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Strength and elastic modulus of a porous brittle solid: an acousto-ultrasonic study

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The strength and elastic modulus of a porous brittle solid such as gypsum have been studied using an acousto-ultrasonic technique. Acousto-ultrasonics has been found to be a sensitive indicator of strength and porosity which are linearly related to some powers of a stress wave factor. New equations for porosity dependence of ultrasonic velocity, elastic modulus and strength of brittle solids have been proposed.

1. Introduction

Acoustic emission and ultrasonics are widely used techniques for the study of microstructural defects and variation, strength and fracture of solids. Pulse-echo ultrasonics generally detects isolated flaws, but because of signal attenuation, no meaningful interpretations of the condition of the material can be made from the response of ultrasonic waves injected into it. In the acoustic emission technique, the material is put under stress and the spontaneous emission of acoustic signals is recorded for the detection of the growth of flaws induced by applied stress. Correlation of microflaws to the strength and fracture characteristics of materials, therefore, often becomes difficult by both ultrasonic and acoustic emission techniques. The limitations of the two techniques have been overcome by combining them and developing a new technique called acousto-ultrasonics. This non-destructive testing (NDT) technique has been developed at NASA, Lewis Research Centre, Ohio, USA, for the inspection of composite and adhesive-bonded structures [1]. The technique operates by injecting a repeating series of discrete ultrasonic waves into the material and then sensing and measuring the simulated stress waves by an acoustic emission transducer. The detected wave form is measured in terms of a stress wave factor (SWF) which essentially indicates the efficiency of stress wave energy transmission in the material and is defined as

$$\varepsilon = GRN$$

where ε is the stress wave factor, N the number of threshold crossings generated by each burst, R the pulser repetition rate, and G the pre-determined time interval. The stress wave factor depends on a number of parameters such as signal gain, reset time, threshold voltage, pulser repetition rate, specimen shape and size, probe spacing, probe pressure, etc.

Since its development, acousto-ultrasonics has found wide applications mostly in fibre composites and adhesive bonded materials [2–6]. Acousto-ultrasonic measurements are strongly correlated with the strength properties, mechanical damage and porosity of such materials. In this paper an acousto-ultrasonic study of the strength and elastic modulus of a porous brittle solid such as gypsum has been made, correlatiing strength with the stress wave factor and ultrasonic velocity with porosity. Equations showing the porosity dependence of the elastic modulus, strength and stress wave factor have also been derived.

2. Experimental techniques

Specimens used in the experiment were prepared from pottery plaster. The specimens were 26 mm cubes and 130 mm × 13 mm × 7 mm bars. Specimens of five different porosities, 46.87, 49.50, 51.89, 54.05 and 56.03%, were prepared by mixing the plaster with 60 to 80% boiled distilled water. After hardening for 48 h, they were dried at 40° C to constant weight. Porosity was calculated from Schiller's relation [7]

$$P = \frac{w - 0.15}{w + 0.36}$$

where P is volume fraction porosity and w, the water–plaster ratio. Taking the density of calcium sulphate dihydrate as 2.31, the porosity was also calculated from the weight and volume of the specimens assuming complete hydration. There was reasonably good agreement between the values obtained by the two methods, the maximum difference being less than 1.8%. Soroka and Sereda [8] also found close agreement between the porosity values of gypsum obtained by the two methods.

Acousto-ultrasonic measurements for the stress wave factor and ultrasonic velocity were performed using an AET 206 AU, Acoustic Emission Technology corporation, USA, using gypsum cubes. The equipment consists of an ultrasound pulsing section, an acoustic emission processing section, an oscilloscope and a digital read-out section that displays the stress wave factor, signal level and the threshold voltage. In our experiment, the stress wave factor was measured by placing the ultrasonic transducer and the AE transducer on two parallel faces of the gypsum cube. The measurements were done in the pulse mode with a trigger rate of 1000 p.p.s., gate width 93.75 μsec, gain 45 dB, rate 0.5 sec, threshold voltage 1.04 V and scale 100. The transducers were coupled to the specimen

