volume and the aggregate volume are respectively 93.25, 82.2%, 65.1% for C/S=0.25, 0.43 and 0.67. With this simple model, it is shown that the interfacial zones represent the main part of cement paste. Therefore, the high air-pores content of the mortars must reduce the effect of transition volumes in gas transport.

4.2 Water permeability

The results of water permeability measurements on non-carbonated mortars are given in table 2. The one-dimensional permeation of water through a porous system can be described by equation (4):

$$J = -\lambda \frac{dH}{dx} \quad (4)$$

Where J stands for the flux of the gas and $\frac{dH}{dx}$ the gradient of moisture and λ the coefficient of permeability. If the water content in equilibrium at a given relative humidity w is used instead of the relative humidity H, the flux of moisture can be written as follow:

$$J = -D \frac{dw}{dx} \quad (5)$$

Where D is the diffusion coefficient. By means of equations (4) and (5), one can write equation (6):

$$D = \lambda \frac{dH}{dw} \quad (6)$$

$\frac{dH}{dw}$ being the inverse of the adsorption isotherm derivative.

Table 2. H₂O permeability and diffusion coefficients of the non-carbonated mortars
Cs: sand concentration by volume

ΔR.H. [%]	Cs = 55.1%		Cs = 48.6%		Cs = 38.5%	
	λ [kg/msPa]	D _{H2O} [m²/s]	λ [kg/msPa]	D _{H2O} [m²/s]	λ [kg/msPa]	D _{H2O} [m²/s]
97-86	1.7-10-12	7.5-10-12	2.0-10-12	5.3-10-12	2.0-10-12	4.4-10-12
86-76	1.7-10-12	1.8-10-11	1.1-10-12	6.9-10-12	9.7-10-13	4.3-10-12
76-55	1.2-10-12	3.6-10-11	7.8-10-13	1.3-10-11	6.8-10-13	8.4-10-12
55-33	2.0-10-12	7.0-10-11	9.3-10-13	2.4-10-11	6.2-10-13	1.6-10-11

The diffusion coefficient of water as a function of the relative humidity, computed according to equation (6), is also given in table 2. These diffusion coefficients decrease as the relative humidity increases; this behavior being more pronounced for mortars with higher total porosity and equilibrium water content. This is because adsorbed and condensed water hinders the transport of vapor. The diffusion coefficient decreases as the aggregate content increases. This is due to more bigger pores associated with mortars having not a sufficient cement content to fill all voids between aggregates. As the adsorption isotherm of carbonated mortar are lower than those of non-carbonated mortars and as the total porosity is little lowered by carbonation, the diffusion coefficient would be only hardly influenced by carbonation, at least for R.H. < 90%.

4.3 CO₂ and O₂ diffusion coefficients

The results of the experiments to determine the diffusion coefficients are shown in table 3. It is obvious that the moisture content has a little influence on the measured diffusion coefficients in the covered range. Measurements have also been carried out on heat hardened cement paste. In figure 4, the diffusion coefficients for CO₂ and O₂ are plotted as a function of the sand concentration. As can be expected at low concentrations, the diffusion coefficients decrease with the sand content. Above 50%, however, a steep increase is observed. Similar behaviour has already been observed with moisture diffusion in concrete.

Table 3. Diffusion coefficient of CO₂ and O₂ of the carbonated mortars
C_s: sand concentration by volume

R.H. [%]	C _s = 55.1%		C _s = 48.6%		C _s = 38.5%	
	D _{O2} [m ² /s]	D _{CO2} [m ² /s]	D _{O2} [m ² /s]	D _{CO2} [m ² /s]	D _{O2} [m ² /s]	D _{CO2} [m ² /s]
55	1.0·10 ⁻⁷	7.0·10 ⁻⁸	1.8·10 ⁻⁸	1.1·10 ⁻⁸	3.2·10 ⁻⁸	2.7·10 ⁻⁸
76	9.8·10 ⁻⁸	6.7·10 ⁻⁸	1.5·10 ⁻⁸	7.0·10 ⁻⁹	3.5·10 ⁻⁸	2.2·10 ⁻⁸
86	1.0·10 ⁻⁷	6.2·10 ⁻⁸	2.0·10 ⁻⁸	8.0·10 ⁻⁹	4.6·10 ⁻⁸	1.5·10 ⁻⁸

This phenomenon can be explained by the role of the interfacial zones or the aureoles de transition in the diffusion process. The composite structure can be simulated in a two dimensional model (see figure 5). As long as the interfacial zones are well separated from one another, the diffusion process is governed by the porosity of the matrix. Above a certain concentration the aureoles de transition become interconnected and thus create channels for easy diffusion.

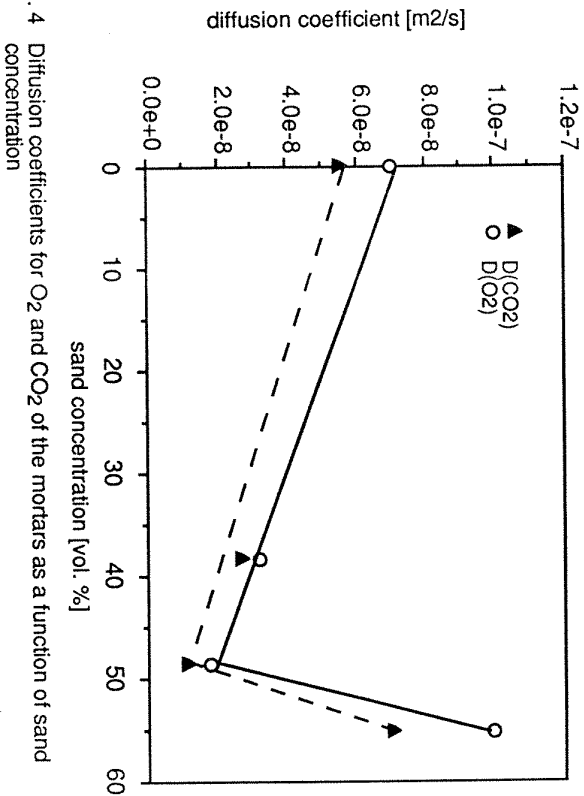


Fig. 4 Diffusion coefficients for O₂ and CO₂ of the mortars as a function of sand concentration

