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Technical note

A Microstructure-based Failure Criterion for Aminadav Dolomites

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INTRODUCTION

Dolomites are heterogeneous in general and therefore predicting their ultimate strength is quite a challenge. Several components contribute to the heterogeneity of dolomites: a wide grain size distribution, a variability in pore shape and size and the co-existence of different types of crystalline mosaic texture within a single rock specimen. These textural differences have a paramount influence on rock strength. Grain boundaries are commonly considered to function as initial stress concentrators during application of deviator stress and consequently crack initiation stress is expected to be inversely proportional to mean grain size [1–3]. Cylindrical pores also function as stress concentrators [4, 5] and therefore crack initiation stress should be inversely related to mean pore radius as well. Finally, the type of crystalline mosaic texture and the proportion between fine grained matrix and crystalline mosaic in a given specimen introduce constraints on the fracture propagation and coalescence mechanisms which ultimately lead to strength failure [6, 7]. To date, there has not been a comprehensive attempt to account for such microstructural parameters in a usable failure criterion. The most popular failure criteria currently used in rock engineering practice involve empirically determined material [8] or fitting [9, 10] parameters, while microstructural parameters which account for rock texture are largely ignored.

In this paper an attempt is made to quantify the influence of microstructure on the ultimate strength of specimens of dolomite rock, using petrographic and mechanical test data performed on 18 specimens of Aminadav dolomite, a sequence of Cretaceous platform dolomites of wide surface and underground distribution in Israel and adjacent countries. Measurable physical quantities such as mean grain size and bulk porosity are used in the model, together with obtainable mechanical quantities such as the elastic modulus and the level of confining pressure. Thus the proposed criterion introduces a first step towards the achievement of a much broader goal, the prediction of brittle heterogeneous rock strength using measurable and meaningful microstructural and mechanical parameters.

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EXPERIMENTAL PROCEDURES

Microstructure of Aminadav dolomites

Aminadav dolomites are used for starting material as they are good representatives of brittle, crystalline, rocks: they are typically heterogeneous and they exhibit linear elastic deformation up to fracture initiation stress. Aminadav dolomites are generally monomineralic, calcite is found in limited abundance in re-crystallized void space and in the fine grained matrix, which is comprised predominantly of ultra fine grained dolomite crystals. Three different types of crystalline mosaic texture are identified in Aminadav dolomites [6, 7], the quantification and scaling of which is presently not attempted. Therefore, the influence of mosaic texture on ultimate strength is not accounted for in the proposed failure criterion. The two microstructural parameters which are quantified and incorporated in the failure criterion are mean grain size and porosity.

Grain size values are determined from measurements performed on scaled SEM micrographs. The grain size distribution within a single specimen is negative exponential, the frequency of the right tail of the distribution however is very low. Some detailed grain size distribution curves are described by Hatzor *et al.* [7]. The mean grain size values are between $d_m = 10\ \mu m$ and $d_m = 50\ \mu m$, with a mean value of $d_m = 26.84 \pm 10.77\ \mu m$. The number of grain size measurements performed for each sample is in the order of $N = 100$.

The porosity of the rock originates from intercrystalline micropores, as well as vugs and channels. The pore size was studied using a calibrated reticule with crossline micrometer in a Zeiss petrographic microscope and by direct measurements on scaled SEM micrographs. The size of the intercrystalline micropores is in the order of several micrometers; the vugs

Table 1. Mechanical and physical properties of studied dolomites

Sample	n (%)	d_m (μ_m)	Dominant mosaic texture	σ_3 (MPa)	E (GPa)	υ	$\sigma_{1,p}$ (MPa)
AD-2	2	49	xenotopic	0	70	0.16	117
AD-5	5.8	35	xenotopic	0	56	0.37	98
AD-6	6.4	12	hypidiotopic	0	35	–	171
AD-13	3.6	25	hypidiotopic	10	61	0.25	253
AD-15	20.9	33	idiotopic	0	29	0.26	67
AD-18	7.9	17	idiotopic	7	58	0.3	209
AD-31	4.6	50	idiotopic	15	42	0.3	206
AD-34	4.24	21	idiotopic	25	63.8	0.28	340
AD-37	13.8	9	xenotopic	15	28	0.35	157
AD-43	5.4	24	hypidiotopic	0	64	0.27	274
AD-80	6.4	26.9	hypidiotopic	0	58.5	0.28	174
AD-81	6.8	27.5	idiotopic	5	59.2	0.18	159
AD-81A	5.7	27.5	idiotopic	10	57	0.27	265
AD-82	10	19.2	hypidiotopic	10	43	0.22	183
AD-82A	13.2	19.2	hypidiotopic	15	41	0.25	225
AD-83	15.4	27.3	xenotopic	0	18	0.25	62
AD-83A	17.9	27.3	xenotopic	5	19.6	0.24	75
AD-84	5.7	33.2	hypidiotopic	10	49.3	0.26	176

Legend: n = initial porosity; d_m = mean grain size; σ_3 = confining pressure; E = elastic modulus; υ = Poisson's ratio: $\sigma_{1,p}$ = ultimate compressive strength.

are typically circular with an average diameter of 427 ± 191 μm. The bulk porosity of the specimens, which consists of all components of void space, was calculated from measured values of dry bulk density (ρ_d) and specific gravity of solids (G_s) and is between 2 and 21%. The initial volume of each specimen was measured using a digital caliper with resolution of 0.01 mm and the dry mass was measured using a digital scale with a resolution of 0.01 g, so that dry bulk density was determined with an accuracy of 0.01 g/cm^3. The precision of the porosity estimation is believed to be within 0.01%.

