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Hardness, elasticity modulus and flexion strength of dry set plaster

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In this study the mechanical properties of dry set plasters are of interest. Shore C hardness of different plasters is given as a function of porosity for porosities ranging from 41 to 65 vol%. The data, and data collected from literature, show that Young's modulus follows an empirical power law for porosities ranging from 26 to 70 vol%. Flexion strengths were measured on samples of different sizes and porosities (41.4–65 vol%). As they are size dependent, strength variation cannot be correlated to the sole porosity. Finally, analysing the results with Weibull's theory led to the proposition of a brittleness scale for plasters. Brittleness increases with decreasing porosity.

1. Introduction

Plaster is one of the earliest building material elaborated by man, the production now being about five million tons a year. In the recent past, prefabricated materials made great strides, but it is most probable that new and still more sophisticated materials will appear on the market in the near future. This is the reason why studies on plaster are still up to date, as crystallization and setting induce properties which can be improved by better controlling the experimental conditions. On the other hand, these properties can be seriously damaged if plaster is set in unfavourable environments. One of the most puzzling problems is to explain how a small amount of water drastically affects the mechanical behaviour of the material.

However, studying the effect of water involves that the behaviour of the dry materials is known. Therefore, the results are first presented by investigating dry set plasters.

Dry plaster is a linear, elastic and fragile material. Its mechanical characteristics are estimated from its hardness, elasticity modulus and strength. Hardness can be measured with different devices, the results being compared to the value of the standard obtained with the same device. Not withstanding that hardness cannot fully describe the mechanical behaviour of plasters, it is very convenient to use it for comparing materials produced under different experimental conditions. Concerning the modulus of elasticity, there are several data in the literature [1–4]. These are used for establishing a model linking Young's modulus to plaster porosity over a wide range of porosity (25–70 vol%). This model is compared to those described in the literature. Hereafter, some results concerning the flexion strength of materials exhibiting different sizes and porosities are presented. Strength and rupture probability are discussed using Weibull's model.

2. Experimental procedure

The samples of set plasters studied were prepared by hydration of a hemihydrate powder, $CaSO_4\ 0.5\ H_2O$. In most cases hemihydrate β was used. Hemihydrate α was only used for obtaining samples with very small porosity ($P = 41.4\%$). Once hemihydrate begins to dissolve, it reprecipitates as gypsum, $CaSO_4\ 2H_2O$, the solubility of which is smaller [5]. The purity of the solid phases is about 96 wt% the impurities brought by hemihydrate being calcium carbonate, alumina and some alkali oxides. Such a purity is rather good for this kind of material. Porosity of the set plasters is adjusted by the addition of different amounts of water to hemihydrate. Porosity changes from 41 to 65 vol% when the ratio water–hemihydrate varies from 50 to 109 wt%. Porosity is determined from the sample mass and volume, taking a volume weight of 2.32 g cm^{-3} for gypsum. Fig. 1 is a microphotograph taken with a scanning electron microscope (SEM) and shows the typical morphology of a set plaster of porosity 57.7%. As soon as the plaster has set, it is left for 24 h at room temperature in an atmosphere of 100% relative humidity. Then, it is dried in an oven at 42 °C and 20% humidity in order to evaporate the excess water trapped in the pores. Even under these soft drying conditions, the material obtains the best possible hardness [6], and can be tested as soon as its mass remains constant. However, this condition is never really fulfilled [6] because some water molecules are slowly extracted from the crystal structure before

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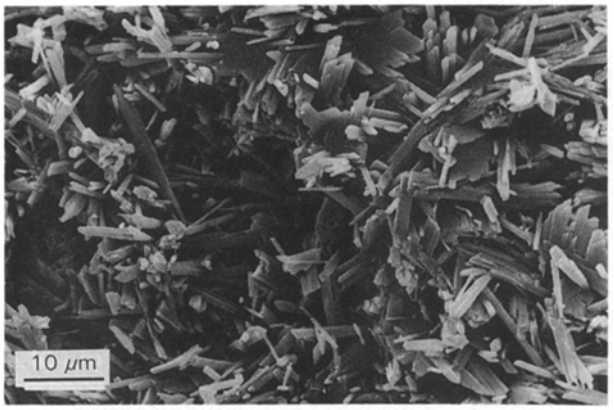

Figure 1 Typical aspect of a set plaster (porosity 57.7 vol %) observed by SEM. Porosity and needle elongation depend on the water:hemihydrate ratio used for producing the material.

