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**IMPREGNATION AS A TECHNIQUE
FOR MEASURING ZERO POROSITY MODULUS
OF POROUS MATERIALS**

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

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IMPREGNATION AS A TECHNIQUE FOR MEASURING
ZERO POROSITY MODULUS OF POROUS MATERIALS

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ABSTRACT

The validity of the equation $E = E_{01} \exp(-bP)$ is tested. This relates Young's modulus of a porous body to its porosity; E_{01} represents the modulus of the zero porosity material and can be determined by extrapolation. Impregnation of the porous body with sulphur, and use of the Reuss equation

$$E_c = 1 / \left(\frac{V_1}{E_{01}} + \frac{V_2}{E_{02}} \right)$$

provides an alternate method for determining E_{01} . Both methods are applied for seven different portland cement systems. The results differ from each other by 5 to 23 per cent.

→ La validité de l'équation $E = E_{01} \exp(-bP)$ est mise à l'essai. Ceci met en rapport le module Young d'un corps poreux avec sa porosité; E_{01} représente le module du matériau à porosité zéro et peut être déterminé par extrapolation. L'imprégnation d'un corps poreux par le soufre et l'utilisation de l'équation Reuss

$$E_c = 1 / \left(\frac{V_1}{E_{01}} + \frac{V_2}{E_{02}} \right)$$

fournissent une méthode alternative pour déterminer E_{01} . Les deux méthodes sont appliquées à sept différents systèmes de ciment portland. Les résultats diffèrent l'un de l'autre par 5 à 23 pour cent.

Porosity is recognized as one of the major parameters controlling the mechanical properties of porous bodies (1-5). Much research has been devoted to understanding the effects of this factor and several expressions have been used to correlate data with it.

One of the most common expressions used to correlate modulus of elasticity with porosity is the following:

$$E = E_{01} \exp(-bP) \quad [1]$$

where E is the modulus of elasticity of the porous body, E_{01} the modulus of elasticity for zero porosity, b a constant and P the porosity. This equation has a theoretical base for its derivation (2, 4) although the final form (Eq. [1]) includes several simplifying assumptions. The constant 'b' depends on pore geometry and orientation of the pores with respect to stress; one kind of pore geometry and orientation is assumed in Eq. [1]; this equation does not hold for high porosities (2). Despite this limitation, data for many ceramic systems such as alumina, magnesia and thoria have been fitted and good agreement has been obtained for intermediate porosities. The constant 'b' was different for each set of data. Uncertainty concerning the residual stresses in the body, the nature of the crystalline boundaries and extrapolation to zero porosity through an equation that may be considered at best semi-empirical, would lead one to question the accuracy to which E_{01} could be determined from Eq. [1] (6) especially for cold pressed systems and systems crystallized from solution.

In a previous paper (7), it was shown that a mixing rule could be used to determine the modulus of a sulphur impregnated porous body and that the impregnated body behaved like a homogeneous two-phase composite. The equation for the composite, derived from Reuss' model is:

$$E_c = \frac{1}{\frac{V_1}{E_{01}} + \frac{V_2}{E_{02}}} \quad [2]$$

where E_c is the modulus of the composite, V_1 & V_2 the volume fraction of the matrix and impregnant, respectively, and E_{01} and E_{02} the moduli of the bulk non-porous matrix and impregnant, respectively. The equation is derived (8) by considering that the composite consists of two flat slabs perpendicular to the application of stress.

E_{01} of a matrix material can be computed from Eq. [2] and a knowledge of the modulus of the composite, the volume fractions V_1 and V_2 , and E_{02} for the impregnant. The purpose of this paper is to compare E_{01} determined by two methods: (a) the mixing rule (Eq. [2]) after impregnation with a known material, and (b) by extrapolation of Eq. [1] to zero porosity.

Experimental

Porous bodies

The bodies for impregnation were prepared by autoclaving portland cement silica mixtures; these contained 5, 10, 20, 30, 50 and 65% silica; room-temperature cured paste was also used. Six water-cement ratios between 0.22 and 0.45 were prepared for each composition. Impregnation techniques and measurement of mechanical properties before and after impregnation have been described (7).

FIG. 1

Modulus of elasticity as a function of volume fraction of sulphur for various silica contents

Results

The dependence of the modulus of elasticity on the volume fraction of sulphur is presented in FIG. 1 by linear regression lines determined from experimentally-determined points. These were presented in a previous paper (7) and represent the seven different preparations mentioned above.