Mechanical test description

The mechanical test program consists of 7 unconfined and 11 triaxial tests which were performed on NX size cores with a length to diameter ratio of about 2.0, end roughness of 0.01 mm and perpendicularity of 0.005 rad. The samples were loaded in a stiff, closed loop, servo controlled, hydraulic load frame (TerraTek model FX-S-33090), with axial force capacity of 1.4 MN, confining pressure capacity of 70 MPa and stiffness of 5×10^9 N/m. The tests were performed under constant stroke rates between 1×10^{-5} to 1×10^{-6} s^{-1}. Piston displacement was monitored using high sensitivity LVDT located outside the pressure vessel, axial and radial strain were monitored using four arm strain cantilever sets with 0.5% linearity full scale and axial and radial strain limit of +7 and −7%, respectively (compression is positive throughout this paper). Load was monitored using a 1000 KN load cell with 0.5% linearity full scale, which was placed inside the pressure vessel and directly below the sample stack.

INFLUENCE OF MICROSTRUCTURAL PARAMETERS ON ULTIMATE STRENGTH

The results of the 18 fully monitored tests are summarized in Table 1 and are discussed below. Ultimate strength results ($\sigma_{1,p}$) in all 18 tests are plotted against confining pressure (σ_3) values in Fig. 1. The great scatter in unconfined compressive strength (62 MPa < σ_c < 274 MPa) is evident and thus the difficulty in the application of normalized failure criteria which use σ_c as a material property. Furthermore, the

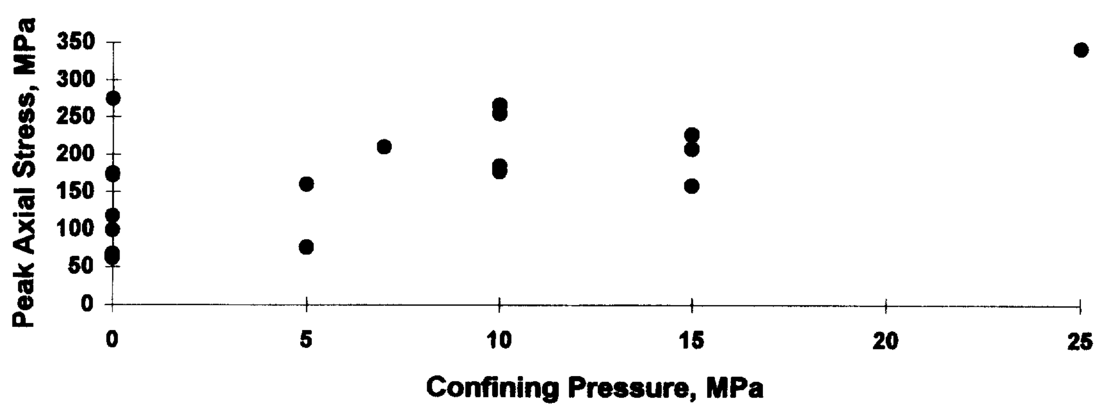

Fig. 1. Results of 18 fully monitored compression tests in $\sigma_{1,p}$ vs σ_3 space.

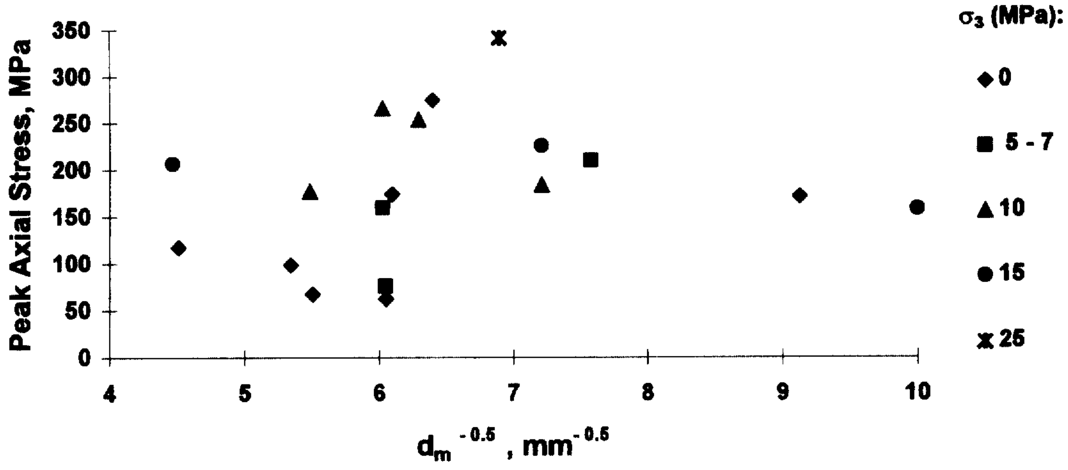

Fig. 2. Relationship between inverse square root of mean grain size (d_m, measured using SEM) and ultimate strength.

data do not seem to follow a linear trend as predicted by the Coulomb–Mohr failure criterion; however a clear strengthening effect due to confining pressure is indicated. The relationship between ultimate strength and mean grain size, bulk porosity and rock stiffness is discussed below.