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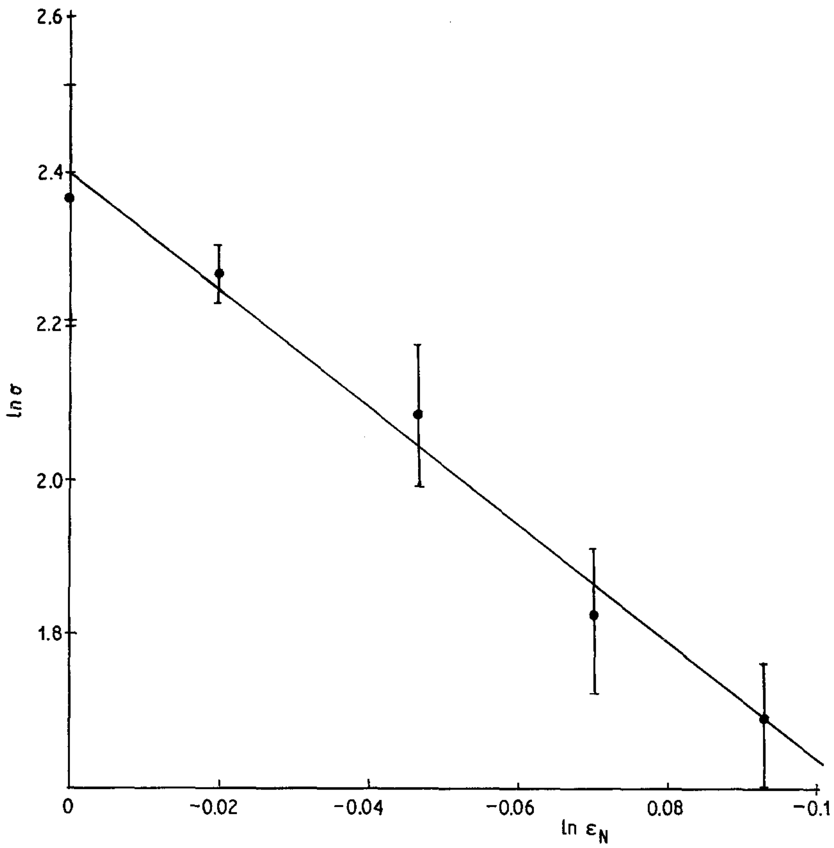

Figure 1 Plot of ln σ against ln ε_N (confidence limit $p = 0.05$).

with AET couplant SC6. The ultrasonic velocity was measured from the spacing of the transducers and the waveform time delay on the oscilloscope. Compressive strength and Young's modulus were determined using an Instron 1195. For compressive strength, gypsum cubes were tested at a crosshead speed of $2\,\text{mm}\,\text{min}^{-1}$. Flexural tests of bar specimens were carried out in three-point bending with a span of 100 mm and a crosshead speed of $0.5\,\text{mm}\,\text{min}^{-1}$. Young's modulus was calculated from the load–deflection curve using the relation $E = Wl^3/4\Delta bt^3$ where E is the Young's modulus, l, the loading span, b the width and t, the thickness of the specimen and Δ the deflection at midpoint due to load W. Young's modulus was also calculated from the ultrasonic velocity using the relation

$$E = V_L^2 \varrho$$

where V_L is the ultrasonic velocity and ϱ is the density of the material. The data on acousto-ultrasonic and mechanical properties of gypsum reported in the paper are based on the average of eight tests.

3. Results and discussion

The log–log plot of compressive strength, σ, against normalized stress wave factor, ε_N ($\varepsilon_N = \varepsilon/\varepsilon_{max}$) is shown in Fig. 1. The plot yielded a strong correlation between σ and ε_N (coefficient of correlation 0.99). The regression line is given by the equation

$$\sigma = K\varepsilon_N^C \qquad (1)$$

where K and C are empirical constants whose values were found to 11.018 and 7.644, respectively. With decrease of porosity, flaw density decreases, consequently leading to an increase of the efficiency of stress wave energy propagation as well as the compressive strength of the material. Hence σ increases monotonically with ε_N. Such correlation between stress wave factor and tensile or shear strength was also found in fibre composites by previous workers [2, 4].

The relation between ultrasonic velocity, V_L, and porosity, P, is shown in Fig. 2. The linearity of the plot $\ln V_L$ against $\ln(1 - P)$ shows that ultrasonic velocity and porosity are correlated by the equation

$$V_L = V_{L0}(1 - P)^m \qquad (2)$$

where V_{L0} is the ultrasonic velocity at zero porosity and m, an empirical constant. The values of V_{L0} and m were evaluated by an iterative least square method given by Lewis [9] and found to be $4250\,\text{m}\,\text{sec}^{-1}$ and 1.009, respectively.

