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Strength and fracture toughness estimation from four-point bending test

✉ Aurélien Doitrand¹, Ronan Henry¹, and ✉ Sylvain Meille¹

¹ Université Lyon, INSA-Lyon, MATEIS UMR CNRS 5510, F-69621 Villeurbanne Cedex, France

The failure stress under four-point bending cannot be considered as an intrinsic material property because of the well-known size effect of increasing maximum flexural stress with decreasing specimen size. In this work, four-point bending tests are analyzed with the coupled criterion for different sample sizes. The maximum flexural stress only tends towards the material tensile strength provided the specimen height is large enough as compared to the material characteristic length. In that case, failure is mainly driven by a stress criterion. Failure of smaller specimens is driven both by energy and stress conditions, thus depending on the material tensile strength and fracture toughness. Regardless of the material mechanical properties, we show that the variation of the ratio of maximum flexural stress to strength as a function of the ratio of specimen height to material characteristic length follows a master curve, for which we propose an analytical expression. Based on this relation, we propose a procedure for the post-processing of four-point bending tests that allows determining both the material tensile strength and fracture toughness. The procedure is illustrated based on four-point bending experiments on three gypsum at different porosity fractions.

Keywords Strength, Four-point bending, Coupled criterion

1 Introduction

Size effect refers to the influence of the characteristic structure dimension on the nominal failure stress. Considering four-point bending as a matter of example, it is usually observed experimentally that the smaller the specimen the larger the maximum flexural stress. An explanation of this size effect relying on the weakest link theory was introduced by Weibull (Weibull 1939; Weibull 1949; Weibull 1951), based on the idea that failure is driven by flaws inside the materials and that the larger the specimen, the larger the probability for a large flaw to exist in the specimen. This approach was later on refined and justified based on statistical distribution of microscopic flaws. It is used for describing the size effect in the fracture of brittle solids (Bermejo et al. 2014). However, it also suffers from some drawbacks since some tests revealed either a stronger (Bažant and Planas 1998) or weaker (Lu et al. 2004) size effects than predicted by Weibull theory. Another objection to this purely statistical approach is that it does not contain any material characteristic length (Bažant 1999).

To avoid this limitation, the idea of combining the theory of plasticity (characterized by the material strength σ_c or yield limit) and Linear Elastic Fracture Mechanics (LEFM, characterized by the fracture energy \mathcal{G}_c) was proposed. While both approaches do not contain any characteristic length, their combination does through the material characteristic length $\frac{E\mathcal{G}_c}{\sigma_c^2}$, where E is the material Young's modulus. This idea led to the possibility of a deterministic size effect that could be an alternative explanation to the statistical size effect. Such deterministic size effect was extensively studied by Bazant et al. (Bažant 1999; Bažant 1984; Bažant and Pfeiffer 1987; Bažant and Xi 1991).

LEFM is mainly limited by the basic assumption of a pre-existing crack preventing the assessment of crack initiation. To overcome this drawback, the coupled criterion (CC) was developed by Leguillon (Leguillon 2002) in order to study crack initiation Weißgraeber et al. 2016. It is basically founded on the simultaneous fulfillment of both energy and stress conditions. Coupling these two conditions allows retrieving the material characteristic length and thus reproducing deterministic size effects. A tool for the finite element implementation of the CC has recently been developed (Doitrand, Martin, et al. 2020).

Crack initiation in laminates (Parvizi et al. 1978) was the first example of size effect assessed with the coupled criterion (Leguillon 2002), which was also studied by García et al. (I. G. García, Carter, et al. 2016; I. G. García, Mantić, et al. 2018; I. García et al. 2019) in 3D, accounting for residual stresses or comparing the CC with other criteria. The size effect assessment by the coupled criterion offered an alternative explanation to the statistical approach. Later on, Leguillon et al. (Leguillon, Martin, and Lafarie-Frenot 2015) combined the CC and Weibull statistics in order to account for both deterministic and statistical size effects. They also pointed out that according to the CC, the tensile strength can be considered as a material parameter, contrary to the flexural strength which depends on the specimen size. Cornetti et al. (Cornetti, Pugno, et al. 2006) studied size effect under three point bending and highlighted the ability of the coupled criterion to reproduce the experimentally observed size effect and catch the concave-convex transition

in bi-logarithmic plot of flexural stress as a function of specimen size when passing from un-notched to notched specimen. Size effect of the notch radius on the initiation loading at blunt notches under pure opening mode was studied by Leguillon *et al.* (Leguillon, Quesada, *et al.* 2007) and Carpinteri *et al.* (Carpinteri *et al.* 2011) using either the classical stress criterion (Leguillon, Quesada, *et al.* 2007) or its averaged version (Carpinteri *et al.* 2011). Size effect on the failure stress in notched structures under mixed mode was studied by Cornetti *et al.* (Cornetti, Sapora, and Carpinteri 2013), considering either self-similar specimens containing a re-entrant cornered hole or square holed configuration undergoing various mixed mode loading configurations. The CC also allows studying the size effect of holed specimens (Leguillon, Quesada, *et al.* 2007; Cornetti, Sapora, and Carpinteri 2013; Doitrand, Estevez, *et al.* 2019; Doitrand and Sapora 2020; Martin, Leguillon, and Carrère 2012; Cornetti and Sapora 2019). The influence of flaw size in ceramics under tensile loading was assessed by Leguillon *et al.* (Leguillon, Martin, Seveček, *et al.* 2018) and Martin *et al.* (Martin, Leguillon, Seveček, *et al.* 2018), showing a transition between a constant to decreasing apparent tensile strength depending on the flaw size relatively to the material characteristic length. Size effect is particularly marked in case of small-scale specimen testing. Indeed, the smaller the specimen, the smaller the energy available for crack initiation and thus the larger the loading required to fulfill the energy condition necessary for crack initiation, which may result in large local stress levels compared to the material tensile strength. For instance, stresses up to 5 GPa are locally reached in micron-scale alumina platelets under three-point bending (Feilden *et al.* 2017), the material exhibiting a ≈ 1 GPa tensile strength considering the CC. The application of the CC allows a correct prediction of the failure force and demonstrates that in such configuration, failure is monitored by the energy condition involving the platelet fracture toughness (Doitrand, Henry, Chevalier, *et al.* 2020). The efficiency of the CC to predict crack initiation at small-scale was also recently illustrated on silicon nanoscale cantilevers (Gallo *et al.* 2020) and UO_2 micro-cantilevers loaded in flexion (Doitrand, Henry, Zacharie-Aubrun, *et al.* 2020). This work is focused on **deterministic** size effect in four-point bending samples without notch, studied experimentally by tests on gypsum and assessed numerically using the coupled criterion. The objective of the paper is to establish a relation between the maximum flexural stress at failure (calculated from the measured failure force), the specimen dimensions and the material properties, namely Young's modulus, fracture toughness and tensile strength.

Section 2 is dedicated to the experimental characterization of gypsum specimens: manufacturing and testing under four-point bending. In Section 3 we present the coupled criterion for crack initiation modeling. In section 4, we use the CC to assess numerically the size effect, which allows establishing a relation between the maximum stress locally reached, the specimen size and material parameters in Section 5. We thus propose a post-processing approach for the determination of \mathcal{G}_c and σ_c based on four-point bending experiments.

2 Experiments

The material under investigation is gypsum. We study three shades of gypsum: α -gypsum, β -gypsum and a dental gypsum referred to as γ -gypsum in the following. The main difference between these three shades is their pore fraction and density, their properties are given in Tab. 1. Manufacturing of samples is made by manually mixing hemihydrate powders with tap water at 23°C. Depending on the powder type, the amount of water varies, leading to different final porosity fraction. γ , α and β samples are made from a type 4 dental gypsum (Fujirock), Prestia model (Lafarge) and a powder supplied in a store retail respectively. **The specimens exhibit typical interconnected pores between the gypsum crystals of the micrometer size as well as some macropores (Adrien *et al.* 2016)** The purity of the powders is larger than 96%. Young's modulus has been determined with ultrasonic method (Grindo-sonic) and Poisson's ratio is estimated for porous gypsum as shown in (Meille and Garboczi 2001; Sanahuja *et al.* 2010).

Material	Pore fraction	density ρ (g/cm ³)	Young's modulus E (GPa)	Poisson's ratio ν
γ -gypsum	0.15	1.94 \pm 0.03	38 \pm 1	0.21
α -gypsum	0.30	1.59 \pm 0.03	16 \pm 0.5	0.21
β -gypsum	0.50	1.03 \pm 0.03	3.8 \pm 0.5	0.21

Table 1: Material properties of α -, β - and γ -gypsum (Meille and Garboczi 2001; Meille 2001; Sanahuja *et al.* 2010).

