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**EFFECT OF APPLIED STRESS ON THE HELIUM INFLOW
CHARACTERISTICS OF HYDRATED PORTLAND CEMENT**

by R.F. Feldman and J.J. Beaudoin

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RÉSUMÉ

Des études du ciment Portland hydraté poreux ont été menées pour évaluer quantitativement certains aspects de la déformation de la microstructure due à une contrainte appliquée. Une pâte de ciment Portland hydratée durcie pendant six ans à un rapport eau/ciment de 0,6 a été tranchée en disques de 1,25 mm d'épaisseur. Ces disques ont été placés sur un support d'échantillon spécialement conçu qui a été utilisé avec un appareil de mise en charge permettant d'appliquer aux échantillons des contraintes de compression uniaxiales de 7,1, 14,8 et 22,8 MPa. Des mesures au pycnomètre et des mesures d'admission d'hélium ont été prises à des diminutions de teneur en eau de 0, 2,5, 4,5, 7,0 et 9,0 pour cent à partir d'une humidité relative à 11 pour cent. Les effets les plus importants se sont produits après une diminution de 7,0 pour cent de la teneur en eau; l'admission d'hélium a alors été réduite de 48 pour cent à 40 h. Les densités ont également été réduites par la contrainte, et on en a conclu que les orifices d'entrée à l'espace inter-couches rétrécissaient pendant l'application de la contrainte.

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EFFECT OF APPLIED STRESS ON THE HELIUM INFLOW CHARACTERISTICS
OF HYDRATED PORTLAND CEMENT

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ABSTRACT

Studies of porous hydrated portland cement have been carried out to assess quantitatively some aspects of microstructural deformation due to applied stress. Hydrated portland cement paste cured for six years at a water/cement ratio of 0.6 was sliced into discs 1.25 mm thick. These were positioned in a specially designed sample holder and loading device that permitted the specimens to be placed under uniaxial compressive stress at 7.1, 14.8 and 22.8 MPa. Helium intake and pycnometric measurements were made at 0, 2.5, 4.5, 7.0 and 9.0 per cent moisture loss with respect to the 11 per cent RH condition. Maximum effects were observed at 7.0 per cent moisture loss, at which helium intake was reduced by 48 per cent at 40 h. Densities were also reduced by stress, and it was concluded that entrances to interlayer space were constricted during application of stress.

Introduction

The mechanical properties of cementitious materials such as cement paste, mortars and concrete are dependent on porosity and pore size distribution, properties that are very critical with regard to permeability and resistance to chemicals (1,2). The porosity of these materials is difficult to define and measure; it has to be done in a specific way because a large part of the space vacated by water is interlayer space. In contrast with other pore space, interlayer space has special properties, for example, it is possible to increase substantially the modulus of elasticity at a given porosity if complete re-entrance of interlayer water occurs after it has dried initially (3). In addition, interlayer space does not retain its size and shape when water is removed, and this results in changes in mechanical properties (4).

Many studies have been made of the effect of stress on cement paste or concrete (5). It has always been assumed that deformation of the specimen following applied stress is not primarily a result of change in the geometry of

the pores but of a change in a basic property of the solid material of the specimen. Such studies have not included any quantitative assessments of microstructural deformation due to applied stress.

The helium inflow technique has been used in studying hydrated tricalcium silicate and portland cement systems, permitting observation of changes in solid volume, density and volume of interlayer space that occur during removal and re-entry of water (4). Helium intake versus time curves for a sample at different moisture content levels provided the data and ultimate basis for an assessment of these changes. The changes in properties and composite terms calculated from these measurements were compared and provided a means of determining the extent to which the space vacated by the water was affected.

It was the purpose of the present study to assess the effect of applied stress on the rate of flow of helium into hydrated portland cement. Study of the changes in quantities measured by the helium inflow technique that resulted from changes in applied stress will allow assessment of the effect of stress on the pores and should provide some insight into the nature of the pores. It should also provide some understanding of the basic phenomena responsible for creep of concrete under applied stress.

Experimental

Materials

A type I portland cement was hydrated at a water/cement ratio of 0.6 for approximately six years. Discs 1.25 mm thick and 32 mm in diameter were cut from cylinders and used as samples for helium intake measurements. Each disc had a 7.94 mm hole at its centre and radial grooves every 20 deg.

Helium Comparison Pycnometry and Helium Inflow

The apparatus and procedure for helium inflow measurements have been described (4). The apparatus differed, however, in that the sample-holding device, which maintains the specimens in a stressed state, was placed in the pycnometer sample cylinder with the hydrated portland cement discs installed. Helium intake into the sample was measured by the pycnometer for 40 h, then the density of the material was measured. The technique uses the gas laws and the ideal gas assumption for calculating solid volume of the sample. Interlayer spaces or very small and partially entrapped spaces that might delay flow of helium into the sample were considered as part of the solid or as pores in calculating density.

Apparatus for Stressing Samples

The apparatus for holding the specimen in place and the stressing equipment are shown in Fig. 1. The sample is composed of about 15 discs resting on top of each other on a hardened steel base. A threaded prestressing wire is attached to the base and passes through the central hole in each disc. A sufficient number of discs are mounted so as to overlap, with the threaded portion at the top of the wire. A threaded cap is screwed in place on top of the last disc.

