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INTERRELATION OF HARDNESS, MODULUS OF ELASTICITY, AND POROSITY IN VARIOUS GYPSUM SYSTEMS

BY

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RELATION ENTRE LA DURETE, LE MODULE D'ELASTICITE ET LA POROSITE D'ECHANTILLONS DE PLATRE DE TEXTURES VARIEES

SOMMAIRE

Les auteurs ont étudié la relation entre dureté, module d'élasticité et porosité de quatre échantillons de plâtre à textures différentes. Les résultats de leur étude montrent que l'enchevêtrement et l'emboîtement des cristaux semblent influencer notablement leur comportement mécanique, et tout au moins leur dureté et leur module d'élasticité (E). Ces deux caractéristiques sont en relation avec la porosité (P). On peut écrire cette relation sous une forme empirique contenant un terme exponentiel: $E = E_o e^{-kp}$

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Interrelation of Hardness, Modulus of Elasticity, and Porosity in Various Gypsum Systems

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The interrelation of hardness, modulus of elasticity, and porosity was investigated in four gypsum systems. Results suggest that intergrowth and interlocking of crystals significantly affect mechanical behavior, at least hardness and modulus of elasticity, E. In addition, both properties are related to porosity, p. This relation can be described empirically by the exponential expression $E = E_0 \exp(-kp)$.

I. Introduction

The relation between porosity and mechanical properties of porous solids, including gypsum, has been reported in numerous papers.[1-19] Detailed discussion of the available information is outside the scope of the present paper and can be found elsewhere.[20,21] Generally, for otherwise identical conditions, mechanical properties such as strength and modulus of elasticity were related to porosity, with the relation usually taking the form of an exponential expression. For example, $S = S_0e^{-bp}$, where S is the strength of the specimen with porosity p, S_0 is the corresponding strength for zero porosity, and b is an empirical constant related to the type of material and to its pore geometry.[2,7] A similar expression, $E = E_0e^{-kp}$, represents the relation between porosity and modulus of elasticity fairly well.[9-11] Nevertheless, the use of such expressions is sometimes criticized because they are devised on a purely empirical basis and do not satisfy the boundary condition of $E = 0$ (or $S = 0$) for $p = 1$.

Theoretical investigations of the problem have resulted in other expressions describing the porosity-modulus of elasticity relation.[22,23] In general, all the formulas suggested give reasonably good agreement with experimental data; from this point of view a critical evaluation of the various expressions is hardly feasible. Finally, although some attempts have been made to allow quantitatively for pore geometry and particle size in the mechanical properties-pore fraction relation,[3,7,16,18] the problem has not yet been solved satisfactorily.

Tests reported in the present paper were confined to the effect of crystal intergrowth and interlocking on some mechanical properties of a porous solid. This specific aspect was suggested by recent work in which the mechanical properties of portland cement pastes and compacts of bottle-hydrated cement were compared under exposure to different humidities.[24] Studying a system that is distinctly crystalline, and in which intergrowth of crystals during hydration is a possibility, was considered useful. Gypsum is such a system, and was selected for comparison with the cement system.

II. Experimental Procedure

(1) *Preparation of Specimens*

All specimens were flat disks 1.25 in. in diameter and having a nominal thickness of 0.050 in. They were prepared and tested at 50% rh and 22°C. Only one type of plaster was used, pottery plaster (hemihydrate produced by an "aridized" process in which gypsum is heated with traces of calcium chloride). Specimens of five systems were prepared, with as wide a porosity range as is technically possible for the system in question.

System I, In Situ Hydrated Pottery Plaster: The plaster was mixed in vacuum with boiled distilled water to give cylinders 1.25 in. in diameter and approximately 5 in. long. It was mixed in Perspex tubes rotated continuously during the hardening period. This eliminated sedimentation and bleeding phenomena and resulted in highly uniform specimens. After hardening, cylinders were sliced to give specimens 0.050 in. thick. Specimens of five porosities ranging from 50 to 70% were prepared; the corresponding water-plaster ratio (by weight) was 0.64 to 1.36.

Porosity was calculated from the relation of plaster to mixing water.[5,†] Porosity was also determined from the weight and geometrical dimensions of the specimens, assuming complete hydration. This assumption was verified by determining the amount of combined water using the thermobalance procedure. Agreement between the two methods was reasonably good, the maximum difference in the calculated values being only about 2%.

