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The influence of microstructure geometry on the scale effect in mechanical behaviour of heterogeneous materials

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Abstract: There are a significant number of factors which have impact on the scale effect in the mechanical behaviour of composite materials. In this paper, the influence of the microstructure on this phenomenon is examined. In particular, how the results of the uniaxial compression test are affected by the microstructure geometry is verified. For the purposes of this study, two different materials are chosen, i.e. pure gypsum and mortar. Firstly, the microstructures of the two considered materials are compared with the use of different microstructure measures, i.e. attenuation profiles, porosity and pore size distributions, calculated based on the images obtained from the X-ray microCT. Then, a series of uniaxial compression tests is performed for a large number of cylindrical specimens made of the two materials under study. Four different sample diameters are assumed in order to investigate the size effect in the considered composites. For both materials, the results of uniaxial compressive strength and the Young modulus are presented. The relationship between the microstructure of the material and the scale effect in mechanical properties is proved. The scale effect is more demonstrable in the case of the material which exhibits a more heterogeneous microstructure.

Keywords: composite; computed tomography; microstructure; scale effect; uniaxial compression.

1 Introduction

For several years, composite materials have taken a significant part in the realisation of structures designed for civil engineering, transport, medicine, etc. Recently, a large number of attempts have been made in order to investigate the scale effect in composites, geomaterials, rocks, etc. Alzebedeh et al. [1] have investigated the scale effect in the process of fracture of random matrix-inclusion composites. Several scales (by investigation of different “windows of observation”) and statistics of the corresponding scale-dependent effective stiffness and strength of aforementioned composites have been studied. In the work of Sutherland et al. [2] the size and scale effects in the prediction of strength of fibre-reinforced composites were investigated. In addition, some basic principles in the evaluation of scaling laws are presented. The paper indicates also the problem whether a separately manufactured specimen could be comparable to the specimen cut from the full-scale structure. The same authors focused on the size and scale effect in the tensile and flexural strength of unidirectional [3] and woven-roving [4] laminates. It has been shown that the properties of considered laminates are influenced by the scale of production. The scale effect in the mechanical behaviour of rocks has been widely discussed in the literature [5–10]. In the paper of Pisarenko and Gland [11] a scale effect of damage in cemented granular rocks is investigated. A probabilistic model of damage of such materials has been proposed. The model exhibits an explicit scale dependence of certain macroscopic parameters. The most remarkable conclusion deriving from those considerations is that during the interpretation of numerical simulations of granular materials the scale effect should be taken into account. In addition, this effect should be accounted for in attempts to extrapolate the mechanical and transport properties of granular rock from laboratory to larger scales. Pan et al. [12] studied a failure process and scale effect using cellular automata. A considered material was a rock specimen under uniaxial compression. In the study, the scale effect was treated and investigated as a problem consisting of two separate

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aspects, namely, size effect and shape effect. The size effect was studied by comparing the results obtained for samples with the same length/diameter ratio but with different diameters. On the other hand, the shape effect was investigated by keeping the diameter of the sample constant and changing the length of the sample (different length/diameter ratios). The results of numerical calculations showed that the longer the specimen, the more brittle the material becomes after the peak stress. Another important conclusion is that, as the length/diameter ratio is $>2:1$, the strength of the rock specimen becomes stable. Based on the laboratory results, Zhang et al. [13] proposed a new expression to evaluate the dependence of the uniaxial compressive strength on the specimen volume. It was shown that, for different rocks, the proposed relation can fit the uniaxial compressive strength versus specimen size data very well. In the paper of Poulsen and Adhikary [14] the coal strength problem is widely considered. Laboratory samples of coal are found to be highly variable in strength (a range from 10 to 40 MPa is observed), while coal strength at the mass scale is rather uniform – it is in the range of 5.4–7.4 MPa. In the paper, a numerical Bonded Particle Model for coal strength behaviour was proposed, and the scale effect was introduced by adoption of a random distribution of defects. The problem of scale effect for coal behaviour was also the subject of the paper of Scholtes et al. [15]. The influence of scale effect on the strength of coal was investigated using a discrete element model for numerical simulations of a true triaxial test. The size/scale effect concerning the mechanical behaviour of concrete or other cement-based composites (quasi-brittle materials, in general) has been also considered in a large number of scientific papers. A great contribution to the theoretical description of this phenomenon has been performed by Bažant (e.g. [16–18]). A large “input” to the understanding of the concrete size/scale effect has also been done by the papers of Carpinteri (e.g. [19, 20]). In the paper of Van Mier and Van Vliet [21] the influence of concrete microstructure on size/scale effects in the case of tensile fracture is widely discussed. For several years, a group of scientists from Laboratoire Central des Ponts et Chaussées has also been working on solving this problem; e.g. [22] is a very important paper on this subject.