Since $V_L = (E/\varrho)^{1/2}$ and $\varrho/\varrho_0 = 1 - P$, where ϱ_0 is the density at zero porosity, Equation 2 can be transformed to

$$E = E_0(1 - P)^n \qquad (3)$$

E_0 being the Young's modulus at zero porosity and $n = 2m + 1$. Equations 2 and 3 satisfy the boundary conditions $V_L = V_{L0}$ and $E = E_0$ for $P = 0$, and $V_L = 0$ and $E = 0$ for $P = 1$. Since $m = 1.009$, it is apparent from Equation 3 that a plot of $\ln E$ against $\ln(1 - P)$ for gypsum should yield a value of $n = 3$. A plot of $\ln E$ against $\ln(1 - P)$ for values of Young's modulus of gypsum obtained by the flexural test, and those calculated from ultrasonic velocity, is shown in Fig. 3. The data satisfied Equation 3 well, having correlation coefficients of 0.991 and 0.995, respectively. The values of E_0 were found to be 32.34 and 35.87 GPa and those of n 2.73 and 2.86, respectively (close to the expected value of $n = 3.018$).

A number of equations have been proposed to relate Young's modulus to porosity of polycrystalline

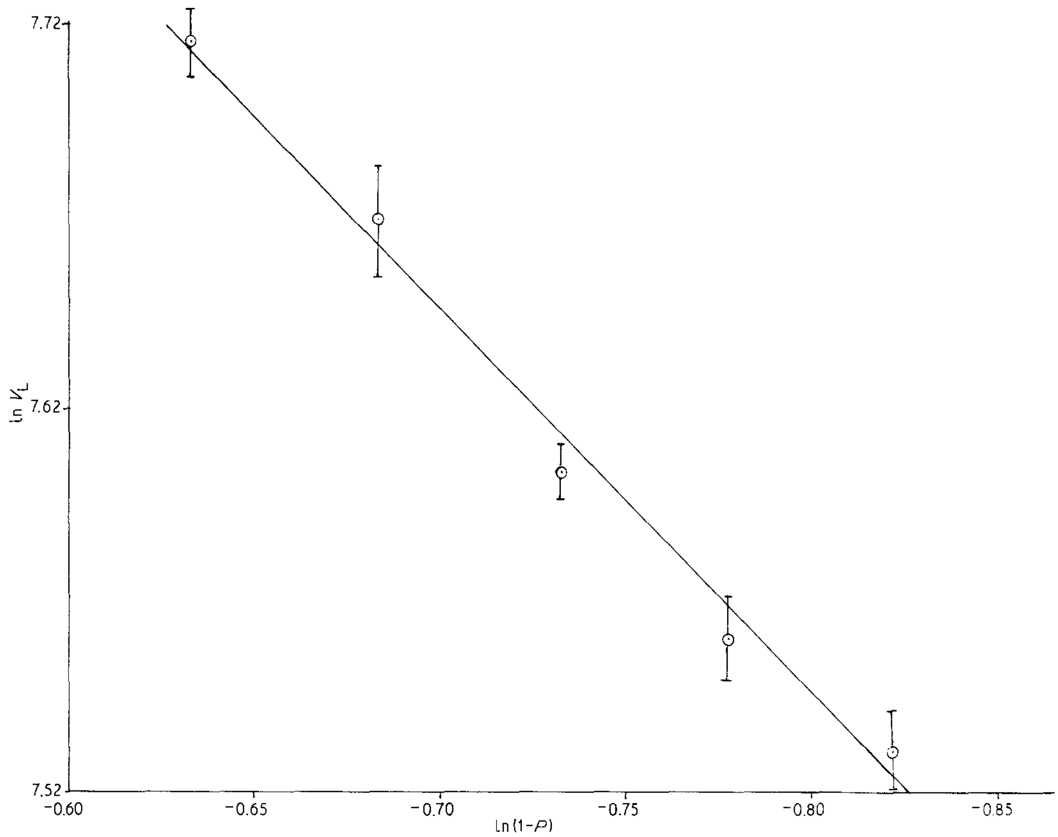

Figure 2 Plot of ln V_L against ln $(1 - P)$ (confidence limit $p = 0.05$). $V_L = 4250\ (1 - P)^{1.009}$.