System II, In Situ Hydrated Pottery Plaster Compacted at a Later Stage: After being machined and sliced, some of the in situ hydrated specimens (system I) were compacted in a closely fitting mold (0.003-in. nominal clearance). In each case thickness of the sliced specimens was adjusted to yield the nominal thickness of 0.050 in. after compaction. Porosity, controlled by the pressure applied, ranged from 5 to 49%. Because the initial porosity was known, porosity after compaction was determined simply by the change in the dimensions of the specimen involved.

System III, Compacts of Unhydrated Pottery Plaster: Compacts of hemihydrate were made by the technique described elsewhere,[25] the only change being that a Teflon coating was applied to the compacting surface of the two pistons. This treatment reduced friction and considerably reduced the number of imperfect specimens produced. In fact, the Teflon treatment was critical for system II; otherwise it would hardly have been possible to produce perfect specimens.

Specimens of five porosities ranging from 18 to 42% were prepared. Again, porosity was controlled by the applied pressure and determined from the weight and geometrical dimensions.

System III, comprising a different material (i.e., hemihydrate as opposed to gypsum in the remaining systems), is not directly related to the present investigation and was prepared mainly to facilitate production of system IV. Consequently, although relevant test data are presented in some detail, its properties and performance are discussed hereafter in a limited way only.

System IV, Hydrated Compacts of Pottery Plaster: Some of the hemihydrate compacts (system III) were allowed to hy-

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† The densities of the hemihydrate and dihydrate were taken as 2.76 and 2.31 g/cm³. These values were used throughout the study.

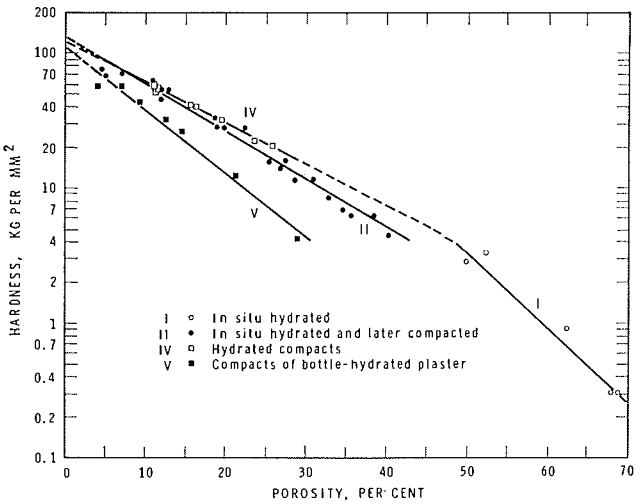

Fig. 1. Hardness vs. porosity.

drate, being exposed to water vapor for 24 hr and then dipped into water for approximately 1 min. Four such cycles of exposure to vapor and immersion in water were made. Complete hydration, however, was not reached and the degree of hydration (calculated from the amount of combined water as determined from the thermobalance test) varied from 80 to 89%, depending on the porosity of the unhydrated compact. This procedure resulted in compacts of eight porosities ranging from 11 to 26%.

System V, Compacts of Bottle-Hydrated Pottery Plaster: The bottle-hydrated plaster was prepared by mixing a 1:10 mixture of plaster and water for 4 hr in a tightly closed bottle mounted on a rotating disk. After being dried at 30% rh, the hydrated plaster was used to prepare compacts of seven porosities ranging from 4 to 29%.

(2) *Tests for Mechanical Properties*

When specimens reached equilibrium at 50% rh and 22°C, two tests were conducted: (1) Vickers hardness was determined with a Leitz Miniload hardness tester, with the load adjusted in each case to give indentation to a depth of 35 to 50μ. Hardness was taken as the average of 10 tests made along an arbitrary diameter at 0.1-in. intervals. (2) Young's modulus of elasticity was determined with an apparatus described elsewhere.[24]

To calibrate this apparatus, the modulus of elasticity in compression of gypsum cylinders 1.25 by 2 in. was compared with the results for deflection of disks cut from the cylinders. The average of five independent tests was considered as Young's modulus for any given set of conditions.