Considering a large number of papers concerning the subject of scale effect in random heterogeneous materials, it becomes evident that the scale effect exists. In the case of some materials, it is more noticeable than in the case of others. It is obvious that many factors have an impact on how demonstrable this effect is. At this point, a very important question appears, i.e. whether the microstructure geometry can have an impact on the scale effect. In

other words, is it possible to prove the following relationship: the more complex (heterogeneous) microstructure the more demonstrable the scale effect? Therefore, in the current work the focus is on investigation of the influence of microstructure geometry on the scale effect in the mechanical behaviour of composite specimens. It is postulated that the geometry of the microstructure can have a significant impact on the results of uniaxial compression tests when comparing results obtained from specimens with the same shape but different sizes. Hence, what we study is the size/scale effect understood as the variation of macroscopic properties with the specimen size.

For the purposes of this work two different materials are considered, i.e. gypsum plaster and cement mortar. A large number of cylindrical samples with length/diameter (L/D) ratio equal to 2 are prepared. The first group of samples are cylindrical samples of different diameters D made only of gypsum plaster, and the second one are cylindrical samples of different diameters D made of cement mortar. First, a few specimens for both materials are chosen, and the 3D computed tomography imaging is performed. It is shown that these artificial materials exhibit a significant difference in microstructure heterogeneity. The comparison of microstructure heterogeneity is carried out by making use of different microstructure measures, namely, an attenuation coefficient, porosity, and pore and particle size distributions. The quantitative analysis performed using micro-computed tomography (microCT) made it possible to evaluate, in an objective way, the differences between the two materials that are not visible at the macro level. The main goal of the research was to determine the so-called size of the heterogeneity which could have an impact on the mechanical behaviour of specimens under uniaxial compression. Then, the main postulate stated in this work is verified, namely, “whether there exists an influence of the microstructure geometry on the scale effect in the mechanical behaviour of composite materials”. Based on laboratory investigations, it is shown that the scale effect with respect to uniaxial compressive strength (UCS) and Young modulus E is more demonstrable in the case of the material which exhibits more heterogeneous microstructure. A series of uniaxial compression tests is performed for a large number of cylindrical specimens with four different sample diameters D . It should be mentioned that in the sense of the approach proposed in [12] only one aspect of scale effect, i.e. size effect, is considered in our work (the results obtained from specimens having the same shape but different sizes are compared; the $L/D=2$ ratio is the same but different diameters are considered).

The paper is organised in the following way. In Section 2 the description of experimental setup is provided. The preparation of the tested materials as well as the testing methods, i.e. X-ray microtomography and uniaxial compression, are described in detail. In Section 3, the microstructures of the two considered materials are compared based on microstructure measures derived from X-ray computed tomography imaging. Moreover, the results of uniaxial compression tests are presented. The scale effect is discussed in Section 4. Final conclusions end the paper.

2 Materials and methods

The experimental investigation focuses on underlining the role of microstructure on the scale effect in the mechanical behaviour of composite materials. In what follows, a detailed description of tested materials, methods of sample preparation and experimental devices used to perform both imaging of the microstructure and mechanical tests (uniaxial compression) is provided.

2.1 Tested materials

The composites studied in this work are made of ready-to-use mixes, commonly available on the market. They are prepared by simply adding a specific, provided by the manufacturer, amount of water to the mix containing Portland cement with sand or “pure” gypsum plaster, respectively. The mortar powder CEMAX (Kreisel, Poznań, Poland) used to make the sample is the type of mortar often applied to build concrete screeds. In the European Standard EN 13813-2003 [23], the properties of this mortar are designated as CT-C16-F4. The gypsum plaster (Dolina Nidy, Leszcze 15, Poland) is a typical building material fulfilling the requirements of the European Standard EN 13279-1-A1 [24]. However, the properties of each of the mixes have been investigated in order to present their detailed composition, for this research to become comparable with other studies concerning cement- or gypsum-based composites. The proportions of the constituents composing each of the materials under investigation are listed in Table 1. In addition, the sieve analysis was performed in order to gather information about the grain sizes of sand composing the mortar mix. The results of the test are presented in Figure 1.

PCV forms of different sizes are used for samples preparation (Figure 2). Vaseline is applied to grease the inner part of the tubes before placing the material in them to facilitate its extraction. All specimens are removed

Table 1: Constituents of composites.