It can be observed that the Young's modulus is strongly dependent on pore fraction. Similar relations can be found for the tensile strength σ_c and critical stress intensity factor K_{Ic} . The tensile strength and critical stress intensity factor were determined experimentally (α - and β -gypsum) and estimated (γ -gypsum) based on the gypsum pore fraction (Meille and Garboczi 2001; Sanahuja *et al.* 2010; Meille 2001):

$$\cdot \gamma\text{-gypsum: } \sigma_c \approx 12 \pm 2 \text{ MPa}, K_{Ic} \approx 0.4 \pm 0.05 \text{ MPa.m}^{0.5}$$

- α -gypsum: $\sigma_c \approx 9.5 \pm 1.5$ MPa, $K_{Ic} \approx 0.35 \pm 0.03$ MPa.m^{0.5}
- β -gypsum: $\sigma_c \approx 2.7 \pm 1$ MPa, $K_{Ic} \approx 0.1 \pm 0.03$ MPa.m^{0.5}

Several specimens of each gypsum shade are tested under four-point bending. The material exhibits a linear elastic behavior until brittle failure. Different specimen heights are used in order to assess a possible size effect. Accordingly, two bending apparatus (with ($L_{span} = 90.5$ mm, $l_{span} = 30$ mm) and ($L_{span} = 35$ mm, $l_{span} = 10$ mm) lower and upper spans distance respectively) have been used depending on the specimen height. The specimen dimensions are given in Appendix in Tabs. 6 (α -gypsum), 7 (β -gypsum) and 8 (γ -gypsum) together with the failure force F_c and the corresponding maximum flexural stress σ_{max} , calculated as:

$$\sigma_{max} = \frac{3 F_c (L_{span} - l_{span})}{2 t h^2}, \quad (1)$$

where t is the specimen thickness and h the specimen height.

The specimen dimensions are depicted in Fig. 1a. A size effect can be observed since the maximum flexural stress measured experimentally increases with decreasing specimen height. As mentioned in

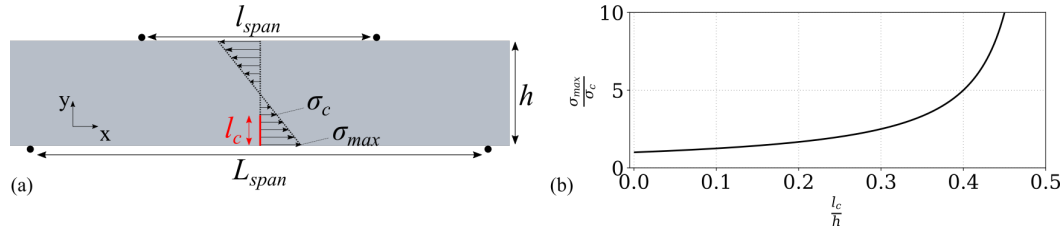


Figure 1: a) Four-point bending specimen dimensions and b) maximum stress to strength ratio as a function of the initiation crack length to specimen height ratio given in Eqn (3).

(Leguillon, Martin, and Lafarie-Frenot 2015), the maximum flexural stress (or flexural strength) cannot therefore be considered as an intrinsic material property. It can also be noted that a given specimen height, a statistical scattering is observed which is linked to the presence of macropores acting as critical flaws (Sanahuja *et al.* 2010).

The objective in the sequel is to assess numerically the size effect and provide a post-processing approach establishing a link between the measured specimen dimensions and maximum flexural stress (h and σ_{max}) and the material properties (tensile strength σ_c and critical stress intensity factor K_{Ic} or fracture toughness \mathcal{G}_c).

3 The coupled criterion

The coupled criterion aims at predicting crack initiation by combining stress and energy conditions. It states that crack nucleation requires, on the one hand, that the stress just before crack initiation overcomes the tensile strength over the whole crack path, and on the other hand, that a sufficient amount of energy is dissipated by crack initiation. The energy condition reverts to comparing the potential energy released by crack initiation $-\Delta W = W(0) - W(l)$, where l is the crack length and W the potential energy, to the energy required for crack initiation $\mathcal{G}_c l$, where \mathcal{G}_c is the material fracture toughness. Coupling both conditions consist in determining the initiation displacement $U_0 = U_c$ and the corresponding initiation crack length l_c that simultaneously satisfy both conditions, which can thus be written as follows ($y = 0$ corresponds to the specimen mid-height, cf. Fig. 1a) :

$$\begin{cases} \sigma(y = -h + l, U_c) \geq \sigma_c \quad \forall l \leq l_c, \\ G_{inc}(l_c, U_c) = -\frac{\Delta W(l_c)}{l_c} = \mathcal{G}_c. \end{cases} \quad (2)$$

Solving the coupled criterion reverts to the determination of the minimum imposed loading U_c for which both criteria are simultaneously fulfilled. The corresponding crack length l_c is the initiation crack length. It requires the calculation of the potential energy release as a function of the crack length $-\Delta W = W(0) - W(l)$. This quantity is obtained by means of Finite Element (FE) calculations, successively unbuttoning nodes along the predefined crack path. The calculation allowing the determination of $W(0)$ corresponds to a configuration without a crack, which also enables calculating the stress variation along the crack path before crack initiation. It can be noted that the stress variation could also be obtained analytically using beam theory provided the specimen dimensions verifies the corresponding hypotheses. 2D plane strain linear elastic FE calculations are set-up employing AbaqusTM. Since the stress fields are homogeneous within the specimen thickness, the 2D analysis is expected to provide results close to 3D analysis. The boundary conditions consist in imposed displacements at the spans and support locations.

The mesh is refined in the vicinity of the crack initiation location. The minimum mesh size is set to $\frac{l_{\text{mat}}}{100}$, where $l_{\text{mat}} = \frac{E' \mathcal{G}_c}{\sigma_c^2}$ (with $E' = \frac{E}{1-\nu^2}$) is the material characteristic length.

The stress distribution is a linear function of the position along the specimen height that varies from $-\sigma_{\text{max}}$ to σ_{max} , σ_{max} being the maximum flexural stress, therefore the stress conditions gives a relation between the maximum flexural stress to strength ratio and the initiation length to specimen height ratio:

$$\frac{\sigma_{\text{max}}}{\sigma_c} = \frac{1}{1 - \frac{2l_c}{h}}, \quad (3)$$

shown in Fig. 1b. The initiation l_c is not known *a priori* and depends both on the material properties and specimen geometry but can be determined using the CC. The incremental energy release rate is a monotonic increasing function of the crack length and tends to zero when the crack size tends towards zero. Fig. 2a shows a graphical representation of the CC solution including the stress (dashed line) and the energy (thick solid line) criteria for i) a too small imposed displacement (dashed line) and ii) an imposed displacement corresponding to the initiation displacement (plain lines). The stress criterion is fulfilled for

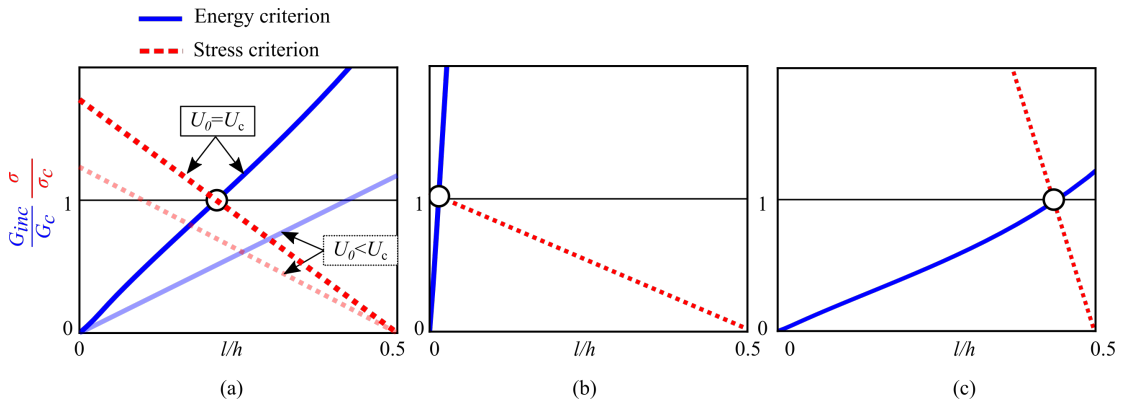


Figure 2: Stress to strength (stress criterion) and incremental energy release rate to fracture toughness (energy criterion) ratios as a function of crack length to specimen height ratio. a) Classical case, b) dominating stress criterion (small initiation length to specimen height ratio) and c) dominating energy criterion (initiation length to specimen height ratio close to 0.5).

all the lengths for which $\frac{\sigma}{\sigma_c} \geq 1$. Therefore, for a given imposed displacement U_0 , it yields an upper bound for the admissible initiation crack lengths. Similarly, the energy criterion is fulfilled for all the lengths for which $\frac{G_{\text{inc}}}{\mathcal{G}_c} \geq 1$. It thus provides a lower bound for the admissible initiation crack lengths for a given imposed displacement. For a too small imposed displacement ($U_0 < U_c$), it can be seen that both the stress and the energy criteria are satisfied for two non-intersecting ranges of crack lengths (the upper bound given by the stress criterion is smaller than the lower bound given by the energy criterion). Increasing the imposed displacement allows increasing the upper bound given by the stress criterion and decreasing the lower bound given by the energy criterion until they match for an imposed displacement $U_0 = U_c$. It thus enables determining the crack length for which both criteria are simultaneously fulfilled, *i.e.* the initiation crack length (white dot in Fig. 2).