III. Results and Discussion

In Figs. 1 and 2, respectively, hardness and modulus of elasticity are plotted versus porosity, together with the corresponding regression lines. These lines were calculated to fit test data using the least-squares method and assuming a semilogarithmic relation between the factors involved. Such an assumption resulted in fairly good agreement with the experimental data and holds for each of the systems prepared.

Perhaps the most important feature presented in Figs. 1 and 2 is the fact that, although the semilogarithmic relation holds for all systems prepared, in each this relation is represented by a different regression line. This in turn implies that, although hardness and modulus of elasticity are related to porosity, these properties are governed also by additional factors. Under the conditions of this study, when all the systems are of the same material, it may be argued that any difference in their properties can only be attributed to some inherent difference in their structure, such as the particle size and shape, the effective area of contact, or the degree of crystal intergrowth and interlocking.

For the same porosity, specimens of systems I and IV gave the highest values for hardness and modulus of elasticity (Figs. 1 and 2). In both systems the structure is formed by growth of the gypsum crystals from a supersaturated solution, allowing crystals to grow together at points of contact and to interlock on hardening. Under these conditions there can be recrystallization at acute angles of contact, bringing about an increased area of contact. All these effects contribute to the strength of these systems.

Systems I and IV should form the two ends of a common system and be represented by a single regression line for each of the hardness and modulus of elasticity versus porosity relations. The deviation noted in Figs. 1 and 2 suggests that the semilogarithmic relation does not hold over such a wide range of porosity (11 to 70%).

System II specimens exhibited intermediate hardness and modulus of elasticity. It appears that during the formation of this system by compaction the primary structure of system I is progressively destroyed and replaced by a new structure corresponding to that of system V. This is verified by the fact

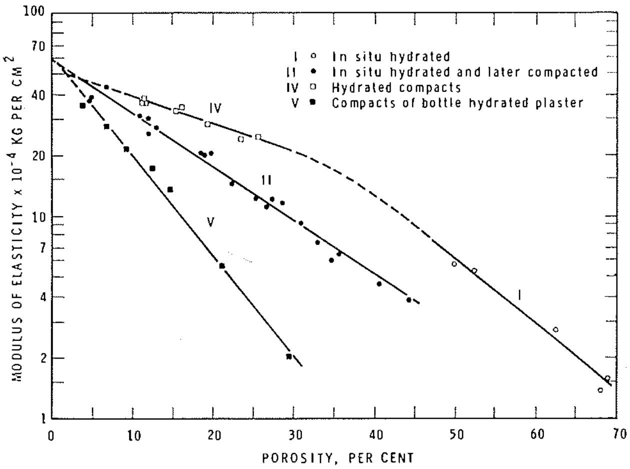

Fig. 2. Modulus of elasticity vs. porosity.

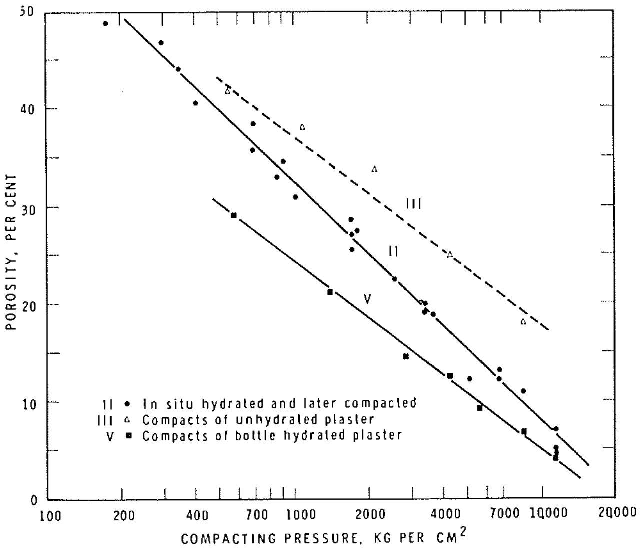

Fig. 3. Porosity vs. compacting pressure.