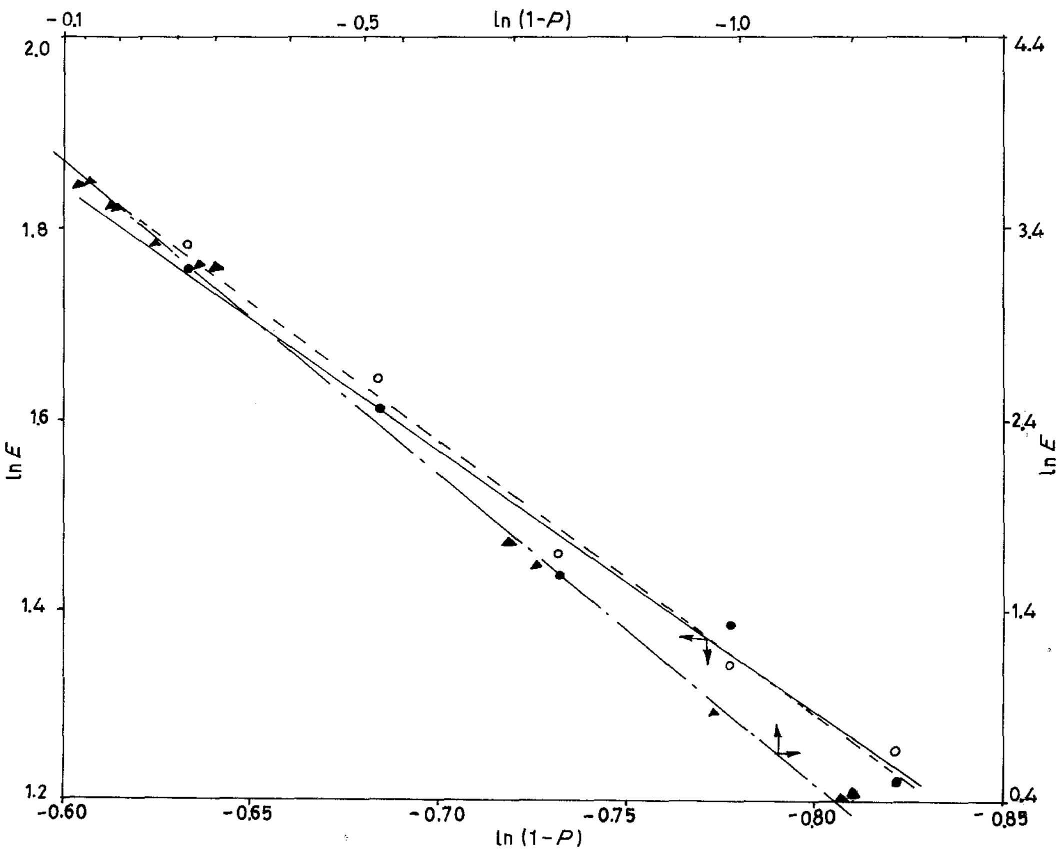

Figure 3 Plot of ln E against ln $(1 - P)$. (●) $E = 32.34(1 - P)^{2.73}$ flexural modulus (present study), (○) $E = 35.87(1 - P)^{2.86}$ velocity of sound Young's modulus (present study), (▲) $E = 56.50(1 - P)^{3.20}$ (Soroka and Sereda [7]).

brittle solids [10–14]. Of these, the exponential equation

$$E = E_0 \exp(-aP) \quad (4)$$

and the linear equation

$$E = E_0 (1 - hP) \quad (5)$$

where a and h are empirical constants depending on the pore geometry of the material, are widely used since they provide minimum standard error of estimate of fit. An empirical equation on a physical phenomenon should satisfy the boundary conditions. From this point of view the limitations of Equations 4 and 5 are evident. Putting $P = 1$ in both the equations does not yield the condition $E = 0$ as required. This will affect the values of E_0, a and h obtained from experimental data. Because of these limitations the equations do not always show reasonable agreement with experimental data over a wide range of porosity. Soroka and Sereda [8] studied the porosity dependence of Young's modulus of gypsum using Equation 4, and found that the equation does not hold good over a wide range of porosity. For a common gypsum system (I and IV) they obtained widely different values of E_0 over different porosity ranges. The value of E_0 was 51.4 GPa over the porosity range $0.11 < P < 0.30$ and that over the range $0.49 < P < 0.70$ was 265.9 GPa. We also fitted the data of the present investigation on gypsum over the porosity range $0.45 < P < 0.57$ to Equation 4 and obtained a very high value of $E_0 = 97.5$ GPa. The data of Soroka and Sereda [8] on gypsum over the entire range of porosity $0 < P < 0.7$ was fitted to Equation 3 (Fig. 3). The plot of $\ln E$ against $\ln(1 - P)$ gave an excellent fit with a correlation coefficient of 0.999, the values of E_0 and n being 56.5 GPa and 3.20, respectively. This indicates that Equation 3 holds good over a wide range of porosity and does not have the limitations of Equation 4.