The relationship between inverse square root of mean grain size and ultimate strength is shown in Fig. 2. Assuming a Hall–Petch relationship [11] is valid for brittle rock [1, 2] the plotted data should show an increasing, linear trend. Wong *et al.* [3] for example have shown a good linear correlation between inverse square root of mean grain size and unconfined compressive strength of Yuen Long marbles. Fredrich *et al.* [2] have shown three linear trends for three different levels of confining pressures, with remarkable correlation coefficients, for the yield stress of marbles and limestones. The good correlation which has been reported for carbonate rocks [1–3] implies that either grain size is the only microstructural parameter which influences rock strength, or that the tested rocks [1–3] exhibit exceptional material homogeneity, such that mean grain size is the only microstructural parameter which exhibits size and/or shape variations. The first assumption has been invalidated by recent studies where it has been shown that mean grain size alone could not be used to predict neither crack initiation [6] nor peak stress [7] and that the influence of additional microstructural parameters, such as porosity, had to be considered. The second supposition has been invalidated in numerous papers on the petrography of carbonate rocks, where great variability in pore size, mosaic texture, micrite content, etc. is reported (e.g. Refs. [12, 13]). Because the tested rocks are heterogeneous, the influence of and the interaction between other microstructural parameter explain the weak linear correlation which is presented in Fig. 2.

The relationship between bulk porosity and ultimate strength is shown Fig. 3. Clearly ultimate strength is inversely related to bulk porosity. The observed trend in Fig. 3 supports the assumption that initial pores are significant stress concentrators [4, 5], although ultimate strength is plotted against bulk porosity and not mean pore radius.

In addition to mean grain size and porosity, rock stiffness may be used to scale a third microstructural

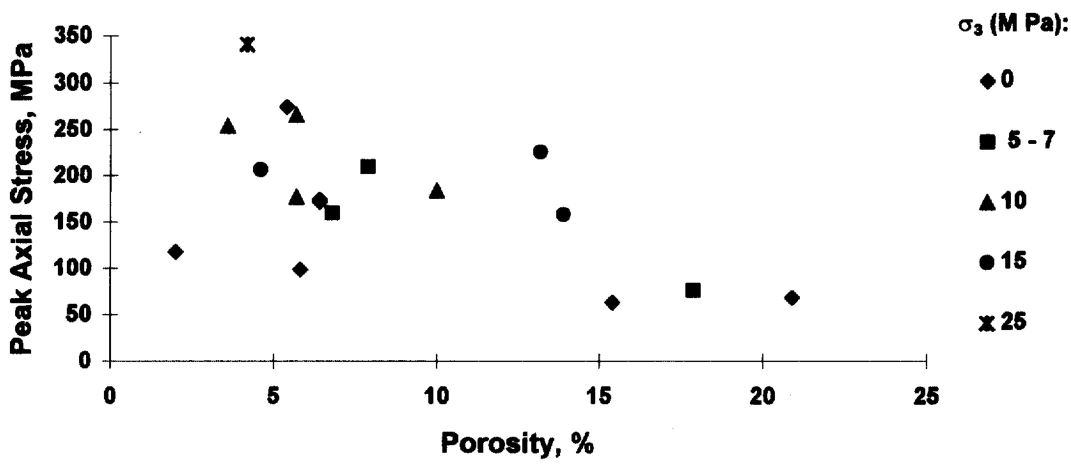

Fig. 3. Relationship between bulk porosity and ultimate strength.

parameter: the ratio between the more compressible fine grained matrix and the stiffer crystalline mosaic, in a given rock specimen. The stiffness of the rock system (including all textural and crystalline components) is given by the elastic modulus of the rock, which is measured along the linear segment of the stress–strain curve. The relationship between ultimate strength and rock stiffness can be inferred from Fig. 4 where peak axial stress is plotted against elastic modulus. Clearly ultimate strength is directly proportional to stiffness. We have argued above that fracture initiation stress depends upon the size of initial flaws such as grain boundaries and cylindrical pores, because they function as stress concentrators. Once fractures are initiated, however, their growth and coalescence depend upon the availability of propagation paths, which increases with increasing proportions of fine grained matrix in the specimen. This proportion is scaled here by the stiffness, or the elastic modulus, of the rock.

A NEW MICROSTRUCTURE-BASED FAILURE CRITERION FOR DOLOMITES

The influence of microstructural parameters on the ultimate strength of Aminadav dolomite has been shown in Figs 2–4. We now develop a new, empirical, failure criterion for this lithology on the basis of the experimental results. We believe that such an approach could be adopted for ultimate strength prediction in other brittle, heterogeneous, lithologies where microstructural parameters are expected to play an important role in the determination of ultimate strength.

There is a substantial amount of experimental evidence [1–3, 11] showing that the ultimate strength in brittle materials, denoted here as $\sigma_{1,\mathrm{p}}$, increases with the inverse square root of mean grain size (d_m). This relationship could be generally expressed using:

$$\sigma_{1,\mathrm{p}} = A\frac{1}{\sqrt{d_\mathrm{m}}} \tag{1}$$

where A is an empirical parameter. Our experimental results clearly show that ultimate strength increases non-linearly with confining pressure (σ_3, Fig. 1) and elastic modulus (E, Fig. 4) and decreases non-linearly with porosity (n, Fig. 3). Therefore we may express parameter A using the following implicit function (Equation (2)):

$$A = f\left[\frac{(E, \sigma_3)}{n}\right] \tag{2}$$

Using the least squares fit method we found that the relationship between ultimate strength and elastic modulus (Fig. 4) best follows a power law, whereas the relationship between ultimate strength and porosity (Fig. 3) best follows an exponential law. Therefore Equation (2) could be written using a power or exponential law, or using a combination of the two laws. For example, parameter A could be written using one of the forms $A = (E^{k1}\ \sigma_3^{k2}/n^{k3})$; $A = (e^{k1E}\ e^{k2\sigma_3}/e^{k3n})$; or using combinations, e.g. $A = (E^{k1}\sigma_3^{k2}/e^{k3n})$, where k_1, k_2 and k_3 are correlation coefficients. We have analyzed each of the possible laws, as well as possible combinations using the least squares method and successively corrected the selected law and correlation coefficients. We found that the explicit function A in Equation (1) having the largest squared regression coefficient ($R^2 = 0.84$), has the following forms:

$$A = \frac{aE^c}{n^b} \tag{3}$$

for unconfined compression and

$$A = \frac{a(E\sigma_3)^c}{n^b} \tag{4}$$

for triaxial compression where a, b and c are correlation coefficients. For the studied Aminadav dolo-