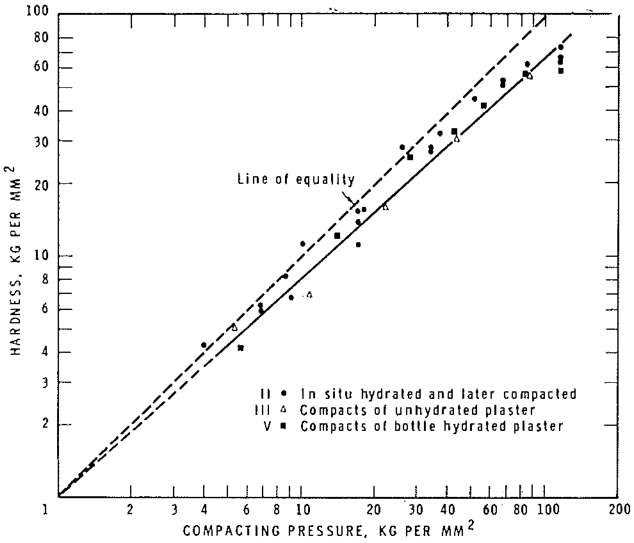

Fig. 4. Hardness vs. compacting pressure.

that the regression lines of systems II and V intersect at approximately zero porosity.

The values for hardness and modulus of elasticity at zero porosity should be the same for systems II and V because they represent the polycrystalline material that would be obtained by compaction at very high pressure. For systems I and IV, however, these values at zero porosity have no practical significance because such a material could not be produced by the hydration reaction.

Figure 3 shows the semilogarithmic relation between porosity and compacting pressure for specimens of systems II, III, and V. Such a relation was found in previous work[24,25] for a variety of materials.

From the semilogarithmic relation between hardness and porosity on the one hand, and porosity and compacting pressure on the other, it follows that a relation should exist between hardness and compacting pressure (Fig. 4). The data indicate that all the systems involving compaction are represented by a single regression line and that this line passes through the axis origin. This implies that the hardness of a specimen depends on the pressure to which it was compacted. It can be explained on the grounds that in a porous material the indentation produced in the hardness test is primarily a localized compaction as opposed to the plastic deformation occurring in a nonporous material.

The deviation of the regression line from the line of equality in Fig. 4 is probably due to the friction between the specimen and the wall of the mold; its value increases with the pressure as would be expected. It appears, therefore, that the compacting pressure can be used to indicate the hardness of specimens made by compaction if the correction for friction could be estimated.

In the foregoing discussion the large differences in hardness and elastic behavior for the same porosities but different methods of preparation were attributed to variations in their structure and identified with crystal intergrowth and interlocking. These features in the structure could be verified by electron microscopy. To this end the fracture surfaces of specimens from the different systems were examined with an EM75 electron microscope (Philips Electronic Equipment Ltd., Toronto, Canada) and a scanning electron microscope.

The electron micrographs in Fig. 5 (*A* through *F*) show a typical structure for each of the systems. The very great dif-