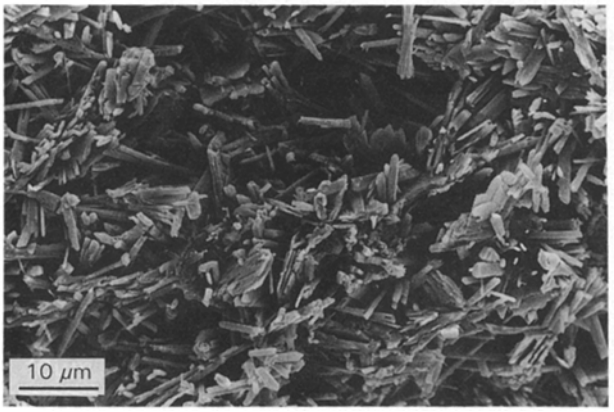

Figure 2 Aspect of a set plaster (porosity 57.7 vol %) initially seeded with a powder of ground gypsum crystallites.

all the water has been removed from the pores. This is valid, even under these drying conditions, but does not affect the mechanical properties of the material even when drying lasts over long periods of time.

In some cases, a small mass of ground gypsum crystals (0.2 wt % with respect to hemihydrate) was added to the hemihydrate powder prior to hydration. The main effect of this solid additive was to induce heterogeneous nucleation, resulting in the formation of a larger number of smaller gypsum crystallites (Fig. 2). After setting, the properties of the material were significantly modified with respect to the standard obtained without gypsum seeds. In a few cases, sorbitol ($C_6H_{14}O_6$) was also used as an additive. It was dissolved in water (2 wt %) prior to hemihydrate hydration. This additive was selected from several others because it slightly improved the mechanical characteristics of set plasters, without significantly changing either the size or the crystal habit of gypsum needles.

Finally, it is noteworthy that, all plaster samples tested issued, after sawing, from a volume of large plaster blocks ($5 \times 25 \times 25\ cm^3$) in order to obtain good sample homogeneity and to avoid altering the hardness measurements by crystal orientation due to surface effects. Compression of the blocks either before or at the end of setting was avoided. A consequence of this non-compression is that it is impossible to get extremely small porosities, less than 25%; but a large porosity range was investigated above this value, up to about 70%.

3. Hardness of set plasters

Vickers hardness of several plasters was determined by Soroka and Sereda [3], while Brinnel hardness was determined by Röbler and Odler [1]. In the former case, the drying conditions were 20 °C and 50% relative humidity. In our opinion, these conditions are too soft to give to the material the best mechanical properties, because residual water in the pores greatly affects these properties [6]. In the latter case, the measurements were performed on plasters dried at 40 °C. Their porosity ranged from 34.5 to 57%.

In this case, hardness was measured on large flat surfaces by means of a Zwick device, graduated from 0 to 100 shore C units. Unit conversion from shore C units into $kg\,cm^{-2}$ has already be given by Amathieu and Boistelle [7]. Further conversion into MPa units can be made easily by dividing the latter units by ten. Here, indentation hardness is dealt with, i.e. the resistance offered by plaster to the penetration of a needle of 1.3 mm diameter applied with a force of 50 N. It is noteworthy that the measurements must be performed rather rapidly once the samples are taken out from the oven at 42 °C. As a matter of fact, at 20 °C and 60% humidity, hardness decreases within 20 min by 3 or 4 shore C units. The situation becomes rapidly worse with increasing relative humidity. Furthermore, due to the preparation of the plaster paste, there are some local inhomogeneities in the samples (large pores, air bubbles), so that hardness may drastically change from one impact point of the needle to another. Consequently, to obtain the proper value of hardness, about 100 measurements were performed on several samples of the same porosity. If all precautions are taken (flatness of the samples, drying conditions, large number of measurements), then the standard deviation is small, as shown in Fig. 3. It is also seen that plasters set in the presence of the two impurities selected, exhibit a slightly improved hardness. These results give the first information on the plaster properties. However, the test concerns the surface of the sample rather than its core, which is better characterized by the modulus of elasticity and strength.

