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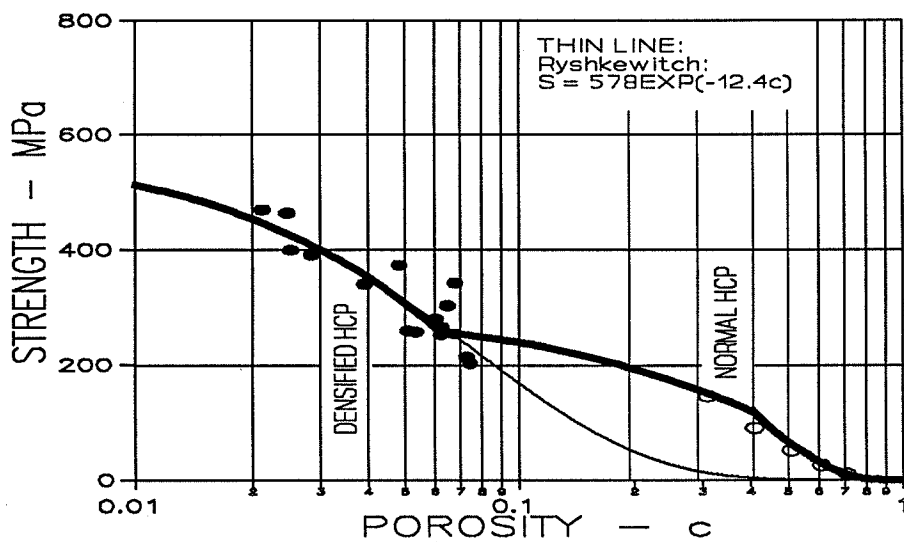
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Strength of porous materials

with special reference to densified systems

Lauge Fuglsang Nielsen



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LAUGE FUGLSANG NIELSEN

Abstract

Some results of the authors previous work in the field of strength and stiffness of porous materials are used in this paper to establish a consistent strength porosity description for porous materials with pores having one type of geometry at low porosities and another type of geometry at higher porosities. Densified pore systems are typical multi porous materials.

The paper is used as a text note in Course 6108 (Porous Building Materials) at the Building Materials Laboratory where strength properties of simple pore systems are considered with only one type of pore geometry at any porosity. Course 6108 students can read the paper with special reference to such materials by simple exclusion of sections where more general pore geometries are considered.

The complete paper is used in a special Course 6110 on Materials Mechanics at the laboratory (Composite materials, Rheology, Strength, and Lifetime).

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1. Introduction and model

The influence of porosity on the mechanical behavior of porous materials have been studied intensively over the last 4 decades. A number of such studies are referenced in (1,2,3) where contributions are also given by the present author on strength and stiffness of porous systems as related to porosity and pore shapes.

Some results from (1,2,3) are used in this research note to illustrate how a consistent strength porosity description can be made for "multi-porous" materials with pores having one type of geometry at low porosities and another type of geometry at higher porosities. Both pore systems mentioned (at higher and lower porosities) are subsequently referred to as simple systems the mechanical behavior of which can be described separately by the relatively simple expressions developed in (1,2,3). The major problem considered in this paper is the following: how does multi-porous materials behave at moderate porosities where geometry changes from one simple system to the other simple system.

Examples of multi-porous systems are porous materials made by powder sintering where special pressure or densification techniques have been used to produce the lower porosity section of the material from "virgin" pore systems with higher porosities. The former pore geometry is considered in this paper as a forced modification of the latter geometry. A transition area of medium porosities exists where pore geometry changes from one to the other.

A number of technical ceramics are expected to yield this materials description. And so is densified hardened cement paste (HCP) where approximately equal strength and stiffness properties can be assumed for the components forming the solid phase, namely cement, bulk gel substance, and added silicon fillers. HCP porosity is total porosity which include both gel pores and capillary pores.