In most of cases, crack initiation is driven by both criteria and thus depends on both \mathcal{G}_c and σ_c (Fig. 2a). Special configurations may also arise for which one of the two criteria is dominant. For instance, the stress criterion may be dominant if the initiation length is very small compared to the specimen height (Fig. 2b), therefore the maximum flexural stress is close to the material tensile strength ($l_c \ll h \Rightarrow \sigma_{\text{max}} \approx \sigma_c$). Since the initiation length is a fraction of the material characteristic length l_{mat} (Martin, Leguillon, Seveček, et al. 2018), this situation is expected for large specimens. On the contrary, the energy criterion may be dominant for initiation length close to half the specimen height (Fig. 2c), the stress criterion being almost reached over the whole area undergoing traction. This situation is rather expected for small specimens.

The CC allows computing the initiation length l_c and the imposed displacement U_c from which the failure force and corresponding failure stress are obtained by means of finite element calculations. Therefore, for given specimen geometry (h) and material properties (E , ν , \mathcal{G}_c , σ_c), the CC allows calculating the corresponding maximum flexural stress σ_{max} , which is exploited in next section in order to assess the size effect.

4 Size effect

In this section, we study the deterministic size effect related to the interaction between the initiation length predicted by the coupled criterion and the dimensions of the sample. It is not related to any statistical distribution of defects.

4.1 Size effect for given material properties

We first consider given material properties representative of α -gypsum, *i.e.* $E=16$ GPa, $\nu = 0.21$, $\mathcal{G}_c = 7.3$ J/m² and $\sigma_c = 9.5$ MPa. The corresponding material characteristic length is $l_{mat} = 1.36$ mm. The CC is used to predict failure of specimens with different heights from $0.1l_{mat}$ to $90l_{mat}$. Fig. 3 shows the initiation length (normalized by the material characteristic length - Figure 3a - or by the specimen height - Figure 3b) as a function of the ratio of the specimen height to material characteristic length $\frac{h}{l_{mat}}$.

It can be observed first that for specimen with a large height relatively to the material characteristic length ($\frac{h}{l_{mat}} \geq 20$), the initiation length reaches a plateau and is independent of the specimen size, typically leading to a situation for which the stress criterion prevails (Fig. 3a). When decreasing the specimen height, the initiation length also decreases whereas the ratio of initiation length to specimen height $\frac{l_c}{h}$ increases. When $h \rightarrow 0$, $\frac{l_c}{h} \rightarrow 0.5$ so that the energy criterion becomes more and more dominant (Fig. 3a). The classical situation involving both criteria is encountered for intermediate ratios of specimen height to material characteristic length. Therefore, the initiation length to specimen height ratio decreases from 0.5 to zero with increasing specimen height (Fig. 3b). Since the stress variation is linear as a function of the position along the specimen height and since crack initiation ensures that $\sigma(l_c) = \sigma_c$, it yields that the maximum flexural stress increases with decreasing specimen size. Fig. 4 shows the increase in the maximum flexural stress as a function of $\frac{h}{l_{mat}}$, hence reproducing qualitatively the size effect observed

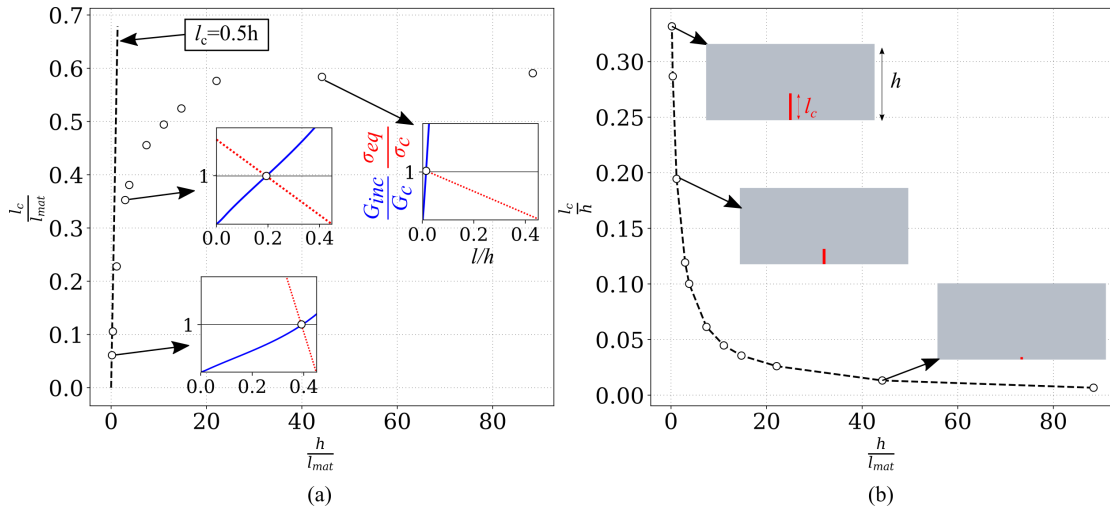


Figure 3: a) Initiation crack length to material characteristic length ratio as a function of specimen height to material characteristic length ratio. The insets show the coupled criterion in the form of stress to strength (dashed line) and incremental energy release rate to fracture toughness (thick solid line) ratios. b) Initiation crack length to specimen height ratio as a function of specimen height to material characteristic length ratio. The insets depict the size of the initiation length l_c compared to the specimen height h .

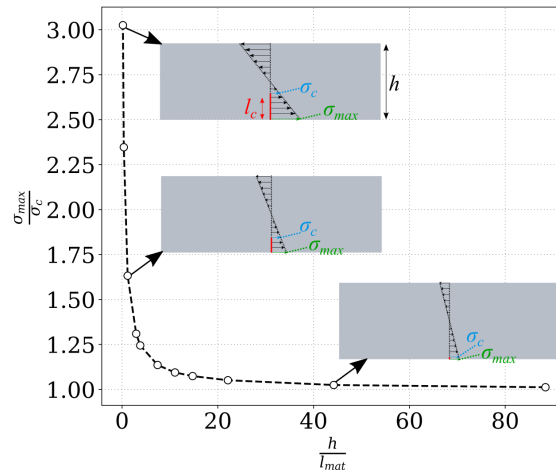


Figure 4: Maximum flexural stress to strength ratio as a function of specimen height to material characteristic length ratio. The insets depict the stress gradient along the specimen height as well as the initiation length l_c for which $\sigma(l_c) = \sigma_c$.

experimentally.

It can be noted that for large enough specimen height ($\frac{h}{l_{\text{mat}}} > 20$), the theoretical difference between the maximum flexural stress σ_{max} and the material tensile strength σ_c is smaller than 5%. For this range of $\frac{h}{l_{\text{mat}}}$, the flexural strength measurement should thus provide a good estimate of the tensile strength, which is not the case for smaller specimen height to material characteristic length ratios.

4.2 Influence of material properties on size effect

We now investigate the influence of the material fracture properties (σ_c, \mathcal{G}_c) on the observed size effect. We consider seven fracture parameter couples given in Tab. 2 corresponding to four different l_{mat} .

Property	#1	#2	#3	#4	#5	#6	#7
σ_c (MPa)	9.5	9.5	6.72	9.5	4.25	9.5	13.4
\mathcal{G}_c (J/m ²)	7.3	14.6	7.3	36.6	7.3	3.65	7.3
l_{mat} (mm)	1.36	2.72	2.72	6.79	6.79	0.68	0.68

Table 2: Strength and toughness couples leading to four different material characteristic lengths.

Fig. 5 shows the initiation length to material characteristic length ratio as a function of the specimen height obtained using the CC for the different fracture parameter couples. First, it can be observed that the same variation is obtained for material parameters giving a similar value of l_{mat} (symbols are superimposed on Fig. 5). The increase in initiation length as a function of the specimen height until a plateau for sufficiently large specimen height is observed for all the studied fracture parameter couples. Besides, the initiation length at the plateau is an increasing function of the material characteristic length, as shown in the inset of Fig. 5.