4. Elasticity modulus and flexion strength

The hardness measurements were performed on several hundreds of samples, and the small standard deviations show that set plaster is homogeneous at least on the scale of the hardness-meter. If it is assumed that this homogeneity exists inside the whole plaster volume, at least on the scale of a few micrometers, one may hope to describe the mechanical properties of the material using Young's modulus and strength.

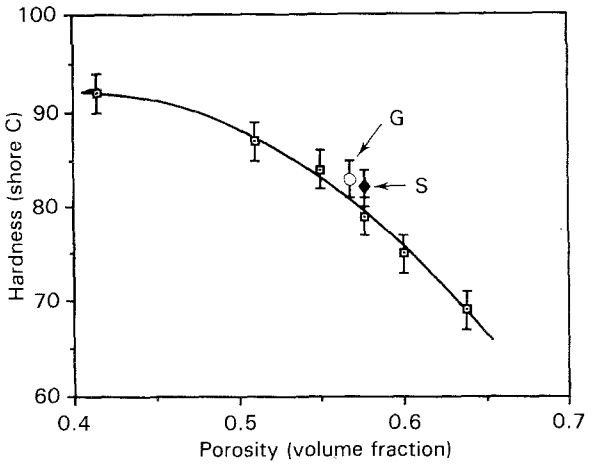

Figure 3 Shore C hardness of pure and poisoned plasters produced in the presence of ground gypsum seeds (G) and sorbitol (S).

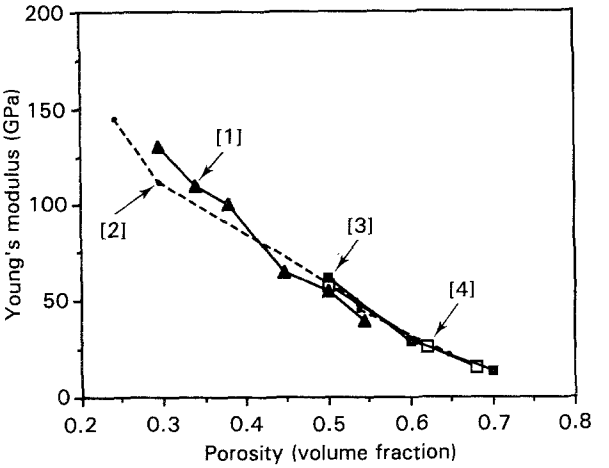

Figure 4 Young's modulus of set plasters according to different sources in a 25–70 vol % porosity range [1–4], see text.

4.1. Elasticity of dry plaster

Several measurements of Young's modulus were performed as a function of plaster porosity [1–4]. The data obtained by hydration of α and β hemihydrates are collected in Fig. 4 for a 25 to 70 vol % porosity range. Not withstanding the different experimental methods, compression [3], ultrasonic [1, 4] and three-point bending [2], the results are in good agreement, with a scattering of about 10%. From the results of the three-point bending test (Fig. 5), Young's modulus, E, can be calculated [8] from

$$E = \frac{F_{rupt}}{d}\frac{L^3}{4bW^3} \quad (1)$$

where d and F_{rupt} are the beam deflection and the rupture force, respectively while L, b and W are the sample dimensions given in Fig. 5. Unfortunately, with this experimental method, there is significant variation of Young's modulus with the changing dimensions of the samples. At small $L:W$ ratios ($3 \leq L:W \leq 5$), some shearing of the sample, and crushing on the device knife-edges, induce small deformations which are superimposed on the deformation due to the sole elasticity. Keeping only the contribution of elasticity yields, the following results for plasters of 53.5% porosity are obtained

$$E = 2.0\,\text{GPa} \quad \text{for} \quad L:W = 3$$

$$E = 5.3\,\text{GPa} \quad \text{for} \quad L:W \geq 16$$

E remains constant under the condition that $L:W \geq 16$.