Type of composite	Mortar	Gypsum
Sand (kg/m ³)	1100	0
Ready-to-use mix (kg/m ³)	1435	585
Cement/gypsum plaster (kg/m ³)	335	585
Water (kg/m ³)	200	350
Water-to-cement/gypsum plaster ratio	0.6	0.6
Volume proportion of sand	48%	0

Figure 1: Results of sieve analysis of sand.

from the moulds after 24 h and wet cured in a curing container for 14 days. A large number of cylindrical samples with length/diameter (L/D) ratio equal to 2 are prepared. For both materials, four different sample diameters are considered, namely, $D=28, 36, 45$ or 72 mm. To perform mechanical tests on sufficient representative specimens, it was decided to have the ratio between the specimen diameter D and maximum heterogeneity size $d_{\max}$ equal to, at least, $D/d_{\max}=9$. This corresponds to the worst case, i.e. the smallest sample diameter $D=28$ mm and the maximum sand particle size $d_{\max}=3$ mm. Therefore, it can be assumed that the specimens are representative volume elements (e.g. [21, 25]). In Figure 2 the exemplary samples of 28 mm diameter are shown just after removing from the moulds (before the treating procedure).

2.2 X-ray micro-computed tomography

X-ray microCT is a non-destructive test method which gives a possibility to examine and analyse the microstructure of the material. This technique has been known for several decades already; however, it has been commonly used mainly in medicine and biological sciences to visualise the bones and soft tissues. Recently, it has become

Figure 2: Twenty-eight millimetre samples of gypsum plaster and mortar after removing from cylinders (before the treating procedure) and PCV forms used for preparation of the composites.

a useful method to investigate materials, and it plays an important role in engineering, material science, electronics etc. (e.g. [26–30]).

The principle of the microCT technique is that from the 2D projections of the scanned object one can acquire the 2D cross-sections of the material (the so-called “slices”) and, as a result, a 3D reconstruction of the investigated sample. The construction of the typical scanner is presented in Figure 3. It consists of the X-ray source built of two electrodes: anode and cathode, between which a high voltage is applied in order to produce radiation. The X-ray beam is transmitted through the sample, rotating on a stage within the pre-set value of unit angle. The projections, for each angle, are recorded on the detector covered by a scintillator – the material that transforms the ionising radiation (absorbed energy) in the form of light. The scintillator is connected to a charge-coupled device camera that changes the light into a digital signal [31]. The scanning requires adjusting a large number of settings such as unit angle, use of filter, voltage, power of the X-ray source etc. The analysis of each material needs a different set of parameters due to its unique properties. As a result of a scan, a collection of object projections is acquired. In order to obtain the cross sections of the sample, the mathematical reconstruction process is necessary. Finally, the

set of slices on the height of the object investigated is gathered, which allows for further image processing including binarisation etc. Based on the binary images, a variety of microstructure measures can be calculated, e.g. porosity, shape factor, specific surface, correlation functions etc. In addition, a 3D reconstruction of the object’s volume can be obtained and analysed [32].

The total attenuation of an X-ray passing through the object can be expressed as a “line integral” – the sum of the absorptions along the path of the beam. The intensity I of the transmitted X-ray beam, according to Beer’s law, verifies the relation [31]

$$I(x) = I_0 e^{-\mu x}, \quad (1)$$

where I represents the intensity of the beam that has already passed through the sample, I_0 is the initial intensity of the beam (at $x=0$) and μ is the material’s linear attenuation coefficient equal to

$$\mu = \sum f_i \mu_i \rho_i, \quad (2)$$

where i denotes an atomic element, ρ_i is a density of the material, f_i is the atomic weight and μ_i represents the mass attenuation coefficient of the beam energy [31].

The experimental campaign started with performing the trial scanning procedure. This trial scanning revealed

Figure 3: The construction of the microCT system.

some difficulties in distinguishing the microstructure of mortar from that of the gypsum. Even though the presence of sand grains in the case of mortar samples was expected, no heterogeneities were visible. It was presumed that the problem might stem from the fact that the constituents of the investigated materials have similar densities or average atomic numbers. This prediction is apparent due to the fact that the attenuation coefficient μ of the material depends on three major factors: the thickness of the specimen, its density and the atomic number of the substance (see Equation (2)). Therefore, if there is a noticeable difference in densities or atomic numbers of components of the investigated material, then its internal structure can be successfully visualised on a radiograph. On the other hand, if the components of the scanned material have similar densities or average atomic numbers, then it is difficult to distinguish them on the microCT image.

The artificial composite materials considered in the work consist of hydrated cement/gypsum and aggregate. Differences in the densities of their components are almost negligible, e.g. hydrated cement = 1800–3300 kg/m³ and sand = 2600 kg/m³. The above is the reason why the individual components could not be extracted from the whole microstructure. In order to enhance the contrast, different settings and parameters of the scanning device were chosen but with no satisfactory effect. Finally, it was decided to stain the samples with a contrast agent. Iodine solution (3%) in ethanol was used. Iodine is a chemical element with a high atomic number, and its compounds are commonly

used in medicine as contrast agents, due to the fact that they are non-toxic. The specimens were immersed in the contrast agent and left for approximately 48 h until they dried out (the ethanol evaporates). The iodine was absorbed by the capillary pores network in the hydrated matrix.