For each preparation a value of E_{01} was calculated with Eq. [2], E_{02} , and the experimental E_c value (modulus of the composite) at the low volume fraction of sulphur end of the E_c versus volume fraction of sulphur line. With this value for E_{01} , values of E_c were computed for a range of volume fraction using Eq. [2]. They are plotted with the experimental data in FIG. 1. For zero porosity sulphur, E_{02} was assumed to be equal to 13.9×10^3 MPa (9). For all preparations the calculated curve deviated increasingly from the experimental line with increasing volume fractions of sulphur, the experimentally determined modulus values being smaller than those predicted by Eq. [2]. The main reason for this deviation was that the residual porosity due to the contraction of sulphur on solidification increased with increase of volume fraction of sulphur. It was shown previously (7) that this residual porosity was composed of two types, one accessible, and the other inaccessible to helium. It was concluded that the inaccessible porosity was trapped in the solidified sulphur. This occurs to a greater extent at low volume fractions of sulphur whereas accessible porosity occurs mainly at the higher volume fractions. It was assumed that the shape and orientation of the pores within the solidified sulphur were such that their effect on Young's modulus compared to the effect of pores accessible to helium was minor (8). It is clear however that this assumption can be a source of error.

The data for the different values of E_{01} , obtained by calculation from Eq. [2] and by the extrapolation to zero porosity technique done previously (5) for the seven preparations, are tabulated in TABLE I; the table includes the volume fraction at which each was calculated; these are also plotted against each other in FIG. 2. The difference between the two values of E_{01} averages to 13.5%, a maximum of 23% for the 10% silica preparation to a minimum of 5.8% for the 30% preparation. This is considered to be extremely good considering the potential source of error for both techniques.

FIG. 2

E_{01} , calculated from mixture law vs E_{01} determined by extrapolation of $\log E$ vs porosity

FIG. 3

Modulus of elasticity vs silica content

TABLE I

Modulus of Elasticity from Mixture Rule and Extrapolation

% Si	V_s	$E_{\text{mixture}} \times 10^{-3}$ MPa	$E_{\text{extrapolation}} \times 10^{-3}$ MPa	$\frac{\{E_{Mx} - E_{\text{ext}}\} \times 100}{E_{Mx}}$
5	0.30	82.4	70.5	+17
10	0.28	71.0	88.7	-23
20	0.21	47.7	56.9	-19
30	0.20	41.1	38.7	+ 5.8
50	0.20	38.4	42.0	- 9.0
65	0.20	42.7	47.8	-11.7
Room Temp.	0.30	33.0	30.0	+ 9.1

Discussion

The fact that two independent procedures:

- extrapolation of the exponential relation (Eq. [1]), and
- fitting of the simple mixing rule relation (Eq. [2]) to the experimental data obtained by impregnation of specimens,

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produce similar values of E_{01} of the matrices is evidence that the procedure of extrapolating Eq. [1] to zero porosity to obtain E_{01} is well-founded and that the constant E_{01} does in fact represent Young's modulus of the matrix material at zero porosity.

Two cases of particular interest can be considered in relation to the above facts.

1. Of the seven preparations, modulus values of the samples made from the 5 and 10% silica are the lowest (see FIG. 3). These preparations are composed of predominantly α -C₂S (5). Impregnation of these samples results in the largest increase in the modulus. Calculation by mixing rule (Eq. [2]) gives a high E_{01} . Likewise, high values for E_{01} are obtained for the matrices by extrapolation of the log modulus to porosity plots despite the low values of modulus obtained for the porous bodies. It is suggested that the shape of the pores or the type of contact between individual crystallites is such that the stress concentration due to low bond area is high, resulting in large deflections under load and low modulus of elasticity. Impregnation is obviously effective in increasing the effective bond area.

Equation [2] does not contain any stress concentration term. It has been suggested (7) that impregnation modifies local stresses for flat pore elliptical geometry. It appears that any stress concentrations that remain after the pores are filled with sulphur do not affect significantly the calculation of elastic modulus of the composite. This also suggests that the determination of E_{01} is not significantly affected by the presence of any remaining stress concentrations after the pores are filled.

2. It has been shown (10) that E_{01} values for cold-pressed compacts of hydrated portland cement are similar to E_{01} values for hydrated paste, both these values being obtained by extrapolation of log E versus porosity plots. It might have been expected that the compaction technique would produce residual strains in the material and that a modulus of elasticity versus porosity relation different than that for hydrated paste would result. It appears that good bonding can be achieved by compaction and that a porous body can be formed by this technique, where the influence of microcracks, if present, is negligible.

Conclusions

1. For several compositions, moduli of elasticity values of the solid phase of porous bodies, E_{01} , obtained by impregnation with sulphur and use of Reuss' mixing rule equation differ from those obtained by extrapolation of the equation $E = E_{01} \exp(-bP)$ by an average of 13%.

2. The validity of the exponential expression $E = E_{01} \exp(-bP)$ used to calculate modulus of elasticity for a variety of portland cement systems is supported by success of the above comparison.

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