The influence of porosity on Young's modulus has already been described with several models for different materials. In two articles devoted to porous alumina [9] and set plaster [4], Phani makes a review of the models linking Young's modulus to porosity, P. The models which are commonly used to give linear laws are

$$E = E_0(1 - \lambda P) \quad (2)$$

or exponential laws

$$E = E_0 \exp(-kP) \quad (3)$$

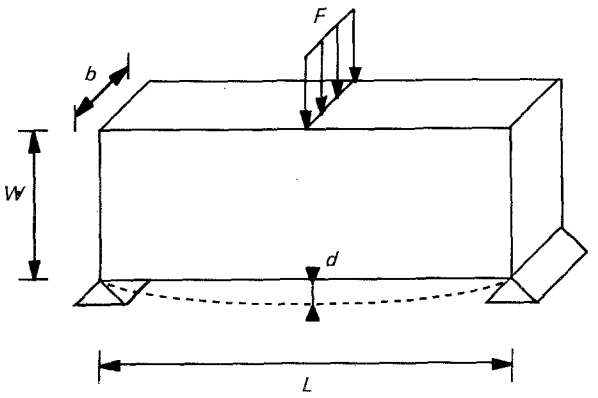

Figure 5 Characteristic dimensions of the samples used in the three-point bending test (see text).

where λ and k are empirical constants, and E_0 Young's modulus at $P = 0$. In the model proposed by Hasselmann [10] there are two adjustable parameters, E_0 and A, which is another empirical constant

$$E = E_0\left[1 - \frac{AP}{1 + (A - 1)P}\right] \quad (4)$$

For set plasters, Phani [4] proposed the power law

$$E = E_0(1 - aP)^n \quad (5)$$

where $a \geq 1$ is a packing geometry factor, e.g. for $P = 1/a$ the solid structure of plaster breaks down. The exponent n is dependent on pore and crystal geometries.

Other models have even more adjustable parameters. In Wang's model [11]

$$E = E_0 \exp[-K(P)] \quad (6)$$

K is a polynomial of second or higher order.

In the model of Knudsen [12] elaborated for spherical ceramics, Young's modulus is written as

$$E = E_0 G^{-a} \exp(-bP) \quad (7)$$

G being the diameter of the particles, while a and b are empirical constants. The results were not fitted with the two latest models because they required more than two adjustable parameters.