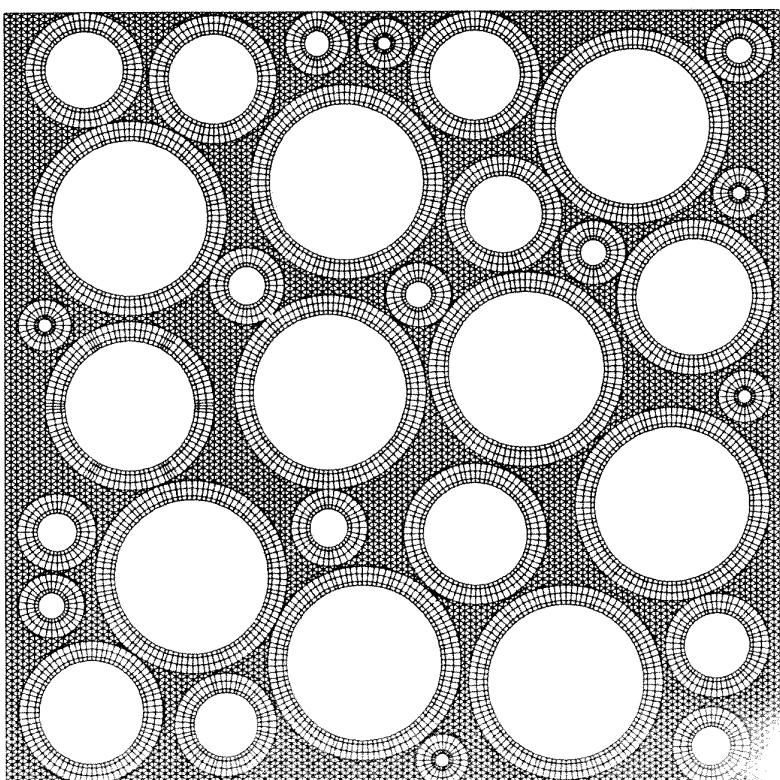


Fig. 5 Finite volume representation of a computer-generated random composite structure (3 phases are present: matrix, aureole de transition and inclusions)

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30 INTERFACE INFLUENCE UPON THERMAL CONDUCTIVITY OF LIGHTWEIGHT CONCRETES

V. S. GRYSLOV
R.I. Laboratory for Testing Building Materials,
Cherepovets, Russia

Abstract

This article contains the results of experimental research of interface influence upon the thermal conductivity of lightweight concrete. A theoretical analysis of this influence has been carried out and a physical and mathematical model has been created. The model makes it possible to predict lightweight concrete thermal conductivity taking into account its aggregate structure and interface areas.
Keywords : Thermal, Conductivity, Interface, Physical and Mathematical Model.

1 Introduction

Modern levels of quality control of lightweight concrete manufacture require more precise prediction of its properties. Therefore, along with improving techniques of increasing lightweight concrete porosity and researching new ones, we need a scientifically grounded concept of regulation and minimization of lightweight concrete thermal conductivity through other structure forming factors, such as : friction composition of aggregates, interlace, binder microstructure etc..., the pre-set strength being minimal.

At present this concept is developing based upon common laws of the general theory of materials as a structural theory of lightweight concrete conductivity.

2 Experimental research

Common principles of mathematical modelling for the evaluation of thermal conductivity of materials are based upon the analysis of two-component systems (matrix-inclusion) such as models made by Krishcher, Ribean, Missenard, Russel, Odelevsky et al.

We have performed experimental testing of the models mentioned with slag concrete taken as a sample.

At the first stage samples of different phase composition were used for our research. Preparing the slag samples included grinding, melting at 1200-1300 ° C and cooling under various conditions. As a result, the degree of slag crystallisation ranged from 30 % to 80 %.

Phase composition was defined by the light microscopy technique as well as the X-ray technique. Thermal conductivity was defined by the technique of stationary thermal flow on flat samples cut out of slag specimens.

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Advanced composites and the fundamental understanding of their behaviour is a rapidly expanding area of civil engineering. These materials can be designed to have outstanding combinations of strength (five-to ten-times that of conventional concrete) and energy absorption capacity (up to 1000-times that of plain concrete). Exciting engineering applications are therefore being developed to take advantage of these properties for blast resistant, earthquake resistant and offshore structures, and also for building components such as cladding, pipes, tiles and roofing.

This book provides a compendium of the most recent research advances and reviews presented at an international workshop held under the auspices of RILEM and the American Concrete Institute in Mainz, Germany in June 1991. It includes over 40 contributions from the leading international specialists and researchers from USA, Europe, Japan and elsewhere and is an essential reference for engineers and researchers who need to be at the forefront of developments in cement composites.

Among the materials discussed are composites containing steel, polypropylene, polyacrylonitrile, carbon, glass and cellulose fibres. SIFCON is extensively covered. The other main topics considered are: technologies of production; composite optimization; mechanical fracture and interface properties; modelling; structural applications and implications.

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M. KAWAMURA
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