The data on the other two gypsum systems (II and V) reported by Soroka and Sereda [8] also showed excellent agreement with Equation 3, the correlation coefficients being 0.993 and 0.997, respectively. The values of E_0 were 51.8 and 53.9 GPa, and those of n were 4.75 and 9.57, respectively. The various test data parameters of gypsum are given in Table I. It can be seen from the table that the values of E_0 of all the systems are similar, but those of n are widely different. This is due to the fact that the plaster used in all the systems was same, while the methods of preparation were different. Electron micrographs of the specimens of the common system (I and IV) showed intergrowth and interlinking of crystals, whereas those of systems II and V showed fragmented and shorter crystals with interconnected pores caused by destruction of the primary structure during compaction, the disorder and fracture being greater in system V. With increasing porosity this led to a larger decrease in Young's modulus at the same porosities thus causing the increase of the value of n from 3.20 to 9.57. This clearly shows that n is a microstructure-dependent parameter, the value of which possibly lies between 2 and 4 for a relatively ordered and less-open pore structure and higher for disordered and interconnected pore structure. Sintered glass [15] with spherical pores has been found to yield a value of $n = 2.1$. The dependence of n on crystal morphology as well as pore geometry is being investigated and will be reported in subsequent papers.

Like elastic modulus, the strength of porous brittle solids is known to be exponentially related to porosity by the equation

$$S = S_0 \exp(-fP) \quad (6)$$

where S and S_0 are the strengths at porosity P and zero, respectively, and f is an empirical constant [16, 17]. As in Young's modulus, the data on compressive strength obtained in the present investigation as well as those reported by Schiller [7], on fitting to Equation 6, yielded very high values of σ (444.5 and 1449.3 MPa, respectively) indicating again that the exponential relation does not hold good over the range of porosity ($0.46 < P < 0.66$) studied. The linearity of the plots of $\ln \sigma$ against $\ln(1 - P)$ of both the data (correlation coefficients 0.987 and 0.999, respectively) (Fig. 4) shows that the porosity–compressive strength relation can be expressed in the form

$$\sigma = \sigma_0 (1 - P)^q \quad (7)$$

where q is an empirical constant. This equation also obeys the boundary condition $\sigma = \sigma_0$ at $P = 0$ and $\sigma = 0$ at $P = 1$. The values of σ_0 and q obtained for gypsum investigated by the present authors are 123 MPa and 3.79, while those obtained from the data of Schiller [7] are 173.6 MPa and 3.76, respectively. Balshin [18] earlier derived this empirical relation for sintered copper powder and observed that the value of q varies from 3 to 6. However, we have found a value of $q = 15.1$ for briquette coal (H. Seam). We intend to study the applicability of Equation 7 to various porous brittle solids and the variation of q with microstructure. On combining Equations 1 and 7, porosity can be correlated with stress wave factor by the equation:

$$P = 1 - \lambda \varepsilon_N^{\mu} \quad (8)$$

where $\lambda = 0.53$ and $\mu = 2.02$.

Fig. 5 shows the graphical representation of Equation 8 along with the experimental data. The data agree well with the empirical equation. Equation 8, on differentiation, gives $dP/d\varepsilon_N = -1.071\varepsilon_N^{1.02}$. ε_N varies from 0.911 to 1 over the range of porosity studied ($0.45 < P < 0.57$). Hence $dP/d\varepsilon_N \simeq -1$ for $0.911 \leqslant \varepsilon_N \leqslant 1$. The plot in Fig. 5, therefore, appears to be linear with a slope of about 135°.