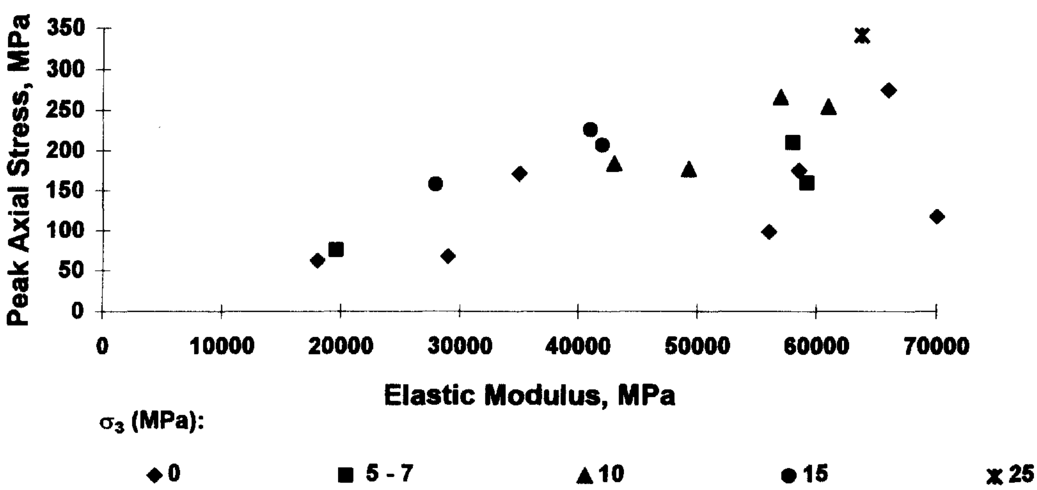

Fig. 4. Relationship between elastic modulus and bulk porosity.

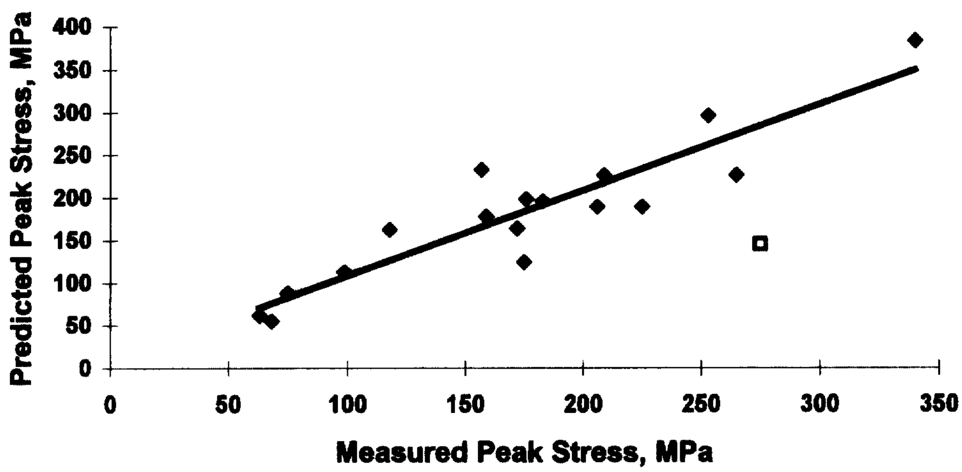

Fig. 5. Predictive capability of the new microstructure-based failure criterion ($R^2 = 0.84$).

mites, the value of the correlation coefficients are: $a = 3.14$ (or π); $b = 0.45$ and $c = 0.25$ for E, σ_3 and $\sigma_{1,p}$ (or σ_c) in MPa; d_m in mm and n in %. Inserting the appropriate form of the function A in Equation (1) we get the new empirical models for ultimate strength under uniaxial (Equation (5)) and triaxial (Equation (6)) compression:

$$\sigma_c = \frac{aE^c}{n^b\sqrt{d_m}} \tag{5}$$

$$\sigma_{1,p} = \frac{a(E\sigma_3)^c}{n^b\sqrt{d_m}} \tag{6}$$

where σ_c is unconfined compressive strength and $\sigma_{1,p}$ is ultimate strength. The proposed criterion is expected to be valid for brittle, heterogeneous dolomites for the following range of physical and mechanical parameters: 20 000 MPa $< E <$ 65 000 MPa; 15 μm $< d_m <$ 50 μm; 2% $< n <$ 21%, 1 MPa $< \sigma_3 <$ 25 MPa. The predictive capability of the proposed criterion (Equations (5) and (6)) is verified against the 18 tested specimens in Fig. 5. The squared regression coefficient (R^2) in Fig. 5 is 0.84 excluding one test (open square). The test which falls out of the regression line (sample AD43) was exceptional. The sample was loaded at a constant strain rate of 1×10^{-6} s^{-1}. Upon attainment of ultimate strength the sample disintegrated abruptly generating a very loud acoustic emission, following which the control loop was opened and both radial and axial cantilever sets were damaged. This type of explosive rupture was not observed in any other sample. We cannot explain the observed behaviour using physical and mechanical properties and we therefore treat this data point as an outlier.