Fig. 6 shows the initiation length to specimen height ratio as a function of either the specimen height (6a) or the specimen height to material characteristic length ratio (6b). It can be noted that for a given specimen height, the same $\frac{l_c}{h}$ is obtained for different fracture parameter couples leading to the same material characteristic length. The initiation length to specimen height ratio decreases with increasing specimen height and for a fixed specimen height, the larger the material characteristic length, the larger the initiation length. Moreover, the initiation length to specimen height ratio as a function of the specimen height to material characteristic length ratio is independent of the material parameters and can thus be described by a master curve.

Fig. 7 shows the maximum flexural stress to strength ratio as a function of either the specimen height or the specimen height to material characteristic length ratio. Once again, the same variation is obtained for different fracture parameter couples leading to the same material characteristic length. The maximum flexural stress to strength ratio decreases with increasing specimen height and for a fixed specimen height, the larger the material characteristic length, the larger the maximum flexural stress. The maximum flexural stress to strength ratio as a function of the specimen height to material characteristic length ratio is independent of the material properties so that it can also be described by a master curve.

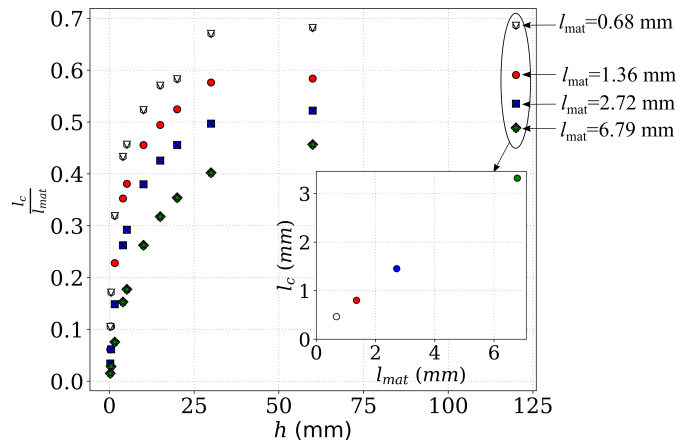


Figure 5: Initiation crack length to characteristic material length ratio as a function of the specimen size for several (\mathcal{G}_c, σ_c) couples corresponding to different material characteristic lengths l_{mat} . The inset depicts the initiation length for the largest specimen as a function of the material characteristic length.

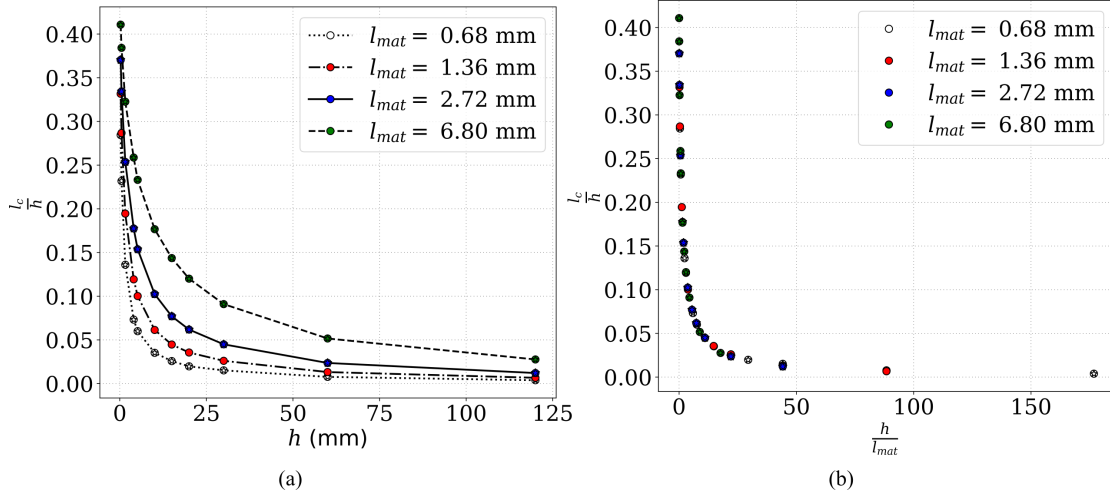


Figure 6: Initiation crack length to specimen height ratio for several $(\mathcal{G}_c, \sigma_c)$ couples corresponding to different material characteristic lengths l_{mat} as a function of a) the specimen size and b) specimen height to material characteristic length ratio.

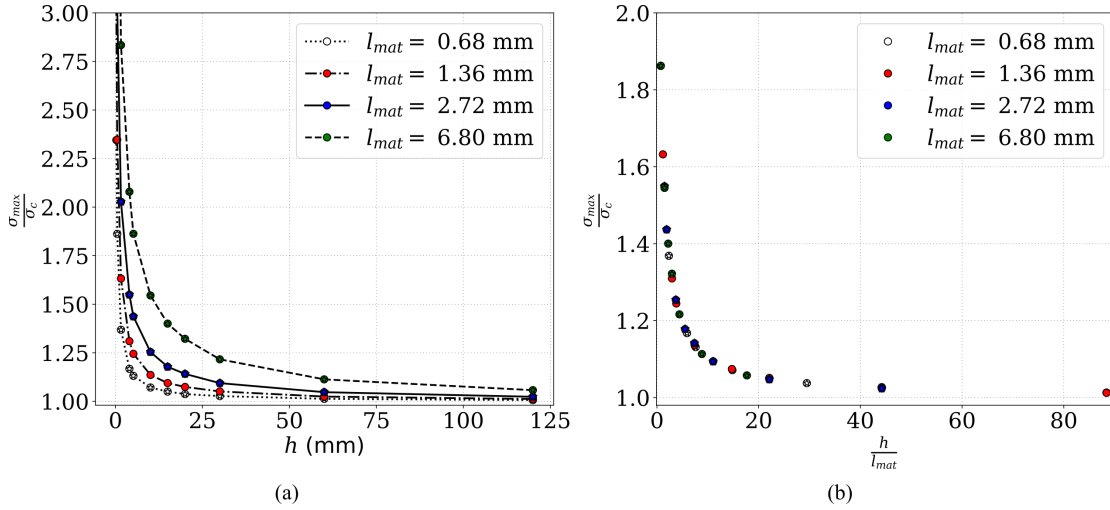


Figure 7: Maximum flexural stress to strength ratio for several $(\mathcal{G}_c, \sigma_c)$ couples corresponding to different material characteristic lengths l_{mat} as a function of a) the specimen size and b) specimen height to material characteristic length ratio.

The deterministic size effect of increasing failure stress with decreasing specimen size under bending can be explained by the interaction between the initiation length predicted by the coupled criterion and the sample dimensions. Note that even if the initiation length is related to the material characteristic length, it also depends on the specimen geometry. Therefore, the initiation length must be confronted to both the specimen height and the material characteristic length to assess the size effect. The CC predicts a unique relation between l_c/h and h/l_{mat} that is monolithically decreasing and tends towards 0 for large h/l_{mat} . In this case, the initiation length is sufficiently small so that the strength is overcome on a short distance and the bending failure stress is close to the tensile strength. Failure is thus mainly driven by the stress criterion in this case. The size effect arises from the interaction between the initiation length and the sample height. Indeed, in configurations for which the initiation length is not small with respect to the specimen height (*i.e.*, for small h/l_{mat} ratios), the stress overcomes the tensile strength over a sufficiently large distance so that the bending failure stress may be much larger than the tensile strength (as observed experimentally, *cf.* Doitrand, Henry, Chevalier, et al. 2020). Failure is driven by both stress and energy criteria in this case, the energy condition becomes more and more dominant as $h/l_{mat} \rightarrow 0$.

5 Post-processing procedure of four-point bending test

In the previous section, we highlighted that both $\frac{\sigma_{max}}{\sigma_c}$ and $\frac{l_c}{h}$ variation as a function of $\frac{h}{l_{mat}}$ follow a master curve that does not depend on the material properties. This section is dedicated to exploit these curves in order to provide a post-processing procedure for the determination of the material strength and fracture toughness based on four-point bending experiments.