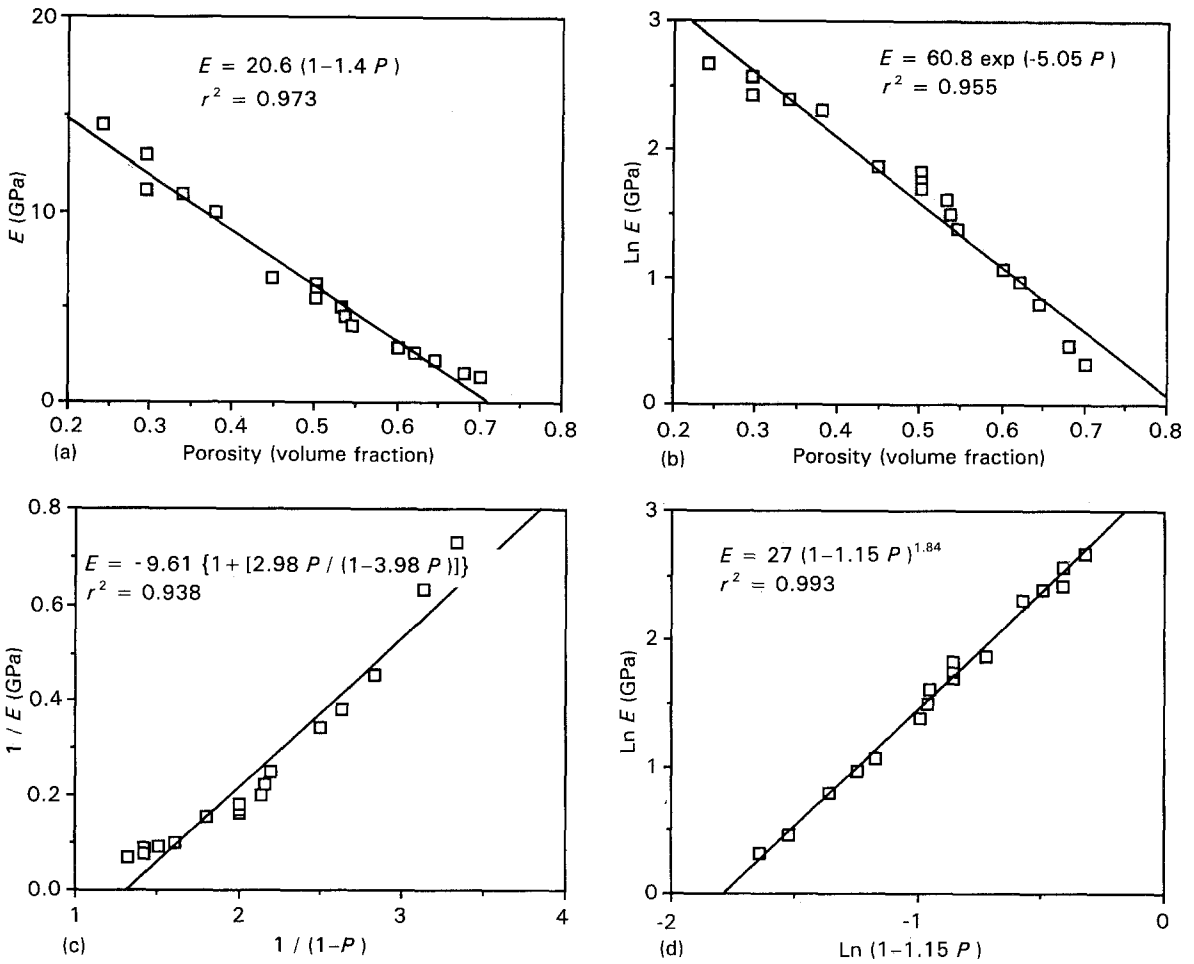

Figure 6 Young's modulus of set plasters fitted with (a) a linear law, Equation 2; (b) an exponential law, Equation 3; (c) Hasselman's model, Equation 4; (d) a power law, Equation 5.

In Fig. 6a–d, the data displayed in Fig. 4 is fitted with Equations 2–5, respectively. The correlation factor, r^2, is given on each figure.

The power law best fits the results with E_0 = 27 GPa at zero porosity. This value is higher, by about one order of magnitude, than the value obtained by Williams [13] with the ultrasonic method on single crystals along the preferential (0 1 0) cleavage plane. In set plasters, the fracture mainly occurs between undefined crystal contacts, the interaction energies of which are unknown.

4.2. Flexion strength

Contrary to the determination of Young's modulus, the determination of strength is more dependent on the technique which is used. For a plaster with $P = 57\%$, the tension strength is 2.1 ± 0.3 MPa, while the compression strength is 10 ± 1 MPa. The flexion strength, measured by means of the three-point flexion test [2] is 3.8 ± 0.3 MPa. As the strength of brittle material may be dependent on the volume of the sample [14] special attention was paid to this point. Measurements of flexion strength (Fig. 5) were performed with the Hadamel-Lhomargy DY 30 press. At the beginning of the study two strain rates were used: 0.1 and 1 mm min^{-1}. As no significant difference was observed on the mechanical behaviour of the samples, the remaining experiments were performed at a strain rate of 1 mm min^{-1}. From the rupture force, F_{rupt}, measured with the press, flexion strength, σ, was calculated for [8] each sample, from

$$\sigma = \frac{3F_{rupt}L}{2b\,W^2} \quad (8)$$

TABLE I Geometry and volume of plaster samples used for measuring the flexion strengths given in Table II

Size	Length, L (cm)	Width, W (cm)	Breadth, b (cm)	Volume, V (cm^3)
A	4	1.2	1.5	9
B	6	1.5	3.0	27
C	10	1.5	5.0	75

Tables I and II summarize sample geometries and mean strengths for each specimen shape and plaster. Three conclusions may be drawn from Table II and Fig. 7. The first concerns the plaster initially seeded with ground gypsum (G) or poisoned with sorbitol (S). The mean strength of these samples is significantly increased with respect to the strength of pure plaster, which confirms the results obtained with hardness measurements. The second remark concerns the standard deviation of the mean strength, about

TABLE II Flexion strengths (MPa) of set plasters with different porosities, P (vol %), where N is the number of tested samples