DISCUSSION

The prediction of the proposed failure criterion is plotted in Fig. 6 for an average elastic modulus value ($E_{aver.} = 46\,700$ MPa). In Fig. 6(a) unconfined compressive strength (σ_c) is plotted against mean grain size and porosity and in Fig. 6(b) and (c) ultimate strength is similarly plotted for confining pressures of 10 and 25 MPa, respectively. A three dimensional plot of Equation (5) for unconfined compression (for the average elastic modulus value) is shown in Fig. 7. The effect of mean grain size and porosity on ultimate strength, as predicted by the proposed criterion, is similar for both uniaxial and triaxial compression. In all cases ultimate strength rapidly decreases with increasing porosity. Consider for example unconfined compression (Fig. 6(a) and Fig. 7) at a mean grain size of 0.015 mm. When porosity increases from 4 to 10% ultimate strength decreases by 34% ($\sigma_{c(n\,=\,4\%)} = 193$ MPa, $\sigma_{c(n\,=\,10\%)} = 128$ MPa), however when porosity increases from 10 to 20% σ_c decreases by only 27% ($\sigma_{c(n\,=\,20\%)} = 93$ MPa). This trend remains the same for all mean grain size values and for all levels of confining pressure. The effect of mean grain size on ultimate strength is similar yet less pronounced. Consider again unconfined compression (Fig. 6(a) and Fig. 7) and bulk porosity of 4%. When mean grain size is increased from 15 to 30 μm ultimate strength decreases by 29% ($\sigma_{c(d_m=15\,\mu m)} = 193$ MPa, $\sigma_{c(d_m=30\,\mu m)} = 137$ MPa) and when mean grain size is increased from 30 to 50 μm ultimate strength decreases by 23% ($\sigma_{c(d_m=50\,\mu m)} = 106$ MPa). This trend also remains the same for all values of bulk porosity and confining pressure.

In the discussion of the criterion above (Figs 6 and 7) the elastic modulus was considered a constant for all samples. Such an assumption implies material homogeneity where the relative abundance of fine grained matrix in all samples is equal. This however is rarely the case in dolomites. The variation in unconfined compressive strength as a function of elastic modulus and porosity is shown in Fig. 8(a) and (b) for mean grain size values of 15 and 50 μm, respectively. For the

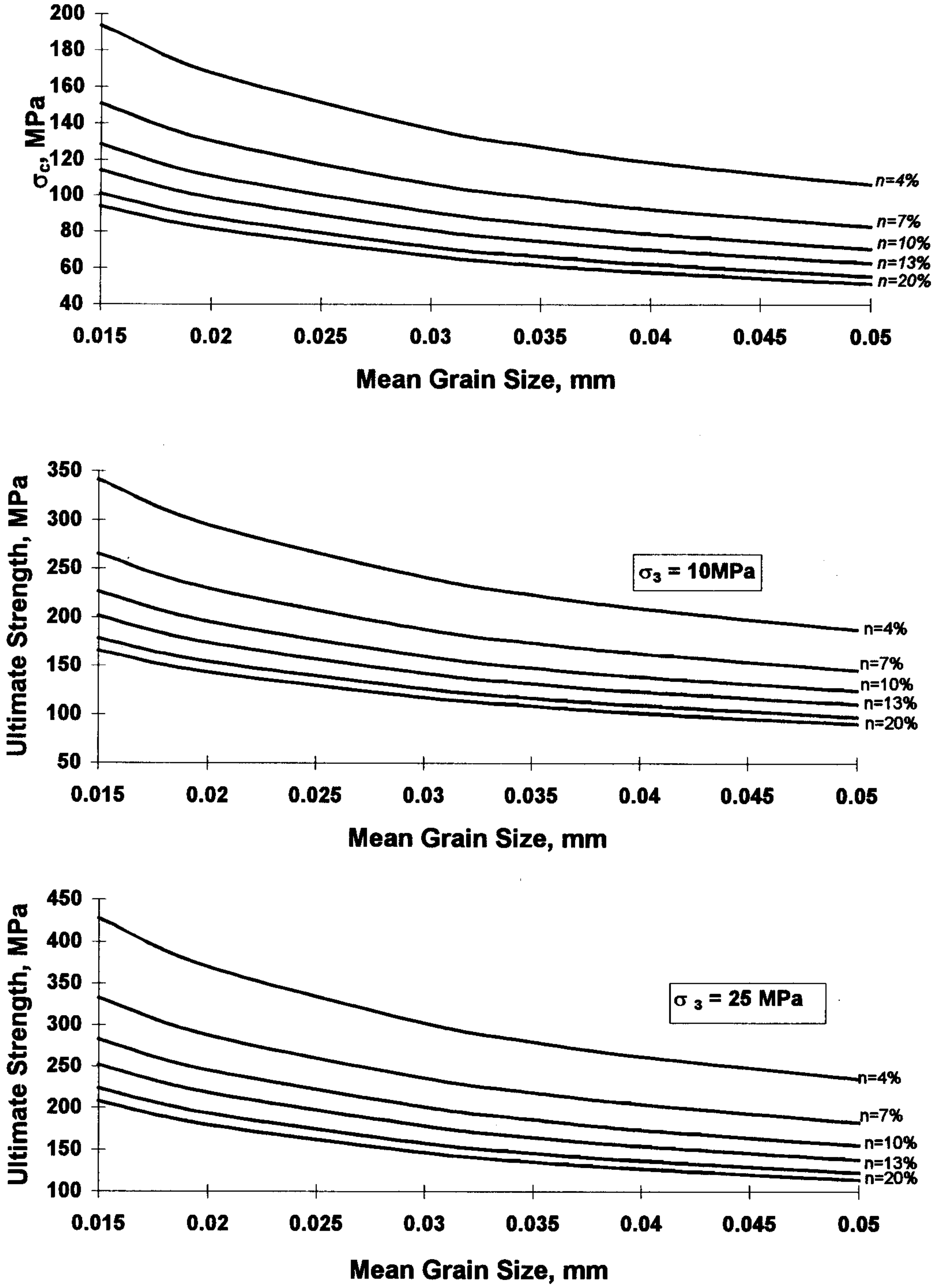

Fig. 6. Predicted influence of mean grain size and porosity on ultimate strength for rocks with a constant modulus value (E = 46 700 MPa): (a) unconfined compression, (b) $\sigma_3 = 10$ MPa and (c) $\sigma_3 = 25$ MPa.