The hydrated cement samples conditioned at 11 percent RH weighed 15.5g. Figure 1B shows how the sample-holding device was placed on a reaction frame. The exposed, threaded end was screwed to a tension rod and this, in turn, was fastened to a bearing nut. Tension to the prestressing wire was applied by a hydraulic jack through the bearing unit. After the required tension was attained, as determined from the calibrated gauge on the jack, the threaded cap was tightened securely to the specimen and the pressure in the jack was released so that

FIG. 1

Schematic of sample-holding device and stressing apparatus

A. SAMPLE-HOLDING DEVICE - SCHEMATIC

B. SAMPLE STRESSING APPARATUS - SCHEMATIC

stress (compressive) was transferred to the samples. (It was assumed that the final compressive load was equal to the initially applied tensile load. Loss of tension in the wire, detected at the load at which the initially threaded cap became loose on unloading, is small and corrections were not made for this.) The assembly was then placed in the sample cylinder of the pycnometer. Pressures used in this work were 7.1, 14.8 and 22.8 MPa. It was estimated that the tension in the prestressing wire attained 445 MPa.

General Procedure

After fabrication, samples were conditioned to 11 per cent humidity and loaded in the holder. The whole assembly was then transferred to the pycnometer sample cylinder placed in the gloved box. Density and helium intake measurements were determined. Prior to increasing stress on the specimens to the next level, the samples were degassed under conditions similar to those necessary to bring them to the moisture level at which measurements were being made. Moisture losses relative to 11 per cent RH, at which specimens were taken, were 2.5, 4.5, 7 and 9 per cent. In order to change moisture content the stress on the samples was reduced to zero and those still in the sample holder were placed in a separate vacuum vessel. The samples were evacuated or heated and evacuated; the last two moisture levels were attained by heating to 75°C in the latter stages of evacuation. Stress was removed or increased by replacing the sample on the reaction frame and connecting it to the tension rod; the pressure in the hydraulic jack was increased to the level to which it had been raised previously. The effective stress on the samples themselves was therefore zero and the threaded cap could be loosened in preparation for degassing treatment.

Results and Discussion

Helium Intake as a Function of Time

The helium intake versus time curve at a weight loss of 7.0 per cent relative to the 11 per cent condition is presented in Fig. 2. Results are shown for samples under compressive stresses of 0, 7.1, 14.8 and 22.8 MPa. Helium intake values are accurate to ± 0.05 ml/100 g. At 14.8 MPa the volume of helium intake after 40 h is reduced by almost 48 per cent compared to that for the sample at no load. Further increase of stress to 22.8 MPa is actually accompanied by an increase in helium intake compared to that at 14.8 MPa. This

FIG. 2

Helium intake versus time as function of applied stress at 7.0% weight loss

reversal takes place as well at the 11 per cent RH condition, but at 2.5, 4.5 and 9.0 per cent weight loss levels the helium intake decreases with applied stress. These curves are typical for all weight loss levels and generally flatten out more abruptly at the higher stresses.

The change in the total volume of helium intake at 40 h as a function of moisture content is shown in Fig. 3 at the four stress levels. The value for zero stress level is similar to that published previously for a water/cement ratio of 0.6 paste (4). The results show that stress has a greater effect in decreasing helium intake at higher moisture losses (up to a loss of 7 per cent). The effect is generally greater with increasing stress, although there is some overlap.

The decrease in total helium intake after 40 h, relative to unstressed samples, is plotted as a function of moisture loss in Fig. 4. The results are shown for the three stress levels used: 7.1, 14.8 and 22.8 MPa. They are compatible with an interlayer model for the pores and can be supported in this view as follows: at the 11 per cent condition (zero moisture loss) there is little change in helium intake under applied stress since the spaces are largely occupied by water. There is, however, a slight increase in intake at 22.8 MPa. As water is removed from the interlayer space without major collapse of the layered structure the vacated space can be filled with helium. Applied stress can reduce the size of the entrances to the water-vacated spaces and possibly lengthen them by bringing surfaces at the entrances closer together (see Fig. 5B), thereby reducing the rate of entry of helium. As the vacated space increases, the helium intake after 40 h (due to constricted entrances) decreases. This effect reaches its maximum at around 7 per cent weight loss, where the effect is greatest not at the applied stress of 22.8 MPa but at 14.8 MPa. This latter effect may be the result of bringing sufficient surfaces into close enough proximity at 22.8 MPa to form new space into which helium flows. At moisture losses greater than 7 per cent the difference between the

FIG. 3

Helium intake versus moisture loss at different stress levels.

FIG. 4

Change in helium intake versus moisture loss at various stress levels

stressed and unstressed samples decreases abruptly. This is due to the fact that in the unstressed state interlayer space also collapses abruptly in this region (Fig. 3). Removal of water alone reduces the entrance size of the interlayer space. At this moisture level, however, some further constriction of the entrances still seems to take place on application of stress.