1.1 Notations

The notations and some important functions used in the paper are explained in the following list. Further explanations are not necessarily given in the paper.

| | Geometry |
|---------------------|---|
| V_p, V | pore volume and total volume respectively |
| $c = V_p/V$ | porosity |
| c_d | critical porosity of original simple pore system |
| c_M | transition porosity (type of pore geometry changes) |
| ρ | densification factor |
| $c_{dp} = \rho c_d$ | critical porosity of densified simple pore system |
| μ_o | shape factor of original pore system |
| μ_{op} | shape factor of densified simple pore system |

| | |
|---------------------------------|---|
| $\mu_{ot} = \mu_{ot}(c)$ | shape factor of pore system in transition area between original simple system and densified simple system |
| $\Gamma = \Gamma(c)$ | normalized shape function. |
| $\mu = \mu(c) = \mu_o \Gamma$ | shape function |
| $F = F(c) \approx \Gamma^{-1}$ | interaction function |
| Pore-crack | |
| $l = l(c), l_o = l(0)$ | crack pore radius in general and at $c = 0$ respectively |
| L | bulk radius of pore-crack |
| $\mu_o \approx (L/l)^{2.5}$ | estimate of shape factor |
| Strength (and stiffness) | |
| $S = S(c), S_o = S(0)$ | strength in general and at zero porosity respectively |
| $E = E(c), E_s$ | Young's modulus of porous material and of solid phase respectively |
| S_{RYSH} | Ryshkewitch strength prediction |
| S_{∞} | real solid strength |
| P | crack length variation factor |
| $D = 1 + P/2$ | strength power |

The shape factor (μ_o) and the normalized shape function (Γ) listed above (and illustrated in Figure 3) are introduced into composite stiffness analysis by the author in (1,4). The normalized shape function varies monotonically from $\Gamma = 1$ at zero porosity to $\Gamma \equiv 0$ from a critical porosity (c_d) above which any structural coherence is lost in the porous material considered. Pore shapes are defined by a shape factor $0 \leq \mu_o \leq 1$. Spherical pores and very flat pores have the maximum of $\mu_o = 1$ and the minimum of $\mu_o = 0$ respectively. Fine structured pore systems in ceramic materials and other well graded powder materials for example have $\mu_o \approx 1/4 - 1/3$. More coarsely structured pore systems as in many building materials have greater shape factors.

Structural change by pressure on coarse pore systems will reduce shape factors. Pores are flattened and bulk diameters are reduced of the "pore-cracks" considered in Section 1.2 and illustrated in Figures 1 and 2. Interaction between pores is "controlled" by the interaction function (F) which is approximately equal to the reciprocal normalized shape function (Γ).

Stiffness: It is of some interest to relate stiffness and strength of porous materials. So-called MOE-MOR relations are useful in non-destructive testing (Modulus of elasticity - Modulus of rupture). The MOE-MOR subject is not pursued in further details in the paper. It is left to the reader himself to do some exercises in this area. The appropriate stiffness expression to be used is presented in Equation 1 reproduced from (1,5).

$$\frac{E}{E_s} = \mu \frac{1 - c}{\mu + c} \quad (1)$$

1.2 Pore-crack

Theoretical strength expressions subsequently referred to are based on the materials concept that a pore system can be modelled as an isotropic mixture of spherical and cylindrical pore-cracks of the kind outlined in Figures 1 and 2 with crack diameters l . Pore characteristics of pore-crack systems are modelled by round figures in Figure 2.

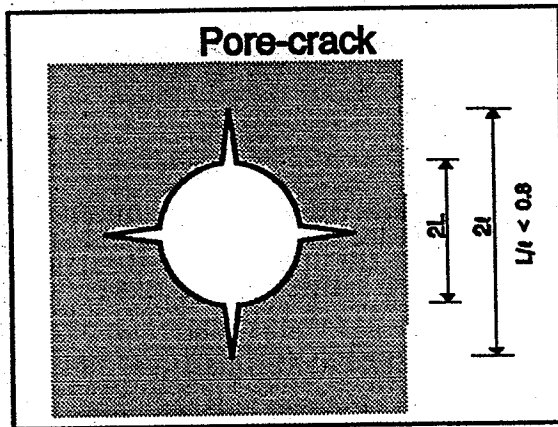


Figure 1. Pore-cracks are spherical or cylindrical voids with co-centric cracks crossing each other at 90° . "Hybrid" pore-cracks have shape factors of $\mu_o \approx (L/l)^{2.5}$.