Figure 4 shows the influence of the staining substance on the imaging of the cement mortar specimen. As mentioned before, after trial scanning of the whole sample, no aggregate was visible. Additional scanning, with a higher resolution of 13.5 μm , did not give satisfactory results – almost no heterogeneities can be distinguished (see Figure 4, top right). Application of contrast agent, without changing the resolution of scanning, demonstrates the significant differences in the image (see Figure 4, middle right). The outline of the sand grains is visible, as the cement matrix starts to absorb more radiation due to the contrast agent present. Nevertheless, the quality of the images is not sufficient for the quantitative analysis of the grain size distribution. After final scanning, with a resolution of 4.8 μm and an angle step of 0.05°, the detailed microstructure of mortar specimen is noticeable (see Figure 4, bottom right).

It should be mentioned that all specimens were scanned with the use of Skyscan 1172 (Bruker MicroCT, Kontich, Belgium) X-ray microtomography with different resolutions and angle steps. The X-ray tube voltage was set to 100 kV, and the tube's power was constant and equal to 10 W. All the scans were performed using built-in Al+Cu filter (Al 1 mm and Cu 0.05 mm). In addition,

Figure 4: The effect of the iodine staining on the microCT imaging of mortar.

for each scan, the flat field correction was applied. The reconstruction was performed using NRecon (Bruker MicroCT, Kontich, Belgium) based on Feldkamp algorithm [33]. For the 3D visualisation of the images, CTBox (Bruker MicroCT, Kontich, Belgium) and CTVol (Bruker MicroCT, Kontich, Belgium) software were utilised.

2.3 Mechanical tests

Uniaxial compression tests for both gypsum and mortar specimens were performed in accordance with the guidelines of ASTM [34]. The bottom and top surfaces of each cylindrical specimen were treated in order to ensure their parallelism and planarity and that they are perpendicular to the peripheral surface. The tests were performed using a compression tester PROETI (Proeti S.A., Algete-Madrid, Spain) with a 3000 kN capacity. The rate of displacement was set to 0.003 mm/s. During each test, the compressive load on the specimen P as well as the change in measured axial length of specimen ΔL was recorded. The latter was obtained by making use of the High-Accuracy Digital Contact Sensor GT2-H12K (Keyence, Osaka, Japan). The measuring range of the sensor is 12 mm, while its resolution is on the level of 1 μm . A change in the measured axial length ΔL was then used to compute the axial strain ε , i.e. $\varepsilon = \Delta L / L$. The compressive stress in the test specimen σ was calculated as the quotient of the compressive load and the initial cross-sectional area of the specimen A ; the UCS is therefore calculated using the maximum (peak) compressive load $P_{\max}$ on the specimen, i.e. $UCS = P_{\max} / A$. In the case of the Young modulus, three different methods of its evaluation are proposed in [34], namely, (1) tangent modulus E_t , (2) average modulus E_{av} and (3) secant modulus E_s . In the current paper, the consideration is limited to one type of the Young modulus, i.e. the average one. The modulus was evaluated for the average slopes of the more-or-less

straight line portion of the axial stress (σ)-axial strain (ε) curve (see Figure 5).

As mentioned earlier, the ratio between the length and diameter of the specimen was set to 2:1. This choice is supported by the results and conclusions obtained by different researchers (e.g. Pan et al. [12]). Moreover, the problem of the appropriate L/D ratio was also studied in the authors' previous work [35] where a series of numerical simulations was performed. The conclusion derived from those considerations is as follows: the minimum L/D ratio, for the results to be stable, is 2. For more details we refer the reader to [35].

3 Results and analysis

3.1 Imaging of specimen microstructure

After scanning of the samples with the final settings (described in the previous section), the reconstruction procedure was applied. As a result, sets of 2D images were obtained. In Figure 6 the comparison of the mortar (Figure 6A) and gypsum (Figure 6B) microstructures is presented. The colours of the images are inverted – higher grey levels (white colour) indicate particles of lower density, whereas lower grey levels are assigned to the denser constituents. Observing Figure 6, one can notice demonstrable differences in the microstructure geometry of the two artificial materials under consideration. In the mortar microstructure large distinguishable sand grains are present, which are obviously not present in that of the gypsum. In addition, it can be observed that the pore sizes differ significantly as well. For the proper comparison of the two microstructures, it is evident, however, that the visual observation has to be supplemented by the quantitative analyses. For that purpose, different microstructure measures – attenuation, porosity, pore and particle size distribution – were chosen and calculated separately for the two investigated materials.