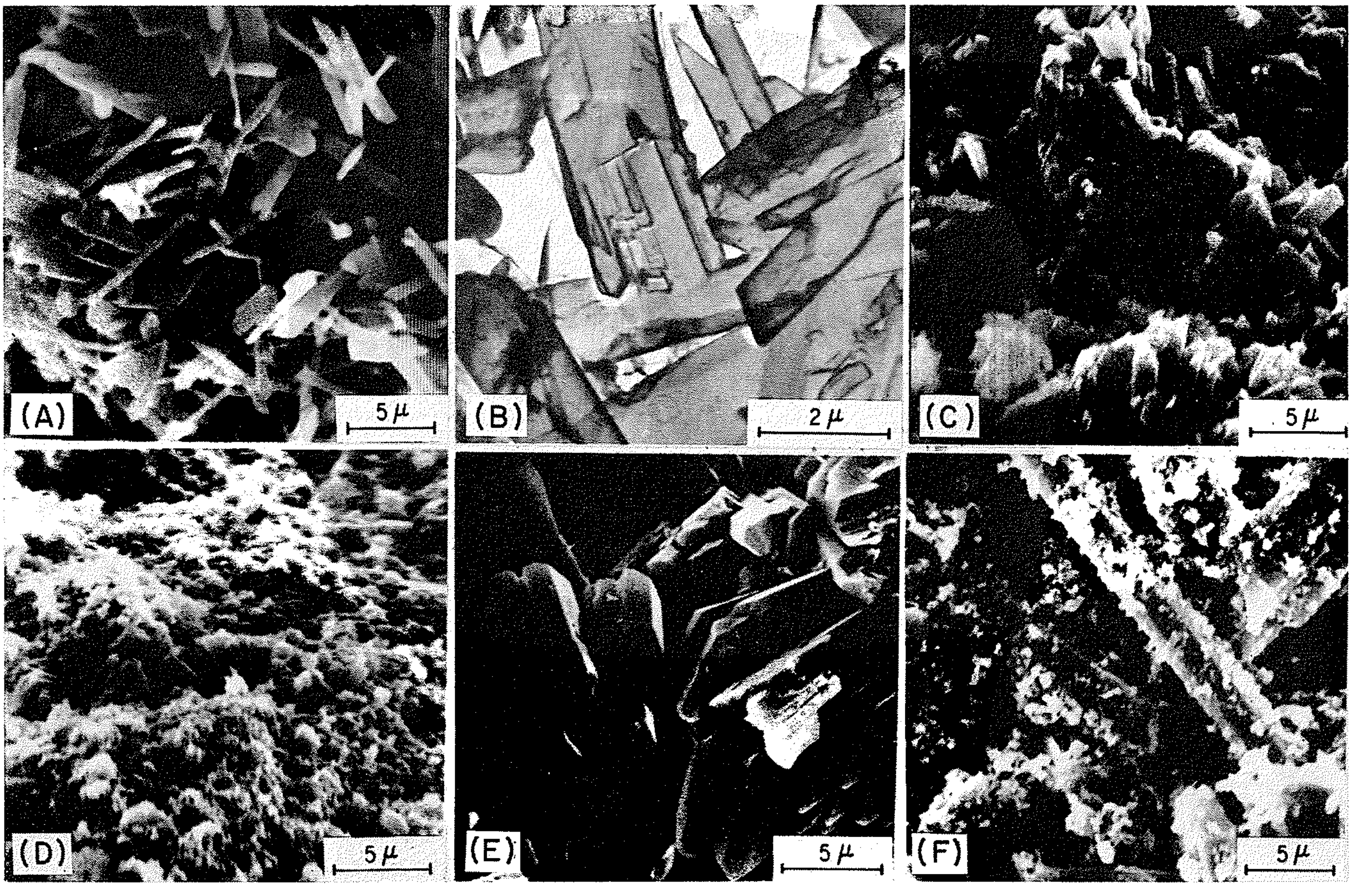

Fig. 5. Surface micrographs of the gypsum systems.

ferences in the physical appearance and character of the structures are apparent. However, the main features that could be definitely identified were the intergrowth of crystals for system I (Fig. 5(*B*)) and the interlocking of crystals for system IV (Fig. 5(*E*)). These features confirm what has been concluded as the reasons for the much higher hardness and modulus of systems I and IV.

The structure of system II (Fig. 5(*C*)) shows evidence for the destruction of the primary structure during compaction. The individual crystals are fragmented and much shorter than they appear in system I (Fig. 5(*A*)) but less disordered and fractured than system V (Fig. 5(*F*)).

Table I. Summary of Test Data Parameters of Regression Lines

	Hardness, H, vs. porosity, p $H = H_0 \exp(-ap)$		Mod. of elasticity, E, vs. porosity, p $E = E_0 \exp(-bp)$	
System	H_0 (kg/mm^2)	a	$E_0 \times 10^{-4}$ (kg/cm^2)	b
I	2117.0	12.85	271.3	7.55
II	133.4	8.08	60.6	6.15
III	395.8	10.35	115.7	7.07
IV	121.3	6.94	52.4	3.06
V	108.9	10.61	63.3	11.44

NOTE: p = pore volume fraction.

IV. Conclusions

Within the limited scope of the present study, several conclusions were reached for a porous polycrystalline solid such as gypsum:

(1) Hardness and modulus of elasticity are related to porosity. This relation can be described empirically in the form of an exponential expression already observed by others for strength[2,7] and modulus of elasticity.[9-11] For present conditions the relevant expressions and parameters established for the various systems are given in Table I.

(2) Intergrowth and interlocking of crystals significantly affect mechanical properties, at least hardness and modulus of elasticity. Depending on porosity, hardness values were as much as four times higher (approximately) and modulus of elasticity up to ten times higher than corresponding values in otherwise identical systems in which crystal intergrowth is not very likely to occur.

(3) Hardness is related to compacting pressure, suggesting the possible use of the latter parameter as a measure of hardness and perhaps some additional properties for porous systems formed by compaction.

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