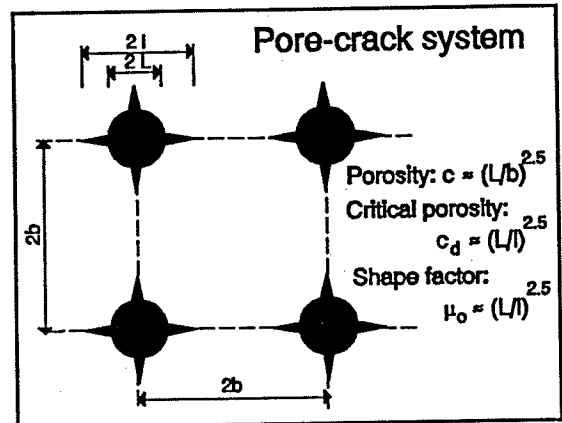


Figure 2. Porous material is modelled as an isotropic mixture of pseudo-cubic elements with hybrid pore-cracks. Pore characteristics (c , c_d , μ_o) are modelled by "round" figures.

1.3 Simple pore systems

A simple (or virgin) porous material is a material with pores made without any internal or external restraint. Such systems have simple shape functions as presented in Equation 2 and illustrated in Figure 3. Corresponding stiffness predicted by Equation 1 is shown in Figure 4.

$$\Gamma = 1 - c/c_d \quad ; \quad \mu = \mu_o \Gamma \quad (2)$$

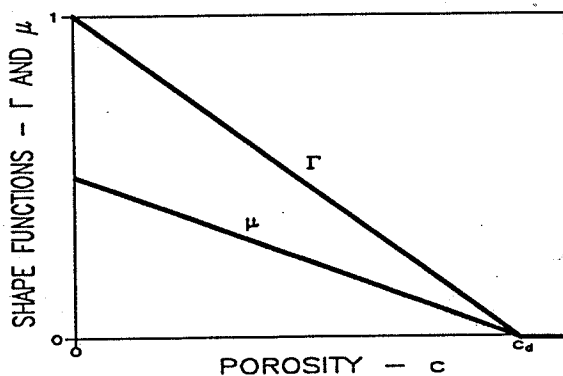


Figure 3. Shape functions for simple pore system: $(c_d, \mu_o) = (0.9, 0.5)$.

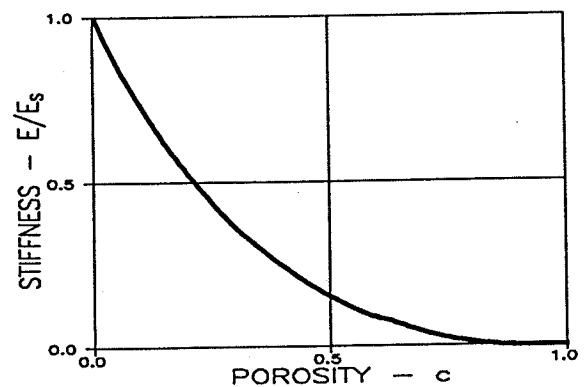


Figure 4. Stiffness of simple pore system defined in Figure 3.

1.4 Densified pore systems

A densified pore system is considered as a combination of two simple pore systems. The higher porosity section is the original (virgin) pore system from which the lower porosity material has been produced by compression such that voids volume has become smaller, meaning that the bulk diameter and the shape factor of the pore-crack model in Figures 1 and 2 become smaller.

A *normalized shape function* (Γ) of the type in Equation 3 and illustrated in Figure 5 is suggested in this paper to represent densified pore systems. Concentration c_{dp} defines the Γ -trend of the densified simple pore system. Concentration c_x defines the end of this system. The transition porosity c_M defines the end of pore type transition between pressed and original pore system.

At the same time a *shape factor function* is introduced as shown in Equation 4 which is concentration dependent in the transition area previously defined. The shape factor of the densified simple pore system is obtained from information in Figure 2 on pore-crack systems.

The *shape function* (μ) of the pore system considered is obtained as shown in Equation 5 by multiplication of normalized shape function with shape factor.