5.1 Analytical expression of the master curves

The specimen height h and the maximum flexural stress σ_{\max} can be measured or obtained experimentally in four-point bending tests. We obtained a relation between $\frac{\sigma_{\max}}{\sigma_c}$ and $\frac{h}{l_{\text{mat}}}$ that does not depend on the tested material through the master curve (Fig. 7). Therefore for given σ_{\max} and h , we can adjust the material parameters so as to follow the master curve. This step could be done by interpolation of the calculated points forming the master curve (Cornetti, Pugno, et al. 2006). A more convenient approach consists in determining an analytical expression that reproduces the master curve, which we propose hereafter:

$$\frac{\sigma_{\max}}{\sigma_c} = \frac{\left(\frac{h}{l_{\text{mat}}}\right)^a + 1}{\left(\frac{h}{l_{\text{mat}}}\right)^a + b}, \quad (4)$$

with $a=0.884$ and $b=0.179$. This expression can be used in the range $\frac{h}{l_{\text{mat}}} > 0.1$ for any material under linear

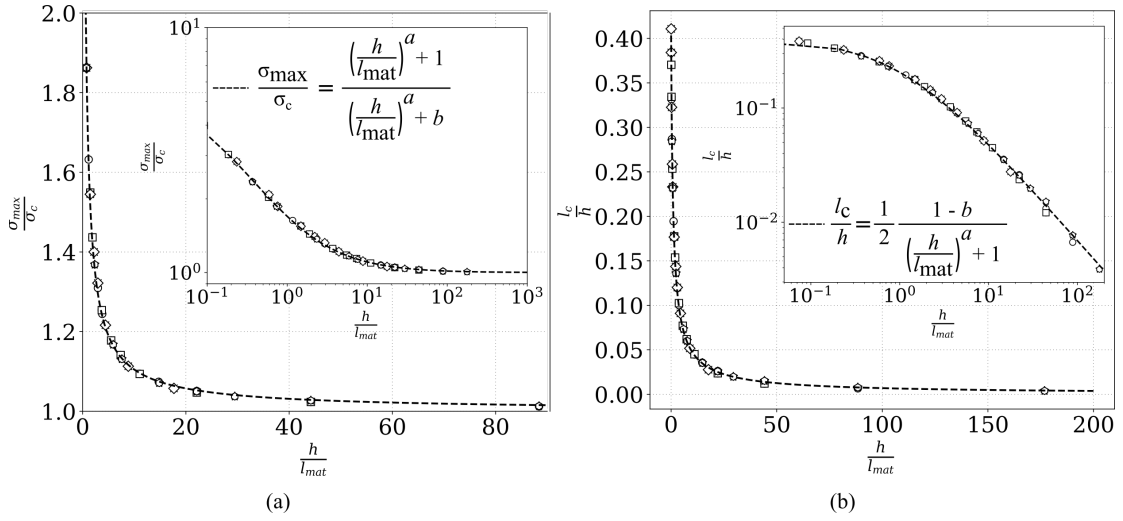


Figure 8: a) Maximum flexural stress to strength ratio and b) initiation crack length to specimen height ratio as a function of specimen height to material characteristic length ratio, also displayed as a log-log graph in the insets.

elasticity and small deformation assumption. Given the relation between σ_{\max} and l_c (Eqn. (3)), it yields

$$\frac{l_c}{h} = \frac{1}{2} \frac{1-b}{\left(\frac{h}{l_{\text{mat}}}\right)^a + 1}. \quad (5)$$

Eqs. (4) and (5) can be applied for any materials with a linear elastic behavior. Fig. 8a shows the proposed functions in classical and log-log space, which correctly reproduces the data obtained numerically to form the master curve. Rewriting the ratio $\frac{\sigma_{\max}}{\sigma_c}$ by replacing l_{mat} by $\frac{E' \mathcal{G}_c}{\sigma_c^2}$ yields

$$\frac{\sigma_{\max}}{\sigma_c} = \frac{\left(\frac{h \sigma_c^2}{E' \mathcal{G}_c}\right)^a + 1}{\left(\frac{h \sigma_c^2}{E' \mathcal{G}_c}\right)^a + b}, \quad (6)$$

which can also be rewritten as:

$$\sigma_c^{2a+1} - \sigma_{\max} \sigma_c^{2a} + \left(\frac{E' \mathcal{G}_c}{h}\right)^a \sigma_c - \sigma_{\max} b \left(\frac{E' \mathcal{G}_c}{h}\right)^a = 0. \quad (7)$$

This equation can be solved in order to determine the fracture parameter \mathcal{G}_c and σ_c . In next sections, we provide examples of the parameter determination in cases:

- i) σ_c is known,
- ii) \mathcal{G}_c is known,
- iii) neither \mathcal{G}_c nor σ_c are known.

5.2 Determination of \mathcal{G}_c

We assume in this section that the only unknown material property is \mathcal{G}_c (or K_{Ic}). The Young's modulus and strength for the different gypsums are those given in Section 2. Rewriting Eqn. (7) yields:

$$\mathcal{G}_c = \frac{h\sigma_c^2}{E'} \left(\frac{\sigma_{\max} b - \sigma_c}{\sigma_c - \sigma_{\max}} \right)^{-\frac{1}{a}} \quad (8)$$

which is an explicit expression of the quantities measured experimentally (specimen height and maximum flexural stress) and of the other material properties (E' and σ_c). Using Eqn. (8), each tested specimen provides a value of \mathcal{G}_c . Therefore, the scattering obtained on the identified values is representative of the statistical scattering for a given specimen size, which mainly depends on the presence of macropores in the case of gypsum. The values obtained for the three shades of gypsum are shown as a function of the specimen height or the specimen density for \mathcal{G}_c (Fig. 9) or K_{Ic} (Fig. 10) and the mean and standard deviation are given in Tab.3. The obtained mean value are in the order of magnitude of those taken from (Meille and Garboczi 2001; Sanahuja *et al.* 2010; Meille 2001) and recalled in Section 2. It can be noted that if only specimens large with respect to the material characteristic length are tested, failure is mainly driven by the stress condition. Therefore, the post-processing of the experiments provides a good estimate of the tensile strength but it is not expected to obtain an accurate value of the fracture toughness in this case. However, for smaller h/l_{mat} ratios, both the tensile strength and fracture toughness can be determined accurately.

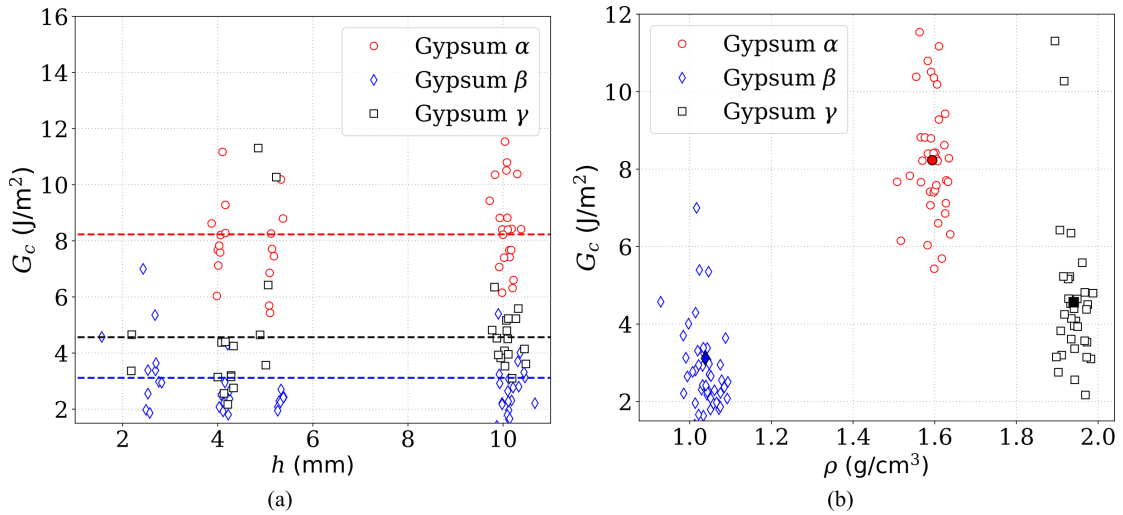


Figure 9: Fracture toughness \mathcal{G}_c identified from experimental measurements of specimen height h and maximum flexural stress σ_{\max} on gypsum specimens assuming $\sigma_c = 9.5$ J/m² (α -gypsum), $\sigma_c = 2.7$ J/m² (β -gypsum) and $\sigma_c = 12$ J/m² (γ -gypsum) as a function of specimen a) height h and b) density ρ .