two extreme grain size values and for every porosity value unconfined compressive strength is increased by 30% when the elastic modulus is increased from 20 to 60 GPa. Thus the strengthening effect of rock stiffness is equal for all values of initial porosity and grain size.

The strengthening effect of stiffness as a function of confining pressure is shown in Fig. 9(a) and (b) where ultimate strength is plotted against elastic modulus and porosity for confining pressures of 10 and 25 MPa, respectively, and a constant grain size value of 35 μm is assumed. When the elastic modulus is increased from 20 to 60 GPa, the ultimate strength increases by 30% for all porosity values and under both levels of confining pressure, a similar strengthening effect of rock stiffness as predicted for unconfined compression for all values of mean grain size.

Finally, the strengthening effect of confining pressure is shown in Fig. 10 where ultimate strength as a func-

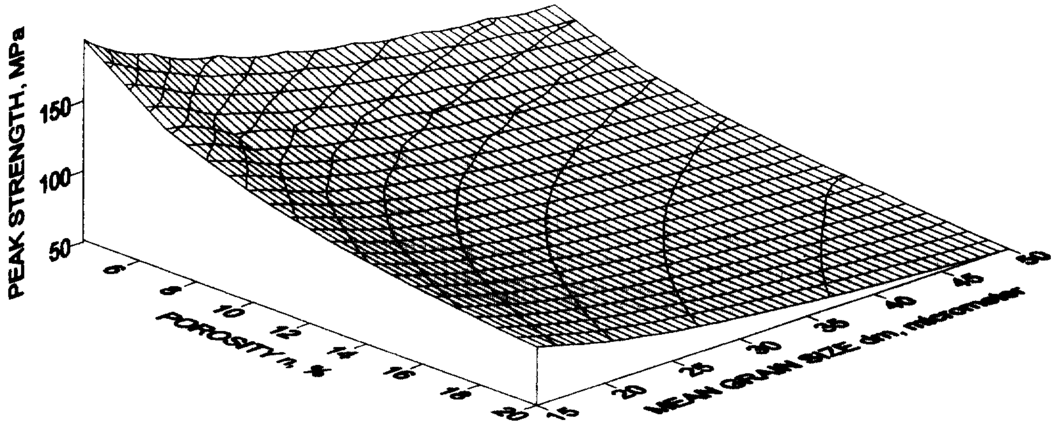

Fig. 7. The shape of the surface (σ_c, n, d_m) as predicted by the proposed model for unconfined compression.

tion of mean grain size and porosity is plotted for three levels of confining pressure. Clearly when the rock is subjected to a small confining pressure (1 to 10 MPa) ultimate strength rapidly increases, this effect is restrained with increasing levels of confining pressure.

SUMMARY AND CONCLUSIONS

The mechanical strength of dolomite rock material is studied using enhanced petrographic and microstructural analysis coupled with triaxial compression tests of 18 fully monitored samples performed in a stiff, servo controlled, load frame. Dolomites are selected as representatives of real rock: they are crystalline, brittle and exhibit linear elastic deformation up to the fracture initiation stress. The influence of heterogeneity on mechanical behaviour is studied in detail as it is argued that real rocks are truly inhomogeneous: they typically consist of initial flaws (pores and grain