Stiffness of the pore system considered is predicted by Equations 1 with shape function from Equation 5. An example is shown in Figure 6.

$$\Gamma = \begin{cases} \text{MAX} \left(1 - \frac{c}{c_{dp}}, 1 - \frac{c_M}{c_d} + \beta(c_M - c) \right) & \text{when } c < c_M \\ 1 - \frac{c}{c_d} & \text{when } c \geq c_M \end{cases} \quad (3)$$

$$\beta = \tan[\text{artan}(1/c_d) - \text{artan}(c_{dp})]$$

$$\mu_o(c) = \begin{cases} \mu_o & ; c \geq c_M \quad (\text{original simple system}) \\ \mu_{oi} = \mu_{op} + \frac{c - c_x}{c_M - c_x}(\mu_o - \mu_{op}) & ; c_x < c < c_M \quad (\text{transition system}) \\ \mu_{op} = \frac{c_{dp}}{c_d} \mu_o & ; c \leq c_x \quad (\text{densified simple system}) \end{cases} \quad (4)$$

$$c_x = c_M \frac{\beta - 1/c_d}{\beta - 1/c_{dp}}$$

$$\mu = \mu_o(c)\Gamma(c) \quad \text{from Equations 3 and 4} \quad (5)$$

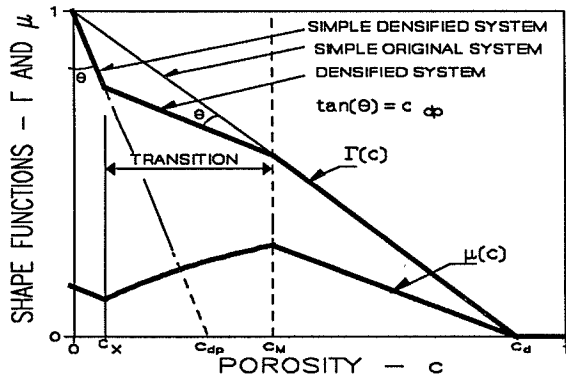


Figure 5. Shape functions for densified pore system: $(c_d, c_{dp}, c_M) = (0.9, 0.27, 0.4)$. $\mu_o = 5$.

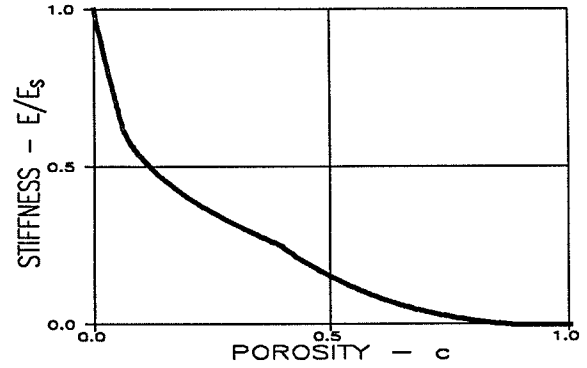


Figure 6. Stiffness of densified pore system defined in Figure 5.

2. Strength

The strength-porosity relation presented in Equation 6 and summarized in Equation 7 is reproduced from (1) where simple porous materials are modelled by pore-crack systems. It is suggested that this expression also applies when densified pore systems are considered with shape functions given by Equation 3. The symbols F and P in Equation 6 denote interaction factor and crack length variation factor respectively. D in Equation 7 is strength power.

$$\frac{S}{S_o} = \sqrt{\frac{l_o}{l}} \frac{1}{F} \quad \text{with} \quad F = \text{EXP}\left(\frac{\Gamma^2 - 1}{2\Gamma}\right) \approx \frac{1}{\Gamma} \quad \text{and} \quad \frac{l_o}{l} \approx \Gamma^P \quad (6)$$

$$\frac{S}{S_o} \approx \Gamma^D \quad \text{with} \quad D = 1 + \frac{P}{2} \quad (7)$$

It is important to notice that reference strength S_o is strength of a solid phase weakened by single pore-cracks of lengths l_o (representing no volume). The real solid phase strength can be estimated by crack mechanics (fx 6): $S_{\infty} \approx S_o \sqrt{l_o/d}$ where d is a characteristic microstructural dimension of the solid phase considered. When solid is a crystalline material, $d \approx$ distance between atoms.

Well-known fit-expressions from the literature on strength behavior of porous materials are recognized as special versions of Equation 7 as it applies when simple pore systems are considered with $\Gamma = 1 - c/c_d$: $S/S_o = (1 - c)^B$ and $S/S_o = 1 - c/c_o$, were suggested by Balshin (7) and Hasselman (8) respectively with constants B and c_o . Also the Ryshkewitch expression $S_{\text{RYSH}} = S_o \text{EXP}(-Ac)$ (9) with constant A is consistent with Equation 7 at low porosities and $c_d = 1$.