	Porosity, P (vol fraction)					
	0.414[a]	0.525[a]	0.577[a]	0.65[a]	0.573[b]	0.573[c]
Size A	For $N = 20$ 8.79 $\pm$ 0.91	For $N = 21$ 5.81 $\pm$ 0.79	For $N = 40$ 4.50 $\pm$ 0.66	For $N = 31$ 2.94 $\pm$ 0.32	For $N = 40$ 5.44 $\pm$ 0.62	For $N = 37$ 5.48 $\pm$ 0.78
Size B	For $N = 15$ 7.35 $\pm$ 0.89	For $N = 30$ 4.93 $\pm$ 0.71	For $N = 28$ 3.85 $\pm$ 0.44	For $N = 26$ 2.81 $\pm$ 0.28	For $N = 16$ 4.32 $\pm$ 0.52	For $N = 18$ 4.38 $\pm$ 0.92
Size C	For $N = 15$ 6.81 $\pm$ 0.79	For $N = 14$ 4.07 $\pm$ 0.49	For $N = 14$ 3.65 $\pm$ 0.38	For $N = 14$ 2.44 $\pm$ 0.18	For $N = 14$ 3.89 $\pm$ 0.42	For $N = 14$ 3.85 $\pm$ 0.58

[a] Measurements made on pure plasters.
[b] Plasters seeded with gypsum crystallites (G).
[c] Plasters poisoned with sorbitol (S).

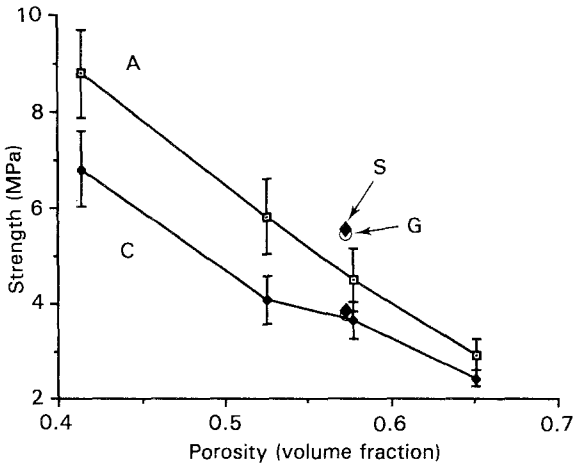

Figure 7 Flexion strengths of set plasters versus porosity for sample sizes A and C in Table I (G = gypsum; S = sorbitol).

10–15%, which seems to be independent of the number of tested samples and the mean value of the stress. From additional measurements on plaster samples of the same porosity ($P = 57\%$) and dimensions ($L \times b \times W$) = ($6 \times 3 \times 1.5$ cm^3) but issued from two different blocks

$$\sigma = 3.77 \pm 0.35 \text{ MPa} \quad (70 \text{ samples})$$

$$\sigma = 3.81 \pm 0.47 \text{ MPa} \quad (84 \text{ samples})$$

The standard deviation of the experimental points is still about 10% or more. The third conclusion is that the mean strength is dependent on the size of the sample with a clear correlation observable between sample volume and strength. Within the size range we have investigated, the size effect is very significant: for a porosity of 52.5%, the mean strength varies by 30% between samples A and C. Accordingly, it is concluded that the strength does not properly characterize the mechanical properties of plaster. This has already been observed on other materials [14]. The hypothesis that the plaster is homogeneous throughout the bulk, is therefore not grounded. To describe strength variations, it is necessary to consider theories which take heterogeneity into account.

5. Discussion

Weibull proposed a model for explaining the important scattering of the mean rupture strength values and their variations with sample size. In doing this, he started from some observations partially drawn from the work by Orowan [15], who suggested that the theoretical strength, σ_{th}, and the surface energy, γ_S, of the sample are correlated by the equation

$$\sigma_{th} = (E\gamma_S/2d)^{1/2} \quad (9)$$

in the case of a monocrystalline, cubic and perfect material. Under these conditions, γ_S is the surface energy of the cleavage plane created by the rupture and d the cell parameter normal to this plane. Applying this formula to calcite, with Gilman's data [16], it was found [6] that

$$\sigma_{th} = 7.37 \times 10^4 \text{ MPa}$$

Comparison of this value with the experimental data is rather disappointing, since tension strengths measured on limestone samples are about 35.2 MPa [17]. The difference is about three orders of magnitude, and Orowan [15] concluded that defects, grain boundaries, microcracks, dislocations and pores induce stress concentrations which may lead to material rupture long before σ_{th} is reached.