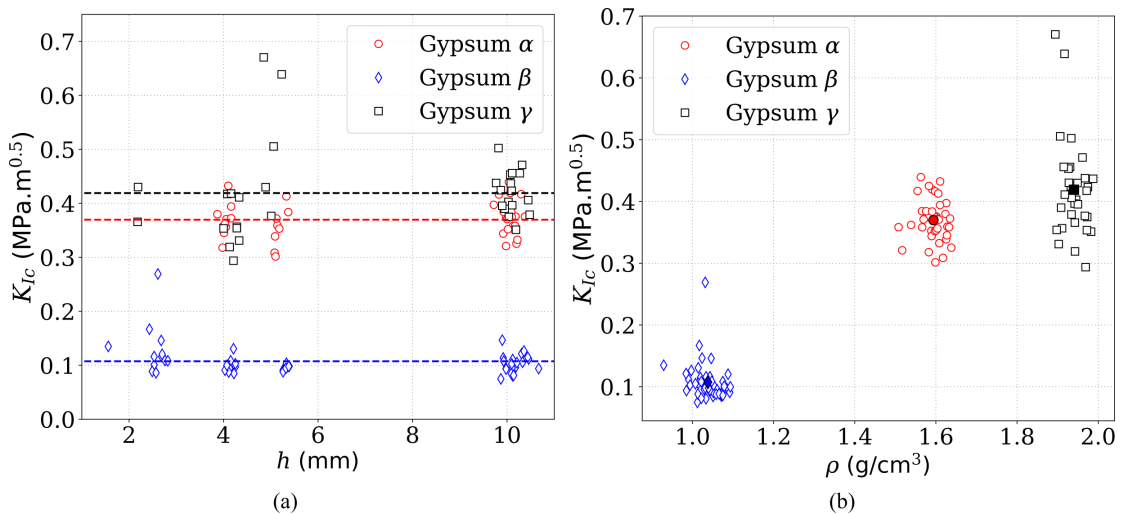


Figure 10: Critical stress intensity factor K_{Ic} identified from experimental measurements of specimen height h and maximum flexural stress σ_{\max} on gypsum specimens assuming $\sigma_c = 9.5$ J/m² (α -gypsum), $\sigma_c = 2.7$ J/m² (β -gypsum) and $\sigma_c = 12$ J/m² (γ -gypsum) as a function of specimen a) height h and b) density ρ .

Material	σ_c (MPa) (fixed)	\mathcal{G}_c (J/m ²) (identified)	K_{Ic} (MPa.m ^{0.5}) (identified)
γ -gypsum	12	4.7 ± 1.9	0.42 ± 0.078
α -gypsum	9.5	7.6 ± 1.4	0.35 ± 0.031
β -gypsum	2.7	3.0 ± 2.2	0.1 ± 0.029

Table 3: Identified fracture toughness \mathcal{G}_c and critical energy release rate K_{Ic} mean and standard deviation for given σ_c values.

5.3 Determination of σ_c

We assume in this section that the only unknown material property is the tensile strength σ_c . The Young's modulus and fracture toughness for the different gypsums are those given in Section 2. Contrary to the case for which only \mathcal{G}_c is not known which provides an explicit solution, the determination of σ_c requires solving Eqn. (7). This can be done numerically which provides a value of σ_c for each tested specimens. The values obtained for the three shades of gypsum are shown as a function of the specimen height or the specimen density in Fig. 11 and the mean and standard deviation are given in Tab.4.

Material	\mathcal{G}_c (J/m ²) (fixed)	σ_c (MPa) (identified)
γ -gypsum	4.0	11.6 ± 1.9
α -gypsum	7.3	9.5 ± 0.86
β -gypsum	2.5	2.7 ± 0.5

Table 4: Identified strength σ_c mean and standard deviation for given \mathcal{G}_c values.

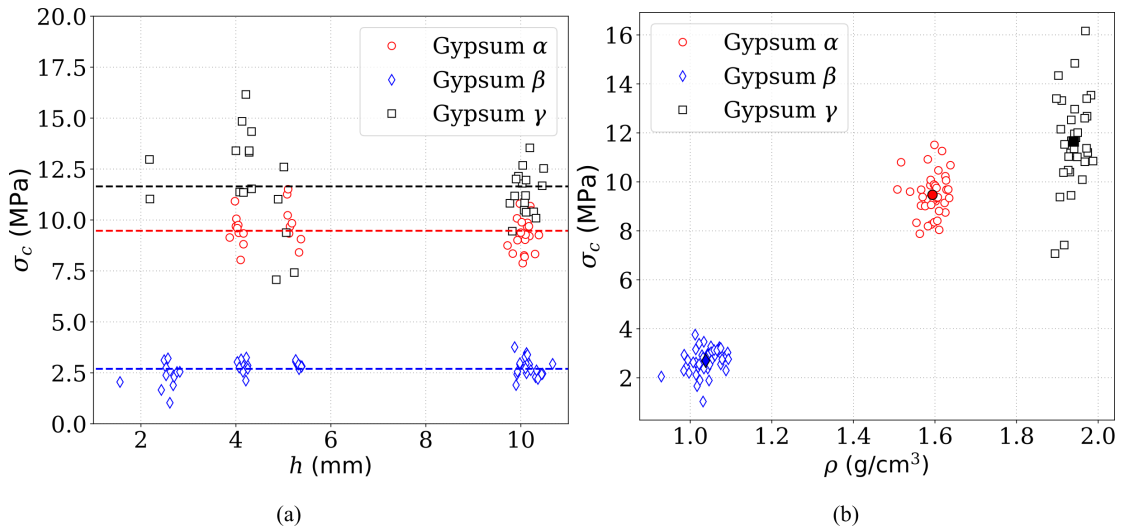


Figure 11: Strength σ_c identified from experimental measurements of specimen height h and maximum flexural stress σ_{max} on gypsum specimens assuming $\mathcal{G}_c = 7.3$ J/m² (α -gypsum), $\mathcal{G}_c = 2.5$ J/m² (β -gypsum) and $\mathcal{G}_c = 4$ J/m² (γ -gypsum) as a function of specimen a) height h and b) density ρ .

5.4 Determination of \mathcal{G}_c and σ_c

The last example concerns the case for which neither σ_c nor \mathcal{G}_c are known. It is possible to deduce simultaneously \mathcal{G}_c and σ_c based on a set of experimental results. Let us consider that N measurements of the specimen height and maximum flexural stress $\{(\sigma_{\max}^{(i)}, h^{(i)}), i = 1..N\}$ are available. Based on Eqn. (7), we define the functions:

$$R^{(i)}(\sigma_c, \mathcal{G}_c) = \sigma_c^{2a+1} - \sigma_{\max}^{(i)} \sigma_c^{2a} + \left(\frac{E' \mathcal{G}_c}{h^{(i)}}\right)^a \sigma_c - \sigma_{\max}^{(i)} b \left(\frac{E' \mathcal{G}_c}{h^{(i)}}\right)^a \quad (9)$$

which should be close to zero provided the fracture parameters are those of the studied material. Therefore, we seek the couple $(\sigma_c^*, \mathcal{G}_c^*)$ that minimizes the following residuals:

$$R(\sigma_c, \mathcal{G}_c) = \sum_{i=1}^N (h^{(i)} R^{(i)}(\sigma_c, \mathcal{G}_c))^2 \quad (10)$$

It can be noted that the residuals are pondered by the specimen height in order to balance the higher measurement uncertainty sensitivity for small specimens (see next section for more details). The minimization of the residuals is performed using a gradient descent algorithm. Fig. 12a shows the residuals for α -gypsum as a function of \mathcal{G}_c and σ_c as well as the obtained minimum (black star). Fig. 12b shows the maximum flexural stress to strength ratio as a function of specimen height to material characteristic length ratio for the identified values of \mathcal{G}_c and σ_c together with the master curve. The optimization process finally reverts to adjusting the experimental data to the master curve on Fig. 12b. The identified tensile strength and toughness corresponding to the residual minimum for each shade of gypsum are given in Tab. 5. It can be noted that the quality of strength and toughness identification depends on the specimen size related to the material characteristic length. Indeed, if only large enough ($\frac{h}{l_{\text{mat}}} > 20$) specimens are tested, it is expected that the maximum flexural stress is close to the material tensile strength, therefore the estimate of \mathcal{G}_c may not be as accurate in this case as for smaller specimens, since for this configuration failure is mainly driven by the stress criterion.

Material	\mathcal{G}_c (J/m ²) (identified)	K_{Ic} (MPa.m ^{0.5}) (identified)	σ_c (MPa) (identified)	l_{mat} (mm)
γ -gypsum	2.74	0.33	12.7	0.68
α -gypsum	6.15	0.32	9.9	1.06
β -gypsum	0.62	0.05	3.3	0.23

Table 5: Identified strength σ_c and fracture toughness \mathcal{G}_c based on four-point bending experiments.