2.1 Deduction of pore parameters

Zero-porosity strength (S_o) and strength power ($D = 1 + P/2$) can be deduced from experimental strength data by the linear regression procedure explained in Equations 8. Shape functions (Γ) are from Equation 3 (or Equation 2, simple system). Densification factor ρ ($= c_{dp}/c_d$) and transition porosity c_M are determined by optimizing the fit quality (r^2) of regression. The critical porosity c_d is considered known from similar regressions of stiffness data (5). A transition porosity of $c_M = 0$ applies when simple pore systems are considered.

Equation 7 linearized: $Y = Y_o + \alpha X$ with

$$X = \log\Gamma \quad \text{and} \quad Y = \log S \quad (8)$$

From intersection and slope: $S_o = EXP(Y_o)$; $D = \alpha$

3. Examples

The strength expressions presented in the preceding sections are evaluated in this section by testing their abilities to describe consistently some experimental data reproduced from the literature. The results are presented graphically in Figures 7 to 12 where experimental data and theoretical results are indicated by dots and lines respectively. The "elbows" on the latter data are due to the mathematical simplifications of the shape function suggested in Equation 3. In general results around sharp bendings should be smoothed out.

For simplicity a constant critical porosity of $c_d = 1$ has been used all over. Zero porosity strength (S_o) and strength power (D) are determined by regression using the method presented in Section 2.1.

The figures speak for themselves. Some brief explanations and comments, however, are appropriate:

Figure 7: Compressive strength of an autoclaved Calcium silicate (CaSi) system. Experimental data are from (10). From fabrication technique described in (10) is concluded that the material considered behaves as a simple pore system.

Figure 8: Compressive strength of compacted HCP. Experimental data are from (11). It is estimated that the compaction technique used in (11) leaves the material considered as a simple pore system.

Figure 9: Bending strength of porous Aluminum Oxide (Al_2O_3) at 25°C. Experimental data are from (12).

It is noticed that zero porosity strength is underestimated by the Ryshkewitch fit $S_{RYSH} = S_o EXP(-Ac)$ often used to estimate this quantity. The reason is that the range of

porosities considered experimentally exceeds very much the low porosity area where the fit method becomes accurate. This has previously been discussed in Section 2.

Figure 10: Bending strength of porous Silicon Nitride (Si_3N_4). Experimental data are from (13) where a number of data are compiled from 6 different sources. The data presented in the figure are obtained at a test temperature of 20°C .

S_o is underestimated by the Ryshkewitch fit method for the same reasons as mentioned in previous example.

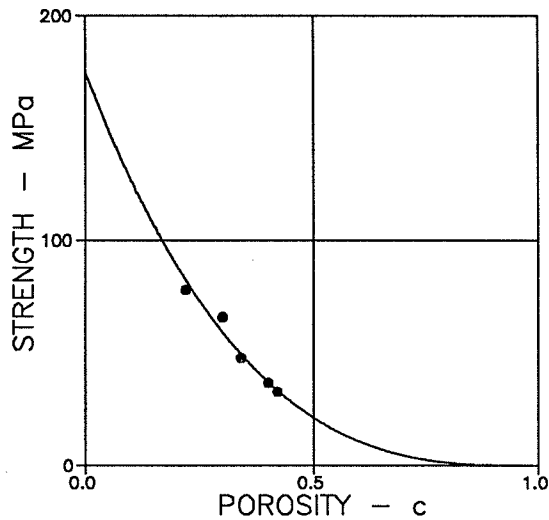


Figure 7. Compressive strength of autoclaved calcium silicate system (10). Simple pore system with $c_d = 1 \Rightarrow 175 \text{ MPa}$ og $D = 3.03$.