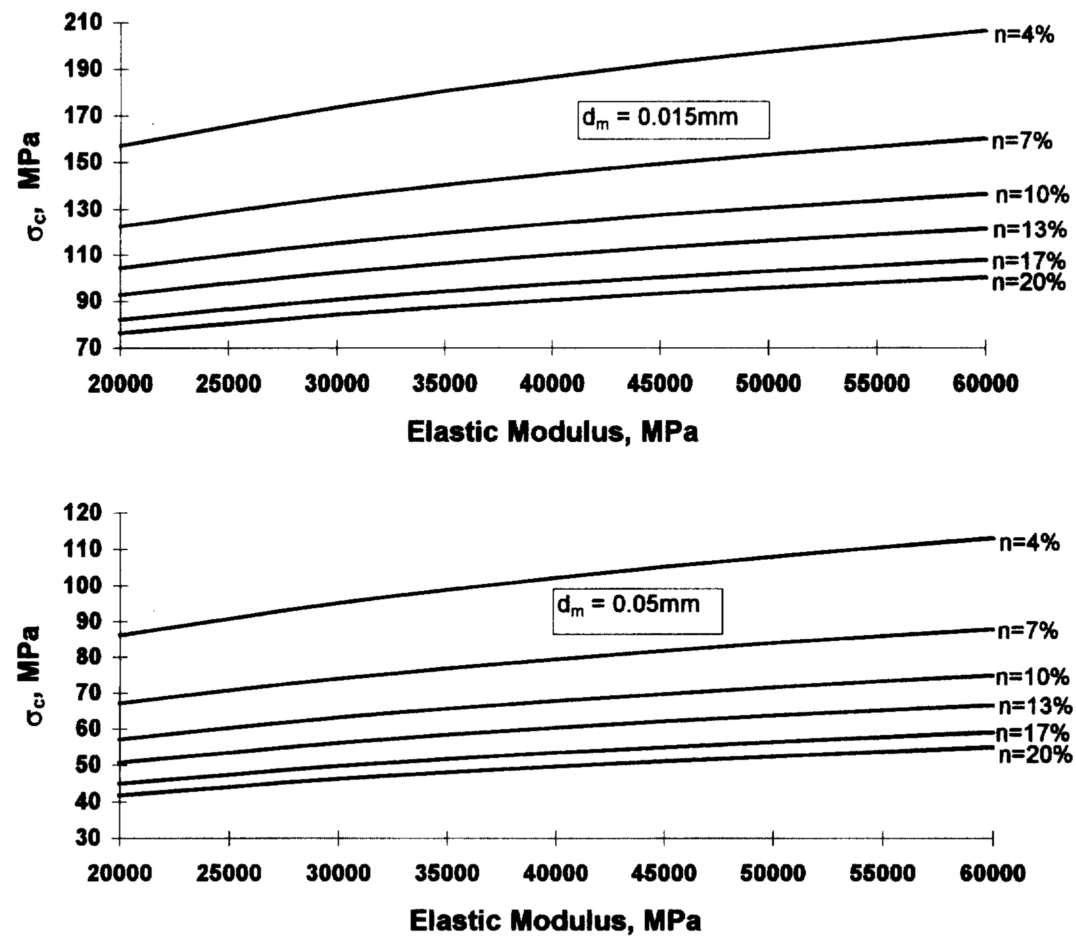

Fig. 8. Influence of stiffness variations on ultimate strength as predicted by the proposed model for unconfined compression: (a) $d_m = 0.015$ mm and (b) $d_m = 0.05$ mm.

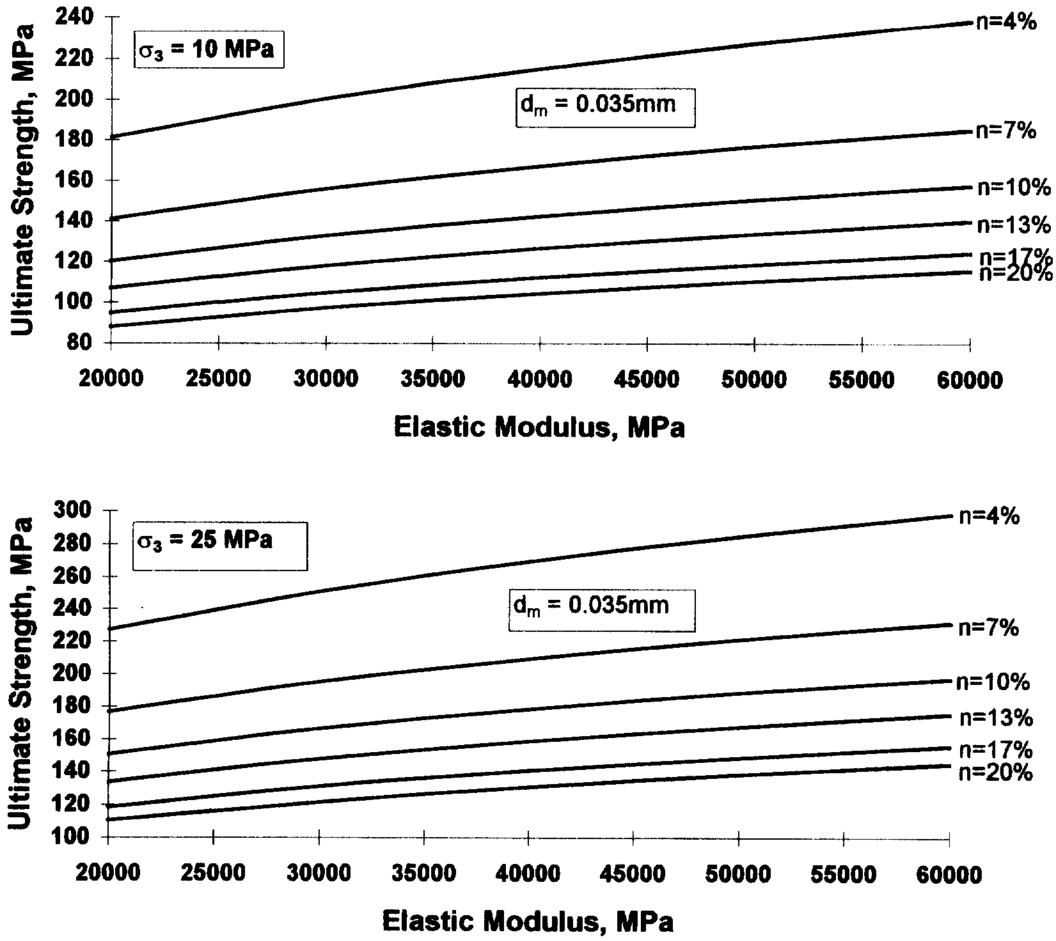

Fig. 9. Influence of stiffness variations on ultimate strength as predicted by the proposed model for triaxial compression, for rocks with equal mean grain size value ($d_m = 0.035$ mm): (a) $\sigma_3 = 10$ MPa and (b) $\sigma_3 = 25$ MPa.

boundaries) which vary in size and shape, consist of varying proportions of fine grained matrix and exhibit different types of crystalline mosaic texture.