Density Measurements

Density values may be calculated in two ways: (1) using the solid volume calculated from data obtained by compressing the helium gas surrounding the sample from one to two atmospheres (this method, in which helium intake has not occurred, is usually used for density measurements); (2) recalculating the values for solid volume obtained in (1) by subtracting the volume of helium intake at 40 h (Fig. 6) (6).

Results of density calculations from (1) vary little with applied stress. At 7.0 per cent weight

FIG. 5

Strain at specific regions due to stress and change in moisture content

loss, where maximum effects occurred with regard to decrease in helium intake, the density over the whole stress range varied from 2.19 to $2.20 \pm 0.01 \times 10^3 \text{ kg/m}^3$, with little or no apparent dependency on stress. It must be concluded that the decrease in rate of helium intake is largely due to constriction of the entrances to vacated interlayer spaces and not to elimination of these spaces.

The results in Fig. 6 are also plotted as a function of applied stress. It was found that up to about 6.0 per cent moisture loss (without applied stress) helium enters all the spaces previously vacated by water at 40 h (4). The more complete the entry of helium the lower will be the calculated solid volume and the higher the density. At zero applied stress density increases with moisture loss up to 7 per cent, then decreases at 9 per cent loss. The effect of stress at zero moisture loss is slight, but from 2.5 to 7 per cent moisture loss it increases progressively. Density mainly decreases with increasing

applied stress, probably as a result of constriction at the entrances and the resultant inability of helium to enter all the space within 40 hours. At 2.5 percent weight loss the density decreases from 2.31 to $2.26 \pm 0.01 \times 10^3 \text{ kg/m}^3$; at 4.5 percent, from 2.34 to $2.26 \pm 0.01 \times 10^3 \text{ kg/m}^3$; at 7.0 percent, from 2.40 to $2.31 \pm 0.01 \times 10^3 \text{ kg/m}^3$; and at 9 percent weight loss, from 2.34 to $2.31 \pm 0.01 \times 10^3 \text{ kg/m}^3$.

It is clear that moisture content has a significant influence on the observed effect of applied stress. As water is removed from the 11 per cent RH condition, the interlayer spaces are vacated. Thus the potential for helium intake is greater and, at the same time, under applied stress the constriction that may occur at the entrances may also be greater. At 9 per cent weight loss this effect of applied stress diminishes, probably because collapse of both spaces and entrances occurs largely by prior removal of water.

It is suggested that portions of the spaces vacated by water, i.e., the entrances and possibly regions near the entrances, may be significantly reduced in cross-sectional area and that water from the spaces may have a significant role in resisting deformation due to stress. A few experimental points in Fig. 6 deviate from the general trend; for example, the point for 4.5 per cent moisture loss at 14.8 MPa stress. Whether this is due to experimental error or to the irreversible effects of previously applied stress is not known.

That "fixed-dimension" pores possibly occurring in the cement paste are not responsible for the differences in helium intake can be explained: Consider a prismatic specimen having modulus of elasticity, E , under compressive stress,

FIG. 6

Change in density with pressure at different water contents

σ , resulting in an over-all average strain, ϵ . If this specimen contains simple cylindrical pores like that shown in Fig. 5A, and if it is assumed that the over-all E , including the pore, is 6900 MPa, there will be a strain ϵ of 0.1 per cent under a compressive load of 6.90 MPa. If, however, one assumes no overlapping of stress fields from one pore to another but considers the use of E_0 (the zero porosity modulus and stress intensification of approximately 3 in the vicinity of the pore itself), it will still be found that strain in the vicinity of the pore is of the order of 0.1 per cent. The maximum stress in the vicinity of the pore is approximately three times the average stress (7). This hardly accounts for the 48 per cent reduction in helium intake. But if local strains at the ends of layers are considered to be derived from the partial collapse of water-vacated interlayer space, as shown in Fig. 5B, where interlayer sheets are partially drawn together by drying alone, it will be understood how applied stress can cause constriction of the entrances and reduction in helium intake of the magnitude observed in these experiments.

Figure 5C shows how the model for a disordered layered structure can provide many sites where constriction of entrances can cause decreased helium intake. A model such as this has already been used to describe irreversible creep (8), and the possibility that these sites may be constricted by the application of stress has significant ramifications with regard to this mechanism. More study will be necessary to further the understanding of the effect of repetitive loading and unloading, and of changing moisture content on a previously loaded sample.

Conclusions

1. Helium intake is decreased almost 50 per cent by applying 14.8 MPa compressive stress to a sample of hydrated portland cement paste.
2. Little reduction in helium intake occurs at 11 per cent RH when stress is applied to the systems.
3. The reduction of helium intake due to applied stress is a function of moisture content, the maximum effect occurring at 7.0 per cent moisture loss (with respect to the 11 per cent RH condition).
4. Applied stress has no significant effect on solid density values and elimination of interlayer space is unlikely.
5. Reduction of helium intake is probably due mainly to constriction of narrow interlayer entrance positions vacated by interlayer water that contributed to the stiffness of the material.
5. Helium intake results are consistent with the hypothesis of a disordered, layered sheet model for hydrated calcium silicate.

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