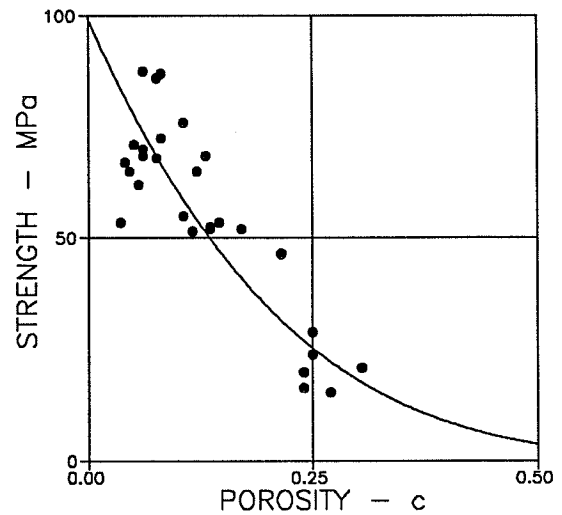


Figure 8. Compression strength of compacted HCP (11). Simple pore system with $c_d = 1 \Rightarrow 99 \text{ MPa}$ og $D = 4.74$.

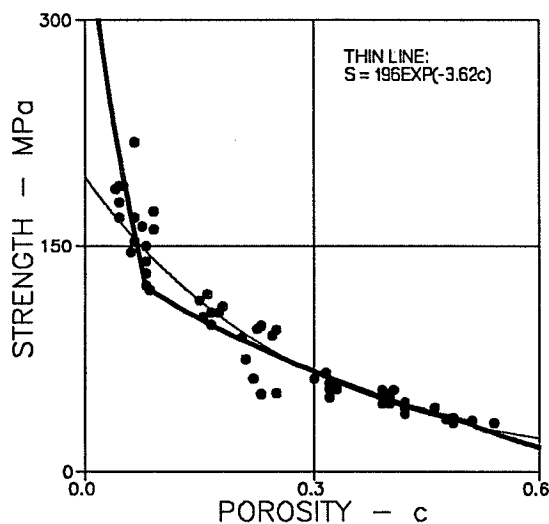


Figure 9. Bending strength of porous aluminum oxide (12). $(c_d, c_M, \rho) = (1, 0.5, 0.3) \Rightarrow D = 3.44$ and $S_o = 369 \text{ MPa}$.

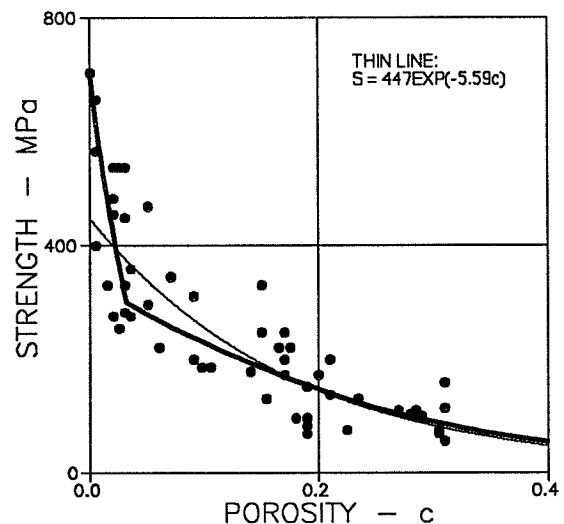


Figure 10. Bending strength of porous Silicon nitride (13). $(c_d, c_M, \rho) = (1, 0.4, 0.2) \Rightarrow D = 5.07$ and $S_o = 710 \text{ MPa}$.

Figure 11: Compression strength of hot-pressed HCP with water-cement ratios (by weight) of $W/C = 0.093 - 0.102$. Experimental data are from (14). The special pressure procedure used (pressure < 540 MPa, pressure time < 2 hours, temperature < 250 °C) is not suspected to change very much the chemical composition of the solid phase relative to that of normally hydrated HCP. The data shown refer to water curing in 28 days after hot-pressing. Normal HCP data (circles) are added to the figure as predicted in (2) with 80 % of cement hydrated (relative to amount of cement which can be hydrated) $\Rightarrow S = 450(1 - c)^{2.94}$. These added data are not included in the regression procedure.

The bend of the strength-porosity graph at $c \approx 6$ % fits well with qualitative expectations one may have concerning the strength behavior at pressed HCP (with no fillers) at low water-cement ratios. The porosity of the virgin pore system is close to the minimum packing porosity of loose cement grains which means that a self optimizing and strength improving powder size grading will take place during the process of pressing.