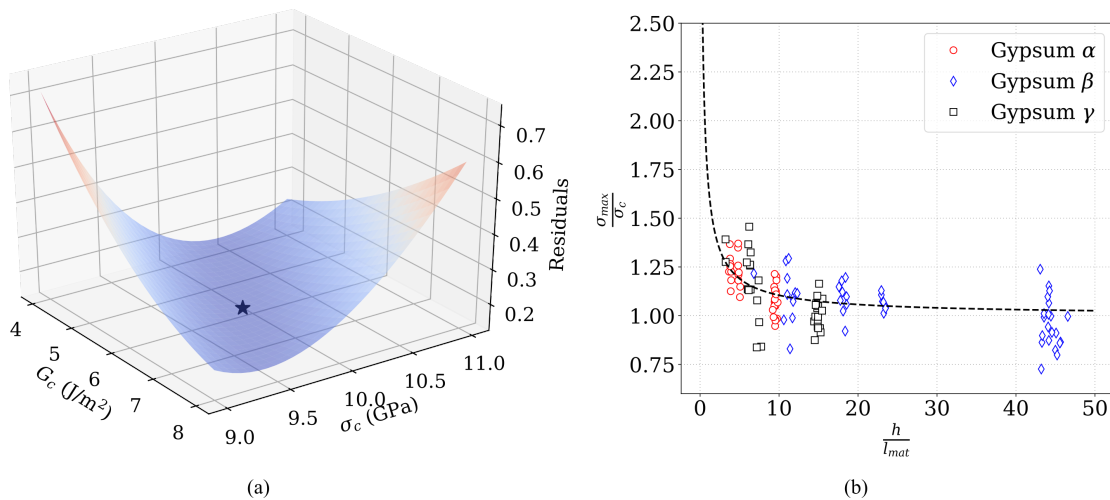


Figure 12: a) Residuals as a function of strength σ_c and fracture toughness \mathcal{G}_c (α -gypsum) and b) maximum flexural stress to strength ratio as a function of specimen height to material characteristic length ratio measured experimentally for the identified fracture properties (α -gypsum: $\mathcal{G}_c^\alpha = 6.15$ J/m², $\sigma_c^\alpha = 9.9$ MPa, β -gypsum: $\mathcal{G}_c^\beta = 0.62$ J/m², $\sigma_c^\beta = 3.3$ MPa, γ -gypsum: $\mathcal{G}_c^\gamma = 2.74$ J/m², $\sigma_c^\gamma = 12.7$ MPa corresponding to the residuals minimum.

5.5 Measurement uncertainty influence on \mathcal{G}_c and σ_c identification

We finally investigate the measurement uncertainty influence on \mathcal{G}_c and σ_c identification. The two quantities that are measured experimentally are the specimen height h and the maximum flexural stress σ_{\max} . We denote Δh and $\Delta\sigma_{\max}$ the measurement uncertainties respectively on the specimen height and the maximum flexural stress. We denote σ_c^{ref} the identified strength without uncertainty measurement ($\Delta h=0$ mm and $\Delta\sigma_{\max} = 0$ MPa).

We first consider a measurement uncertainty on the specimen height and study its influence on the strength identification. Fig. 13a shows the identified strength (taking into account the measurement uncertainty Δh) to reference strength ratios as a function of the specimen height to material characteristic length ratio. For specimens height larger than the material characteristic length, the relative uncertainty $\frac{\Delta h}{h}$ on the specimen size measurement induces an uncertainty on σ_c identification $\frac{\Delta\sigma_c}{\sigma_c^{ref}}$ smaller than $\frac{\Delta h}{h}$. The influence of the measurement uncertainty on geometry is however larger for specimens height smaller than the material characteristic length.

We now consider a measurement uncertainty $\Delta\sigma_{\max}$ on the maximum flexural stress. Fig. 13b shows the

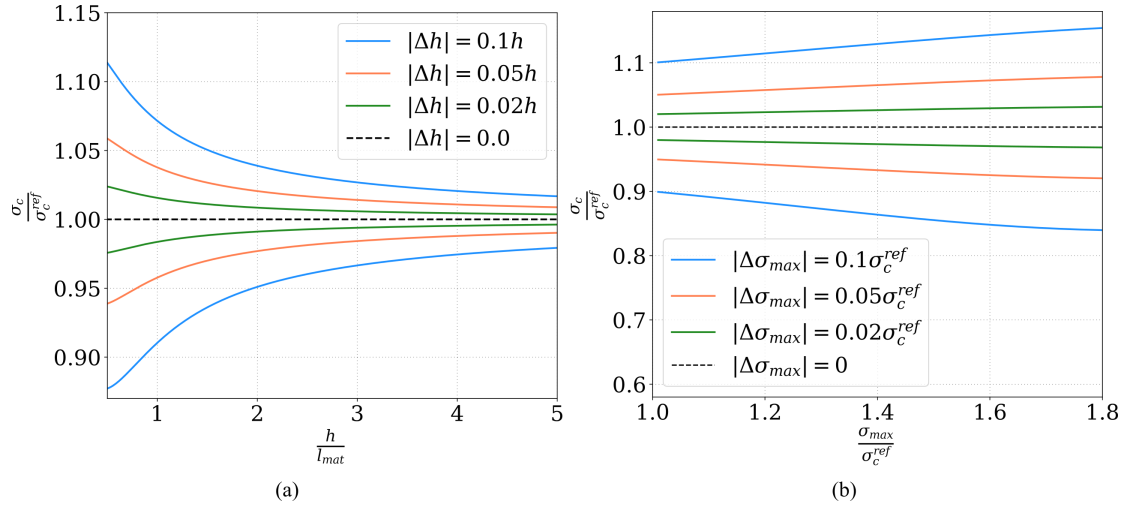


Figure 13: Uncertainty on determination of σ_c as a function of the specimen size induced by several uncertainty level on Δh and σ_{\max} .

identified strength (taking into account the measurement uncertainty $\Delta\sigma_{\max}$) to reference strength ratios as a function of the maximum flexural stress to reference strength ratio. The uncertainty measurement on the maximum flexural stress $\frac{\Delta\sigma_{\max}}{\sigma_c^{ref}}$ induces a uncertainty on the determination of σ_c approximately equal to $\frac{\Delta\sigma_{\max}}{\sigma_c^{ref}}$ for large specimen ($h \gg l_{mat}$, corresponding to ratios $\frac{\sigma_{\max}}{\sigma_c^{ref}}$ close to 1) and increasing for smaller specimens ($h \ll l_{mat}$, corresponding to ratios $\frac{\sigma_{\max}}{\sigma_c^{ref}}$ larger than 1).

6 Conclusion

The maximum flexural stress at failure under four-point bending, sometimes called flexural strength, cannot be considered as an intrinsic material property because of the well-known size effect leading to increasing maximum flexural stress with decreasing specimen size.

This deterministic size effect can be reproduced using the coupled criterion which allows the prediction of the failure force (and hence the maximum flexural stress) and the initiation crack length for given specimen geometry and material properties. The initiation length tends to a constant value for large enough specimens compared to the material characteristic length (failure driven by the stress criterion), whereas it tends to half the specimen height when the specimen height tends towards 0 (failure driven by the energy criterion). For intermediate specimen heights, failure is driven by both stress and energy conditions and thus depends on both the material tensile strength and fracture toughness. The maximum flexural stress tends to the material tensile strength for specimens large enough compared to the material characteristic length. Therefore, **according to the CC**, measurements of the flexural strength can only be considered as a material parameter (actually, the tensile strength) for large enough specimens.

We show that regardless the material under investigation (under the assumption of linear elasticity), the maximum flexural stress to strength ratio variation as a function of the specimen height to material characteristic length ratio follows a master curve, which can be described by an analytical function involving two parameters. Based on this relationship, we establish a procedure for the determination of the material tensile strength and fracture toughness which can be employed for the post-processing of

four-point bending experiments that only requires the measurement of the specimen height h and the failure force F_c in order to calculate the maximum flexural stress σ_{\max} .