Before calculation, the reconstructed 2D attenuation maps (images) are processed. The image processing methodology is based on a trial and error process. Due to the high noise, denoising procedures are applied, i.e. Gaussian and Kuwahara filters. For the purpose of size and shape calculation for each of the material components, namely, porosity and particles properties, the appropriate segmentation procedures have to be implemented. Here, the Otsu's segmentation method [36] is applied with the use of Bruker CTAn software [37]. Due to the partial volume effect, additional morphological procedures on the binarised images

Figure 5: Method for calculating Young modulus from axial stress-axial strain curve; average modulus of linear portion of stress-strain curve.

Figure 6: Microstructure of the investigated materials and 3D reconstruction; results for (A) mortar and (B) gypsum (inverted colours).

have to be employed to support the segmentation procedure, including erosion, opening, dilation and closing.

Firstly, the attenuation profiles of the materials were compared. The attenuation of the material is expressed in grayscale units. Due to the fact that the attenuation coefficient μ is proportional to the density of the material (Equation (2)), very dense elements are presented with white colour in the image, whereas objects of low density are distinguishable as dark grey or even black (in the case of air). For the purpose of this article, the colours were inverted (Figure 6). In Figure 7 the attenuation profiles are plotted versus the distance given in millimetres. The mortar diagram (Figure 7, top) exhibits the presence of the larger heterogeneities (sand grains) with lower frequency of peaks compared to the attenuation profile obtained for gypsum (Figure 7, bottom). Furthermore, the amplitude of the mortar's profile is higher than in the case of that of the gypsum. It proves, therefore, that the microstructure of mortar is more heterogeneous – the differences in attenuation for each pixel are higher than in the gypsum images.

Another measure used for the comparison procedure was porosity. In order to obtain the specific values for each material, firstly, the sets of images were binarised to allow the segmentation of pores from the microstructure. Finally, based on the binarised images, the porosity understood as the number of black pixels divided by the total number of pixels of the chosen region of interest for each 2D slice (cross-section) was calculated. In Figure 8 the changes of porosity versus the height of the sample are graphically presented. It can be noticed that, in general, the porosity of the mortar is higher when compared to that of the gypsum.

Furthermore, the variation of porosity, when moving from point to point, is also greater for the mortar. In the case of gypsum, one can observe a rather constant value of porosity with the distance (height of the sample). In addition, the 3D visualisation of the pores was also performed to show their location within the cylindrical sample (Figure 9). The pore distribution suggests that there are differences in the pore size between the mortar and gypsum specimens. Hence, in order to verify the above statement, calculation of a pore size distribution was done. The results of this analysis are graphically presented in Figures 10 and 11.

In Figure 10 the frequency of the pores, which is the total volume of pores of specified diameter (horizontal axis) divided by the volume of the sample, is plotted against the pore diameter (mm). The pore size diagram exhibits large dissimilarities between the two types of materials under study. It is apparent that the functions of frequency are entirely different. For the gypsum samples, the pore sizes are small, and they almost do not vary within the sample. On the other hand, for mortar there is no dominant pore size – they are distributed evenly in the material, and there is a great variety of sizes. In Figure 11 the pore size distribution is presented, however, as a 3D model of pores for each type of material investigated. Particular pore size intervals are shown with miscellaneous colours to illustrate their location within the composite. It is noteworthy that the pores in mortar (Figure 11A) are big and relatively uniformly distributed, whereas in gypsum the pores are smaller. In addition, in the gypsum plaster, some dense particles were observed. The results of the analysis of those constituents are presented in Figure 12.

Figure 7: Attenuation profiles for the mortar (top) and gypsum (bottom) specimens.

Figure 8: Porosity versus height of the sample.

It is interesting that the maximum size of the particles, 0.12 mm, is greater than the size of the pores; thus, these particles can be treated as gypsum's heterogeneities. Comparing this value to the maximum size of heterogeneity in sand, which is, based on the sieve analysis, approximately 3 mm, it is clearly visible that the materials are significantly different. The right panel of Figure 12 represents the particle shape factor (sphericity); as this value

Figure 9: Three-dimensional visualisation of the pores in (A) mortar and (B) gypsum.

converges towards 1, the shape of the particle is close to a sphere.

It was shown in the previous section that the materials (gypsum and mortar) exhibit significant differences in the microstructure geometry. Based on microCT, and as a consequence on microstructural measures (attenuation, porosity and pore size distribution), it was demonstrated that mortar has a more heterogeneous microstructure when compared to the one of gypsum. Now, it is verified

Figure 10: Pore size distribution.

whether these materials behave also in a different manner at uniaxial compression test.