The strength variation in the porosity area $c \approx 0.3 - 0.1$ is very modest. It seems justified to ask if there is any variation. Does it pay to use pressure techniques in this area of porosities, or do such treatments reduce strength which would have developed without pressure.

The experimental data from the hot-press experiments are well covered by a Ryshkewitch fit because all data refer to low porosities where the fit becomes an accurate strength description. A Ryshkewitch fit covering the whole range of porosities considered in Figure 11 is very poor.

Figure 12: As in Figure 11. But data include results from specimens which have been water cured in 90 days after hot-pressing.

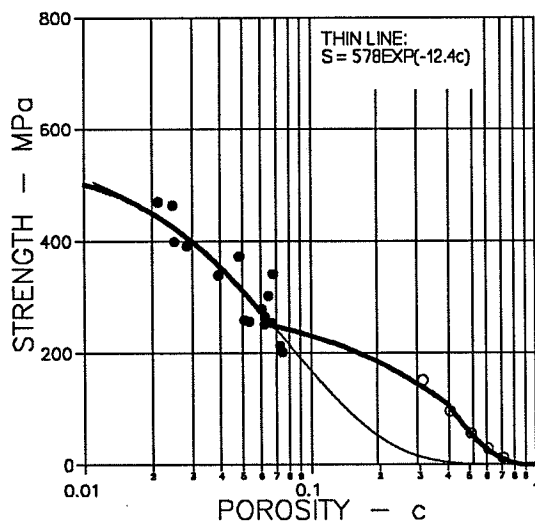


Figure 11. Compressive strength of hot-pressed HCP (14). 28 days after curing in water. $(c_d, c_M, \rho) = (1, 0.4, 0.3) \Rightarrow D = 3.26$ and $S_o = 562$ MPa.

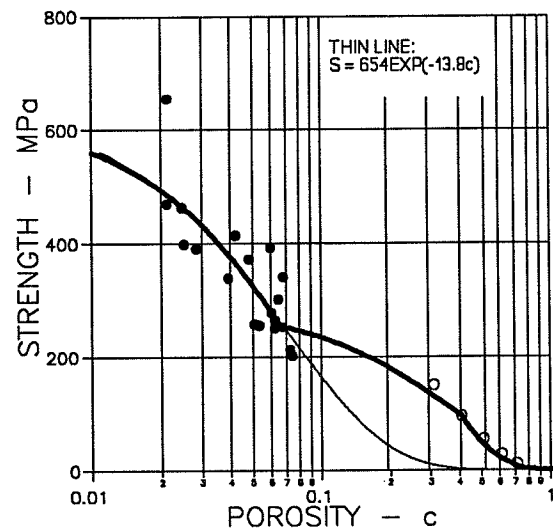


Figure 12. Compressive strength of hot-pressed HCP (14). 28 and 90 days after curing in water. $(c_d, c_M, \rho) = (1, 0.4, 0.3) \Rightarrow D = 3.64$ and $S_o = 633$ MPa.

4. Conclusions and final remarks

The ideas presented in Sections 1.3, 1.4, and Section 2 on strength prediction of simple porous materials and multi porous materials as related to type of pore geometry seem to be well justified by the experimental data presented in Section 3. This means that strength of porous materials can be predicted by Equation 7 with change of pore geometry considered by pore specific normalized shape functions as defined in Equation 3 and illustrated in Figure 5. Of special interest are densified pore systems where shape functions at low porosities are considered as simple modifications of simple shape functions at higher porosities.

The strength-porosity relations predict strength to vary as normalized shape function (Γ) raised to a certain power D which depends on crack length variation with respect to porosity. The magnitudes of D observed in the examples considered are $D \approx 3 - 5$.

Simple fit expressions suggested by Ryshkewitch (9), Balshin (7), and Hasselman (8) in the literature on strength of porous materials are recognized as special versions of the theoretical expressions presented in the paper. It is indicated that these expressions are not very reliable when used on densified porous materials.

Finally, it is of interest to relate stiffness and strength of porous materials. So-called MOE-MOR relations are useful in non-destructive testing (Modulus of elasticity - Modulus of rupture). How such relations can be obtained are indicated in Section 1 of the paper.

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