In addition, the strengths of plastic materials are less scattered than those of brittle materials. In metals, this is the consequence of the presence of large numbers of linear defects (dislocations), about 1×10^5 to 1×10^7 cm cm^{-3} [18]. Once the elastic limit of the material is locally exceeded, many dislocations form and move in order to relax the local stress. Measurement of strength can therefore give a good image of the entire material properties, which is not the case for brittle materials. In the latter case there are far less dislocations ($10 \simeq 1 \times 10^3$ cm cm^{-3}) which, moreover, cannot move and relax the local stress. Rupture arises in the vicinity of the defect which has the highest stress concentration. It is noteworthy that in the case of plaster for instance, some defects pre-exist in the solid structure (pores, microcracks, etc.), defects which favour rupture when the material undergoes some stress.

Brittle fracture is a local phenomenon which happens in a frozen system. Consequently, strengths are more representative of a few defects inside the material than of the material itself.

According to Weibull [19] the defect distribution accounts for the distribution of the strengths of fragile materials. The model, named model of the weakest bond, is grounded on the following hypotheses:

1. the number of defects is large;
2. the material is isotropic and the probability of finding a critical defect is constant inside the bulk; and
3. the rupture of the weakest bond leads to the ruin of the material.

Accordingly, the material is no more considered as homogeneous: it contains singular points without interactions and is dispersed in a perfectly homogeneous matrix.

For a sample of volume, V, undergoing a uniform stress, σ, the distribution function of Weibull [19] is

$$P_f = 1 - \exp\left[- V\left(\frac{\sigma - \sigma_u}{\sigma_0}\right)^m \right] \quad (10)$$

where P_f is the cumulated rupture probability corresponding to the flexion stress σ; σ_u is a strength threshold, which is zero for a brittle material such as plaster; $\sigma_0 = 1$ is a normalization factor; m is Weibull's modulus, an empirical parameter which decreases with increasing fragility. It ranges from 10 to 30 for ceramics and from 50 to 100 for metals.

Fig. 8 displays the variation of the cumulative rupture probability versus the strengths of 57.7% porosity plaster samples, the samples having the dimensions given in Table I. To obtain Weibull's moduli, m, a linear regression on the data displayed in Fig. 8 is made

$$\ln\left[\ln\left(\frac{1}{1 - P_f}\right)\right] = m \ln \sigma + \ln V \quad (11)$$

Fig. 9 shows the fits obtained with this plaster. The data for all plasters studied are collected in Table III: the m values are given for each sample size, A, B and C. The number of tested samples is indicated in Table II.

From the correlation coefficients found by linear regression, it can safely be concluded that plaster enters in the framework of Weibull's model. Weibull's modulus decreases with decreasing porosity. Plasters exhibiting smaller porosities become harder, but more brittle. In the same way, the additives used, known for increasing the strength of the material, induce a decrease of Weibull's modulus and an increase of brittleness.

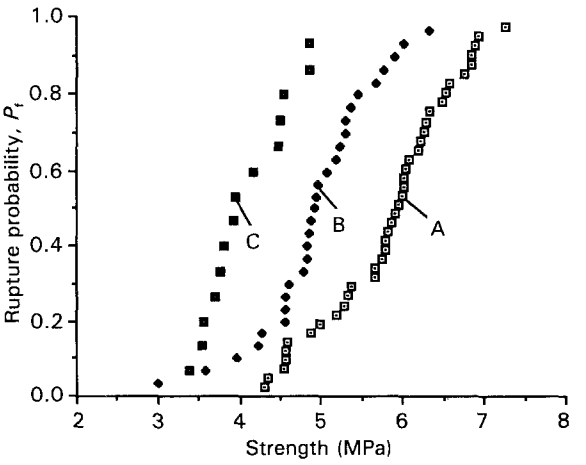

Figure 8 Cumulative rupture probability versus flexion strength for samples A, B and C of a 57.7 vol % porosity plaster.