The most popular textural parameter that has been researched to date is mean grain size. It has been argued that because stress concentration at the edge of initial flaws is proportional to the square root of the initial flaw length an inverse square root relationship should exist between mean grain size and yield or ultimate strength [1–3, 11]. It is shown here that mean grain size cannot be singled out as the primary microstructural parameter which influences rock strength for two reasons: (1) initial pores also function as stress concentrators and as fracture nucleation sites [4, 5] and therefore initial porosity must be considered as well and (2) while fracture initiation stress may be sensitive to the length and distribution of initial flaws, fracture propagation and coalescence mechanisms, which ultimately lead to macroscopic failure are influenced by the relative abundance of fine grained matrix in the sample. It is concluded that previous workers which have reported exceptionally good correlation between mean grain size and ultimate strength (or yield stress) must have tested extremely homogenous samples in which the only textural variable was the mean grain size. This, however, is rarely the case in brittle rocks in general and particularly in carbonate rocks [12, 13].

Porosity is shown here to be inversely related to strength. This observation suggests that initial pores, like initial grain boundaries, function as stress concentrators and as fracture nucleation sites.

Rock stiffness, which is scaled by the elastic modulus, is viewed here as a manifestation of grain arrangement. A microstructural parameter which inversely influences rock stiffness is the relative proportion of fine grained matrix in the rock. It is argued that with increasing proportions of fine grained matrix fracture coalescence requires smaller amounts of mechanical energy due to the abundant availability of propagation paths. Therefore ultimate strength is inversely related to rock stiffness.

A new model for ultimate strength in brittle intact heterogeneous rock is developed using mean grain size, porosity, elastic modulus, confining pressure and three empirical constants. Ultimate strength is shown to be inversely related to the square root of mean grain size and to porosity and directly proportional to the elastic modulus and confining pressure. The empirical parameters (a, b and c) are lithology dependent and must be evaluated analytically.

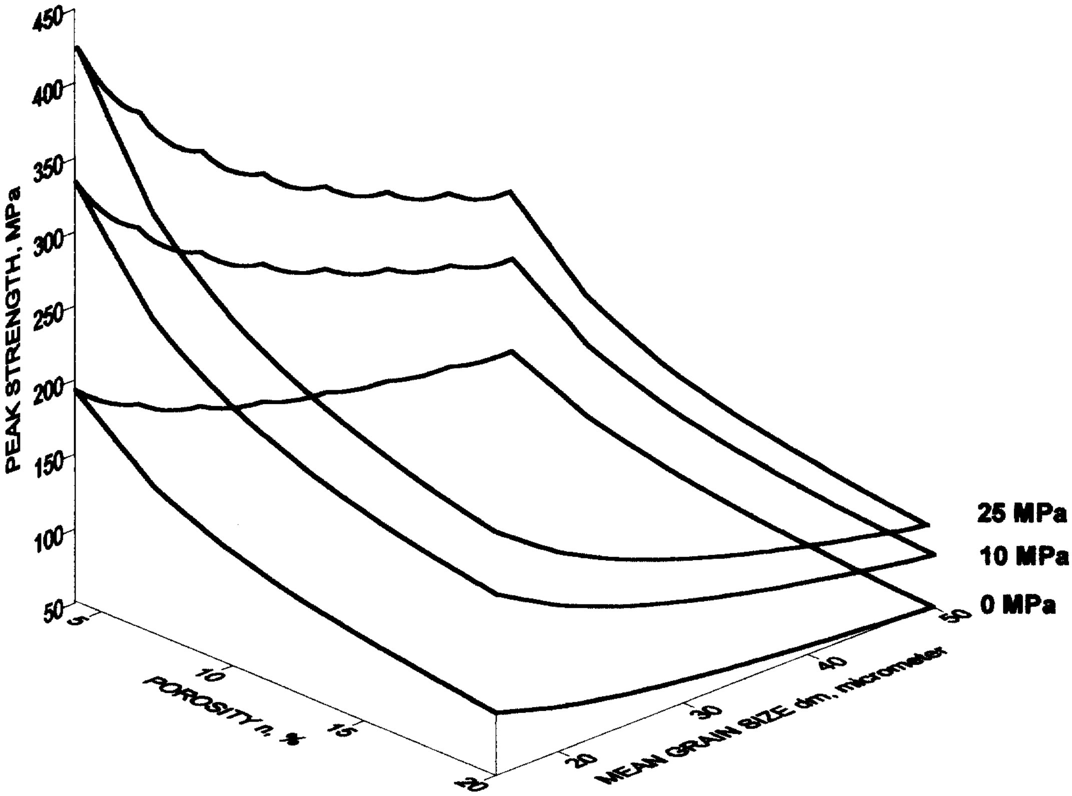

Fig. 10. Predicted influence of confining pressure on ultimate strength for rocks with varying porosity and mean grain size values. The surfaces are calculated for an equal modulus value of 46 700 MPa.

The model predicts that for intact homogenous rocks with a constant modulus value, ultimate strength rapidly decreases with increasing porosity. This trend remains the same for all mean grain size values and for all levels of confining pressure. The effect of mean grain size on ultimate strength is similar yet less pronounced and remains the same for all values of bulk porosity and confining pressure. For heterogeneous rock with varying stiffness the model predicts an increase in ultimate strength by 30% when the modulus increases from 20 to 60 GPa. This effect remains the same for all values of initial porosity and grain size. The model predicts that the strengthening effect of confining pressure on heterogeneous rocks is not linear. When the rock is subjected to low confining pressure (1 to 10 MPa) ultimate strength rapidly increases, this effect is restrained with increasing levels of confining pressure.

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