4. Conclusion

Acousto-ultrasonics is a versatile technique for the study of microstructural defects and variations. The strength and elastic modulus of a porous brittle solid such as gypsum have been studied by the acousto-ultrasonic method. The important observations are:

1. Stress wave factor is a sensitive indicator of strength and porosity which are related to the stress

TABLE 1 Test data parameters of gypsum

	System	Porosity range	V_{L0} (m sec^{-1}), $V_L = V_{L0}(1 - P)^m$	m	E_0 (GPa)		n	σ_0 (MPa)		q
					$E = E_0(1 - P)^n$	$E = E_0 \exp(-aP)$		$\sigma = \sigma_0(1 - P)^q$	$\sigma = \sigma_0 \exp(-hP)$	
Present work	–	0.46–0.57	4250	1.009	32.3*	97.5	2.73	123.0	444.5	3.79
					35.9†		2.86			
Soroka and Serada [8]	I } common system	0.11–0.27	–	–	56.5	265.9	3.20	–	–	–
	IV } common system	0.49–0.70	–	–		51.4	–	–	–	–
	II	0.05–0.45	–	–	51.8	59.4	4.75	–	–	–
	V	0.03–0.30	–	–	53.9	62.0	9.57	–	–	–
Schiller [7]		0.53–0.66	–	–	–	–	–	173.6	1449.3	3.76

*From flexural test.
†From ultrasonic velocity.