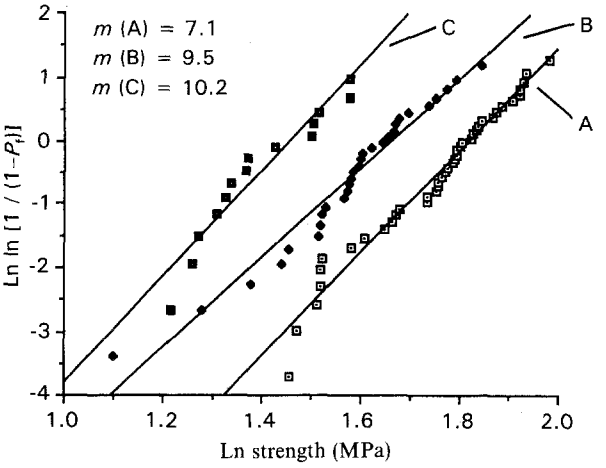

Figure 9 Data of Fig. 8 fitted to Weibull's model, and corresponding Weibull's moduli, m, for a 57.7 vol % porosity plaster.

TABLE III Weibull's moduli, m, for set plasters of sizes A, B and C (Table I). The coefficients of linear regression are given in brackets. The number of tested samples is given in Table II

	Porosity, P (vol fraction)					
	0.414[a]	0.525[a]	0.577[a]	0.65[a]	0.573[b]	0.573[c]
m (A)		8.10 (0.97)	7.10 (0.99)	10.40 (0.91)	9.10 (0.96)	7.95 (0.98)
m (B)	8.30 (0.95)	7.13 (0.96)	9.50 (0.97)	9.03 (0.99)	8.60 (0.99)	5.05 (0.98)
m (C)	8.00 (0.97)	8.30 (0.92)	10.20 (0.96)	13.30 (0.96)	9.35 (0.96)	6.50 (0.97)

[a] Measurements made on pure plasters.
[b] Plasters seeded with gypsum crystallites (G).
[c] Plasters poisoned with sorbitol (S).

TABLE IV Weibull's moduli, m, calculated with Equation 11

	Porosity, P (vol fraction)				
	0.525[a]	0.577[a]	0.65[a]	0.573[b]	0.573[c]
m (A, B)	6.67	7.04	–	4.76	4.90
m (B, C)	5.30	–	7.13	9.79	7.95
m (A, C)	5.93	10.12	11.33	6.33	6.01

[a] Measurements made on pure plasters.
[b] Plasters seeded with gypsum (G).
[c] Plasters poisoned with sorbitol (S).

Finally, it must also be pointed out that the hypothesis that the rupture of the weakest bond leads to the breakdown of the material implies a dependence of the mean strength on the sample volume: the larger the volume is, the higher the probability of finding a critical defect, and the smaller the resistance of the material. If σ_i and σ_j are the average values of samples of volumes V_i and V_j, Weibull's formula leads to

$$\sigma_i/\sigma_j = (V_j/V_i)^{1/m_{ij}} \quad (12)$$

where the ij pairs correspond to pairs AB, AC and BC. This equation was used to calculate Weibull's moduli of the three sample sizes tested. The results are given in Table IV, and can be compared to that obtained from the probability distribution given in Table III. In most cases the agreement is good, which means that the approach of Weibull describes pretty well the brittleness of plaster. The differences observed between some values is to be attributed, in the authors' opinion, to the fact that the number of measurements on some samples is not large enough.

6. Conclusions

In the present work, the effect of porosity on indentation hardness, modulus of elasticity and flexion strength was studied. Hardness rapidly increases with decreasing porosity, while an empirical power law links Young's modulus to porosity in the 25–70% porosity range investigated

$$E = 27\,(1 - 1.15P)^{1.84}\ \text{MPa} \quad (13)$$

Mean flexion strength could not be described with such a law, as an important size effect was observed. Small samples systematically exhibit a higher mean strength than larger samples. In addition, the rather large standard deviation on the value of Young's modulus was also found on the measurements of flexion strengths. Accordingly, it can be concluded that both these parameters are probably not the best ones for describing the brittle fracture of dry plaster.

Therefore, interpretation of the large standard deviation of the flexion strength values by means of Weibull's theory was attempted. It turned out that the mechanical behaviour of dry plaster can be described by this model. With a modulus less than 10, dry plaster may be considered as a very brittle solid. Brittleness increases, while Weibull's modulus decreases, with decreasing porosity.

As the model supposes, the presence of defects homogeneously distributed in the bulk, it was decided to extend the present study by investigating the mechanical behaviour of plaster, using linear elastic fracture mechanisms. This work is in progress.

Acknowledgements

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