3.2 Uniaxial compressive strength and Young modulus

As mentioned, *UCS* as well as the Young modulus E_{av} were determined for different diameters D . It has to be, however, strongly emphasised that for each value of D a

series of tests was performed, and the final result, for a given D , is calculated as the mean value averaged over a specified number of tests. Furthermore, it is obvious that as the specimen size (diameter D) increases, the variance of both the uniaxial compressive strength and the Young modulus should decrease. Therefore, according to the central limit theorem (e.g. [38]) the number of tests, required for a given value of D , should decrease with the increase of specimen size. In other words, comparing the samples with $D=28$ mm and $D=72$ mm, it is evident that the required number of tests is significantly greater in the case of the specimen with smaller diameter, i.e. $D=28$ mm.

After each uniaxial compression test i (for a given diameter D) the mean values of *UCS* and the Young modulus E_{av} as well as the variances of these quantities were evaluated. The mean values were calculated in the following way:

$$E_{av,D} = \frac{1}{n} \sum_{i=1}^n E_{av,D}^{(i)} \quad (3)$$

$$UCS_D = \frac{1}{n} \sum_{i=1}^n UCS_D^{(i)}. \quad (4)$$

Figure 11: Three-dimensional surface models of the pore size distribution for (A) mortar and (B) gypsum.

Figure 12: Gypsum particle analysis.

Having the mean values (Equations (3) and (4)) then, the variances were estimated using the following expressions:

$$\text{Var}(E_{av,D}) = \frac{1}{n-1} \sum_{i=1}^n (E_{av,D}^{(i)} - E_{av,D})^2, \quad (5)$$

$$\text{Var}(UCS_D) = \frac{1}{n-1} \sum_{i=1}^n (UCS_D^{(i)} - UCS_D)^2. \quad (6)$$

In the equations above, $E_{av,D}^{(i)}$ and $UCS_D^{(i)}$ are the Young modulus and the uniaxial compressive strength obtained from the i th test performed on the specimen of diameter D , respectively, and n stands for the number of tests. As mentioned above, the number of tests n that could be recognised as sufficient is affected by the size of the specimen – diameter D . The number of tests n that was assumed to be appropriate was obtained by observing the influence of successive results on the change in both mean values (Equations (3) and (4)) and the variances (Equations (5) and (6)). In other words, if successive results did not affect a significant change of mean values (Equations (3) and (4)) and the variances (Equations (5) and (6)), the number of performed tests was recognised as sufficient. In what follows, some chosen plots are presented, namely, in Figures 13 and 14, the results obtained for mortar and gypsum specimens of diameter $D = 28$ mm.

Analysing the results, two main features can be observed. For both mortar and gypsum samples the mean

value of UCS exhibits faster convergence than the mean value of the Young modulus. Therefore, it is the Young modulus that determines the number of tests n . Furthermore, the convergence of results for gypsum specimens is faster when compared to the results for mortar samples. This is due to the smaller variation of successive results around the mean value for gypsum specimens. This can be observed in Figures 15 and 16 where the variation of Young modulus and UCS for successive tests is presented for mortar and gypsum specimens, respectively. One can simply observe the greater scatter in the results for mortar samples (for both Young modulus and UCS) compared to the ones obtained for gypsum specimens; in both figures the continuous line is the mean value (Equations (3) and (4)), while the dashed line describes the mean value $\pm \bar{\sigma}$, namely, the standard deviation (square root of variance given by Equations (5) and (6)). Due to fact that the same conclusions were drawn when the results for other diameters were analysed and compared, it can be stated that the appropriate number of tests n for gypsum specimens is smaller than that for the mortar samples.

4 Discussion of results

The results of all uniaxial compression tests, for mortar and gypsum specimens of different diameters D , are

Figure 13: The results obtained for mortar specimens of diameter $D = 28$ mm.

(A) the mean value of Young modulus, (B) the mean value of UCS , (C) variance of Young modulus and (D) variance of UCS , as a function of the number of performed tests n .

Figure 14: The results obtained for gypsum specimens of diameter $D=28$ mm.

(A) the mean value of Young modulus, (B) the mean value of UCS, (C) variance of Young modulus and (D) variance of UCS, as a function of the number of performed tests n .

Figure 15: The results of successive uniaxial compression tests for mortar samples ($D=28$ mm).

(A) variation of Young modulus and (B) variation of UCS.

Figure 16: The results of successive uniaxial compression tests for gypsum samples ($D=28$ mm).

(A) variation of Young modulus and (B) variation of UCS.

Table 2: The results of laboratory investigations for mortar specimens of different diameter D .