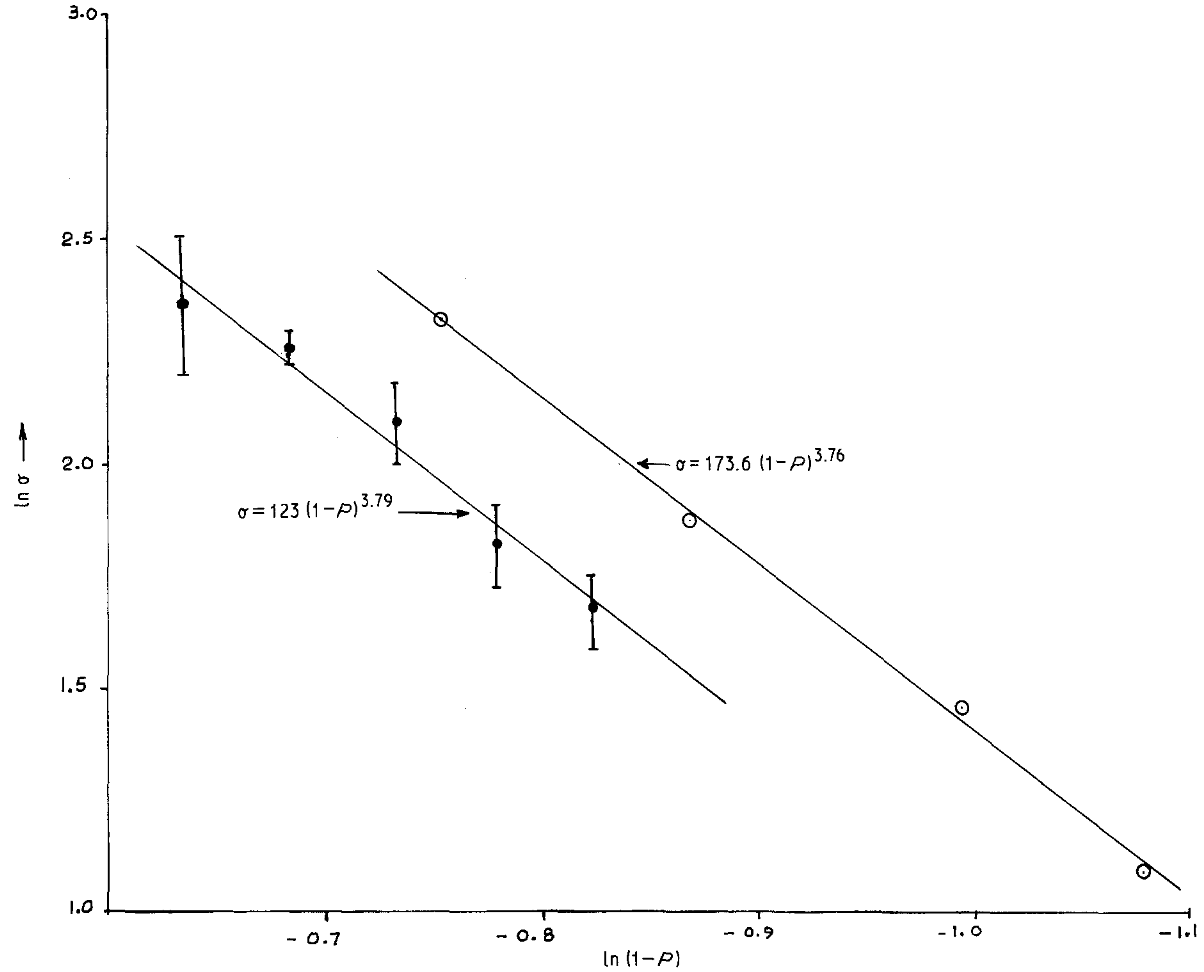

Figure 4 Plot of ln σ against ln $(1 - P)$ (confidence limit $p = 0.5$). (⊙) Schiller [8], (●) present work.

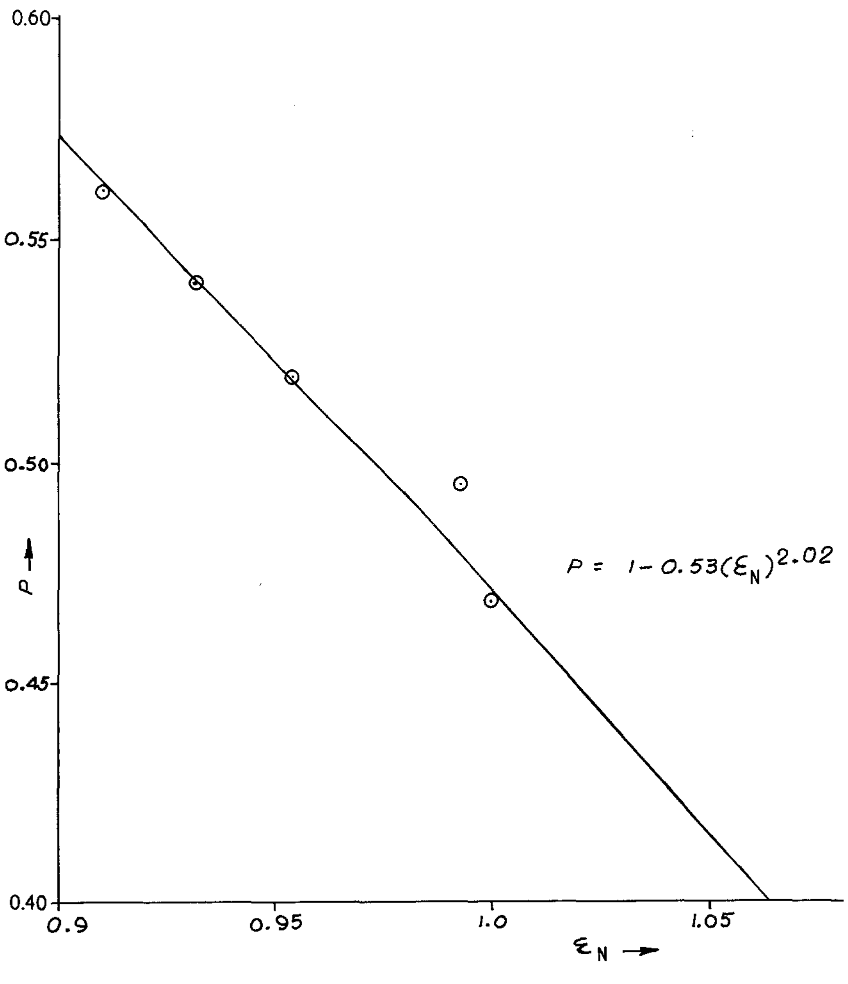

Figure 5 Plot of porosity against normalized stress wave factor.

wave factor by the equations

$$\sigma = K\varepsilon_N^C$$

and

$$P = 1 - \lambda\varepsilon_N^{\mu}$$

2. Ultrasonic velocity, elastic modulus and strength are related to porosity:

$$V_L = V_{L0}(1 - P)^m,$$

$$E = E_0(1 - P)^n$$

and

$$\sigma = \sigma_0(1 - P)^q$$

The values of the empirical constants m, n and q depend on the microstructure. Unlike the widely used existing equations, the proposed equations obey the boundary conditions.

3. Crystal morphology and pore geometry significantly affect elastic modulus and the empirical constant, n. Depending on porosity, an ordered structure with intergrowth and interlocking of crystals yielded values of elastic modulus much higher and those of n much lower than the corresponding values of other gypsum systems in which the crystals were disordered and the pores were interconnected.

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