7 Acknowledgement

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8 Appendix

Specimen $\alpha\#$	1	2	3	4	5	6	7	8	9	10	
h(mm)	4.03	4.05	4.16	4.16	3.98	4.00	4.10	4.01	3.87	4.06	
t (mm)	3.10	3.07	3.00	2.99	3.06	3.01	3.03	2.96	3.12	3.02	
ρ (g/cm ³)	1.53	1.6	1.63	1.61	1.58	1.63	1.61	1.62	1.62	1.60	
F_c (N)	16.61	16.76	16.74	16.09	17.43	16.00	15.08	16.19	15.07	16.15	
σ_{\max} (MPa)	12.37	12.48	12.09	11.66	13.48	12.46	11.1	12.75	12.09	12.16	
Specimen $\alpha\#$	11	12	13	14	15	16	17	18			
h(mm)	5.33	5.18	5.10	5.12	5.08	5.14	5.09	5.37			
t (mm)	10.32	10.35	10.29	9.85	10.49	10.25	10.1	10.13			
ρ (g/cm ³)	1.60	1.60	1.59	1.60	1.61	1.62	1.62	1.59			
F_c (N)	34.91	36.88	39.86	33.15	39.70	35.61	35.77	36.47			
σ_{\max} (MPa)	10.8	12.05	13.51	11.65	13.3	11.93	12.4	11.33			
Specimen $\alpha\#$	19	20	21	22	23	24	25	26	27	28	29
h(mm)	9.83	10.17	9.72	10.00	9.98	10.08	10.02	9.92	10.38	10.14	9.93
t (mm)	10.23	10.63	10.08	10.30	10.56	10.18	10.48	10.23	9.95	10.15	10.29
ρ (g/cm ³)	1.59	1.5	1.62	1.56	1.51	1.58	1.59	1.58	1.59	1.58	1.57
F_c (N)	106.7	132.6	106.6	121.3	138.8	109.7	129.3	125.6	124.5	127.7	115.8
σ_{\max} (MPa)	9.79	10.94	10.15	10.69	11.97	9.62	11.15	11.32	10.53	11.1	10.35
Specimen $\alpha\#$	30	31	32	33	34	35	36	37	38	39	
h(mm)	10.1	10.07	10.09	10.18	10.04	10.22	10.13	10.2	10.3	9.98	
t (mm)	10.12	9.87	10.03	10.16	10.09	10.22	10.15	10.09	10.18	10.35	
ρ (g/cm ³)	1.58	1.59	1.56	1.6	1.56	1.6	1.56	1.63	1.55	1.59	
F_c (N)	120.5	106.7	116.6	121.9	104.7	137.2	125.6	137.1	115.6	120.1	
σ_{\max} (MPa)	10.59	9.67	10.36	10.5	9.34	11.66	10.94	11.85	9.71	10.57	

Table 6: Dimensions, failure force F_c and stress σ_{\max} corresponding to α -gypsum specimens for $L_{span}=35$ mm and $l_{span}=10$ mm (specimens $\alpha 1$ to $\alpha 10$) or $L_{span}=90.5$ mm, $l_{span}=30$ mm (specimens $\alpha 11$ to $\alpha 39$) distances between the lower and upper spans.

Specimen $\beta\#$	1	2	3	4	5	6	7	8	9	10	11	12
h(mm)	1.56	2.57	2.82	2.76	2.53	2.61	2.7	2.69	2.68	2.49	2.53	2.43
t (mm)	6.88	7.05	7.74	7.07	7.17	7.34	6.81	7.9	7.79	7.34	6.61	6.39
ρ (g/cm ³)	0.92	1.07	1.02	1.04	1.03	1.03	1.08	1.04	1.04	1.06	1.07	1.01
F_c (N)	1.78	5.27	6.00	5.27	4.45	3.63	4.66	5.45	4.84	5.10	4.41	3.23
σ_{\max} (MPa)	3.99	4.24	3.65	3.67	3.63	2.72	3.52	3.57	3.24	4.20	3.91	3.21
Specimen $\beta\#$	13	14	15	16	17	18	19	20	21			
h(mm)	4.07	4.03	4.11	4.22	4.25	4.13	4.21	4.25	4.15			
t (mm)	2.95	2.90	2.90	2.93	2.89	2.91	2.99	2.90	2.95			
ρ (g/cm ³)	1.09	1.09	1.07	1.07	1.05	1.07	1.01	1.08	1.07			
F_c (N)	4.61	4.73	5.05	5.46	4.83	4.87	4.27	5.03	4.55			
σ_{\max} (MPa)	3.53	3.76	3.86	3.92	3.47	3.68	3.02	3.6	3.35			
Specimen $\beta\#$	22	23	24	25	26	27	28	29				
h(mm)	5.38	5.32	5.26	5.33	5.39	5.26	5.29	5.35				
t (mm)	10.22	10.1	10.21	10.27	9.75	10.1	10.28	9.97				
ρ (g/cm ³)	1.03	1.04	1.05	1.04	1.03	1.05	1.06	1.03				
F_c (N)	11.10	11.05	11.53	10.68	10.71	11.18	11.16	10.89				
σ_{\max} (MPa)	3.40	3.50	3.70	3.32	3.43	3.63	3.52	3.46				
Specimen $\beta\#$	30	31	32	33	34	35	36	37	38	39	40	
h(mm)	10.21	10.1	10.36	9.93	10.12	9.92	10.33	10.44	10.14	10.09	10.11	
t (mm)	10.67	10.98	10.42	10.6	10.57	10.32	10.98	10.48	10.36	10.69	10.43	
ρ (g/cm ³)	1.01	0.99	1.00	1.03	1.04	1.03	1.01	1.02	1.02	1.05	1.05	
F_c (N)	36.78	38.17	32.29	33.94	34.18	31.69	38.60	35.52	43.43	43.19	38.82	
σ_{\max} (MPa)	3.00	3.09	2.62	2.95	2.87	2.83	2.99	2.82	3.70	3.60	3.30	
Specimen $\beta\#$	41	42	43	44	45	46	47	48	49	50		
h(mm)	9.98	9.98	10.12	10.18	9.87	10.67	10.12	10.31	9.9	10.46		
t (mm)	10.46	10.44	10.45	10.34	10.79	10.57	10.47	10.73	10.84	10.66		
ρ (g/cm ³)	1.04	1.05	1.01	1.03	1.01	0.99	1.03	0.98	1.02	0.99		
F_c (N)	38.04	37.31	41.20	38.65	47.07	43.35	44.70	33.94	27.91	36.47		
σ_{\max} (MPa)	3.32	3.26	3.49	3.27	4.06	3.27	3.78	2.70	2.38	2.84		

Table 7: Dimensions, failure force F_c and stress σ_{\max} corresponding to β -gypsum specimens for $L_{span}=35$ mm and $l_{span}=10$ mm (specimens β_1 to β_{21}) or $L_{span}=90.5$ mm, $l_{span}=30$ mm (specimens β_{22} to β_{50}) distances between the lower and upper spans.

Specimen $\gamma\#$	1	2	3	4	5	6	7	8	
h(mm)	2.19	2.18	4.16	4.00	4.28	4.21	4.13	4.08	
t (mm)	7.07	9.64	3.04	3.03	2.97	2.99	2.99	2.92	
ρ (g/cm ³)	1.92	1.94	1.94	1.97	1.89	1.96	1.94	1.97	
F_c (N)	14.65	21.59	20.15	20.91	23.25	19.39	31.37	18.70	
σ_{\max} (MPa)	16.2	17.67	14.36	16.17	16.02	18.49	17.35	14.42	
Specimen $\gamma\#$	9	10	11	12	13	14	15	16	
h(mm)	4.33	4.28	4.33	5.06	5.01	4.85	4.89	5.23	
t (mm)	3.04	3.07	3.05	10.21	10.13	10.37	9.93	10.26	
ρ (g/cm ³)	1.9	1.91	1.91	1.9	1.96	1.89	1.94	1.91	
F_c (N)	25.57	23.94	21.97	35.37	42.03	28.59	35.84	33.05	
σ_{\max} (MPa)	16.82	15.96	14.4	12.27	15.00	10.63	13.69	10.68	
Specimen $\gamma\#$	17	18	19	20	21	22	23	24	
h(mm)	10.45	9.82	10.1	9.9	10.11	10.07	10.32	10.19	
t (mm)	10.01	10.08	9.50	10.09	10.08	10.06	10.05	10.02	
ρ (g/cm ³)	1.93	1.93	1.97	1.94	1.91	1.92	1.96	1.98	
F_c (N)	156.95	118.99	134.89	146.02	134.95	134.78	136.96	169.50	
σ_{\max} (MPa)	13.03	11.10	12.63	13.40	11.88	11.99	11.61	14.78	
Specimen $\gamma\#$	25	26	27	28	29	30	31	32	33
h(mm)	10.27	10.08	10.04	9.95	9.87	9.77	10.48	10.11	10.03
t (mm)	9.85	9.75	10.05	10.11	10.12	10.15	10.03	10.05	10.04
ρ (g/cm ³)	1.93	1.98	1.97	1.90	1.93	1.96	1.93	1.94	1.94
F_c (N)	136.10	134.45	156.30	149.08	137.36	131.61	167.67	150.84	146.84
σ_{\max} (MPa)	11.88	12.31	14.00	13.51	12.64	12.32	13.81	13.32	13.19

Table 8: Dimensions, failure force F_c and stress σ_{\max} corresponding to γ -gypsum for $L_{span}=35$ mm and $l_{span}=10$ mm (specimens γ_1 to γ_{11}) or $L_{span}=90.5$ mm, $l_{span}=30$ mm (specimens γ_{12} to γ_{33}) distances between the lower and upper spans.

9 Supplementary materials

The supplementary materials are available at the following link: <https://doi.org/10.5281/zenodo.4271971>

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