Diameter of specimen D (mm)	28	36	45	72
$E_{av,D}$ (GPa)	3.692	2.606	1.940	1.696
$\text{Var}(E_{av,D})$ (GPa ²)	1.102	0.839	0.394	0.032
UCS_D (MPa)	17.285	10.438	8.895	8.140
$\text{Var}(UCS_D)$ (MPa ²)	14.744	7.086	7.160	0.352
n	48	32	12	6

Table 3: The results of laboratory investigations for gypsum specimens of different diameter D .

Diameter of specimen D (mm)	28	36	45	72
$E_{av,D}$ (GPa)	2.357	2.168	2.066	1.965
$\text{Var}(E_{av,D})$ (GPa ²)	0.132	0.104	0.047	0.017
UCS_D (MPa)	12.934	11.545	11.041	10.470
$\text{Var}(UCS_D)$ (MPa ²)	3.212	1.847	1.040	0.494
n	28	21	10	6

summarised in Tables 2 and 3, respectively. The tables provide the mean values and the variances. Furthermore, for each diameter size the information on the number of tests n is also presented. In addition, the results are graphically presented in Figures 17 and 18. In particular, Figure 17 shows the values of Young modulus E_{av} as a function of the sample diameter D , while the results of UCS (versus D) are displayed in Figure 18. It was found that both the Young modulus $E_{av,D}$ and UCS_D can be related to the sample diameter D by the following exponential function:

$$f(D) = a \cdot \exp(b \cdot D) + c, \quad (7)$$

where a , b and c are the constants which can be determined based on a fitting analysis of test data. The fitting functions are also displayed in Figures 17 and 18

Figure 17: Young modulus E_{av} as a function of the specimen diameter D . **Figure 18:** UCS as a function of the specimen diameter D .

(continuous lines). In addition, the values of constants, found for a given data set, are provided in each figure.

Observing Figures 17 and 18, it can be seen that the scale effect is present for both considered materials and exists in the case of two considered quantities; i.e. Young modulus E_{av} (Figure 17) and UCS (Figure 18) are affected by the size of the sample diameter D . For both the Young modulus E_{av} and UCS , one can observe that the scale effect is more demonstrable in the case of the mortar specimens. In other words, the difference between the values obtained for specimens with $D=28$ mm and the largest specimens ($D=72$ mm) is much greater in the case of mortar than in the case of gypsum specimens. Note that in the case of the mortar, the results of the Young modulus as well as UCS for the largest samples ($D=72$ mm) are more than 50% smaller than the ones obtained for the specimens with diameter $D=28$ mm. In the case of gypsum specimens, this difference is only about 15%. Therefore, the specimens made of the material which has a more heterogeneous microstructure (the mortar) exhibit more demonstrable scale effect in the sense of UCS and the Young modulus.

Hoek and Brown [39] observed that the uniaxial compressive strength of the intact rock specimen with diameter D can be described by the following relation:

$$\frac{UCS_D}{UCS_{50}} = \left(\frac{50}{D} \right)^A, \quad (8)$$

where UCS_{50} is the uniaxial compressive strength of the specimen with diameter $D=50$ mm. It was found that the best agreement between laboratory data (a wide range of intact rocks was tested) and the proposed model is obtained with $A=0.18$ [39]. In Figure 19A this function (Equation (8)) is fitted to our data. Fitting parameters and R^2 coefficient of determination, for both gypsum and mortar, are also provided. Since the size of the specimen $D=50$ mm was not investigated, the “reference” strength

Figure 19: Fitting of the UCS data of mortar and gypsum by the functions (8) and (9), respectively.

is the one obtained with $D=45$ mm; i.e. in Equation (8), UCS_{50} and 50 are replaced by UCS_{45} and 45, respectively. What is observed is that the fitting parameter for gypsum is $A=0.224$, which is close to the result obtained in [39], $A=0.18$, and therefore the scale effect in gypsum is similar to the one observed for intact rocks. In the case of mortar, the fitting parameter is much greater, namely, $A=1.221$. The model proposed by Hoek and Brown [39] does not take into account the maximum heterogeneity size. Therefore, another expression – originally established for concrete – was used to fit laboratory data: according to [40] the compressive strength of cylindrical specimens with L/D of 2.0 is governed by the following relation:

$$UCS_D = \{af'_c / [1 + (Dd_a^m)/b]^{1/2}\} + cf'_c, \quad (9)$$

where a , b , c and m are constants which have to be found by fitting to laboratory data, d_a is the maximum aggregate size and f'_c is the compressive strength of a standard cylinder. Figure 19B shows the fit of function (9) to data obtained for gypsum and mortar specimens. The maximum aggregate size d_a – used in Equation (9) – for gypsum and mortar was 0.12 and 3 mm, respectively. Here, the maximum aggregate size d_a represents the maximum size of the heterogeneity d_{max} . The value of f'_c for gypsum and mortar was 4 and 14 MPa, respectively. In order to find the relation between the results of UCS and d_{max} , the main focus was on evaluating the value of constant m which is directly related to the size of heterogeneity. Originally, the value of this constant is limited to the range from 0 to 1 [40]. These constraints were used in the fitting procedure. Fitting Equation (9) to laboratory data results in the minimum possible value, i.e. $m=0$, for gypsum; in the case of mortar, the value of m changes drastically – best fit is found for the maximum possible value, $m=1$. Therefore, it is visible that, based on the acquired results, there is no heterogeneity size effect on the UCS of gypsum; on the contrary, for mortar, the size of aggregate plays a significant role.

The sieve analysis of sand grains demonstrated that the maximum heterogeneity size in mortar is

approximately 3 mm. On the other hand, the microCT investigation of gypsum (often assumed to be completely homogeneous) revealed the existence of heterogeneities, i.e. dense particles of a maximum size of 0.12 mm. Each of the materials is in a different regime with respect to the ratio between D and the size of heterogeneity d_{max} ; i.e. for mortar D/d_{max} is in the range from 9 to 24, whereas for gypsum it is from 230 to 600. In the case of gypsum plaster, the specimen size is, beyond any doubt, a representative volume element. The small scatter in the results may be caused by the so-called wall effect [21], which refers to the inability of inclusions to penetrate through the sample borders (the specimens are cast, not cored). This effect is stronger when the size of the specimen is decreasing, which is visible in Figure 19. For mortar, the variation of the results with decreasing D may be triggered by both wall effect (for small specimens) and the small value of D/d_{max} , being significantly lower than that of the gypsum. This may be the cause of some bias in the scale effect evaluation. At this stage, the authors are aware that further research is needed in order to be able to analyse the results in a comparable regime of D/d_{max} ratio which could prevent the aforementioned biases.

5 Conclusions

A considerable amount of literature has been published on the subject of the scale effect in the mechanical behaviour of heterogeneous materials. On the basis of previous results, it can be clearly stated that the scale effect exists, and there is a large number of factors influencing it. In this work the focus was on the investigation of the influence of microstructure geometry on the scale effect in samples under uniaxial compression. For that purpose, two types of materials were considered, i.e. gypsum and mortar. The studies were divided into two groups. First, the microCT for 3D visualisation of microstructures was conducted. Next, the laboratory tests of uniaxial compression were

performed. The following conclusions can be drawn from the 3D microCT imaging:

- (a) Significant differences in the microstructure geometry of the two considered materials can be noticed.
- (b) The attenuation profiles of the materials were compared. They differ significantly as well. The mortar diagram exhibits lower frequency of peaks compared to the attenuation profile obtained for gypsum. Moreover, the amplitude of the mortar's profile is higher than that of the gypsum. In this sense, the microstructure of mortar is more inhomogeneous.
- (c) The porosity (and the variation of porosity when moving from point to point) of the mortar is higher when compared to that of the gypsum.
- (d) In the case of gypsum the pore sizes are rather small – there is one dominant size of pores. On the other hand, for mortar there is no dominant pore size – they are distributed evenly in the material, and there is a great variety of sizes.
- (e) The microCT investigation of gypsum, which is often assumed to be completely homogeneous, revealed the existence of heterogeneities, i.e. dense particles of a maximum size of 0.12 mm.
- (f) Considering the above, it can be clearly stated that mortar has a more heterogeneous microstructure when compared to the one of gypsum.

The following conclusions can be drawn from the uniaxial compression tests:

- (a) The scale effect is present for both considered materials and exists in the case of the two considered quantities, namely, *UCS* and the Young modulus – the obtained results are affected by the sample diameter *D*.
- (b) For both E_{av} and *UCS*, the scale effect is more demonstrable in the case of mortar specimens, the material which exhibits a more heterogeneous microstructure.
- (c) The difference between the values obtained for the smallest specimen ($D=28$ mm) and the largest one ($D=72$ mm) is much greater in the case of mortar than in the case of gypsum. It is approximately 50% in the case of mortar and only 15% in the case of gypsum.
- (d) The scale effect in gypsum is similar to the one observed for intact rocks with respect to the model formulated in [39].
- (e) Considering the model for concrete presented in [40], there is no heterogeneity size effect on *UCS* of gypsum, on the contrary for mortar, where the size of aggregate plays a significant role.
- (f) The small scatter in the results for gypsum plaster may be caused by the so-called wall effect [21] (the specimens are cast, not cored).

- (g) The variation of the results with decreasing *D* for mortar may be triggered by both wall effect (for small specimens) and the small value of D/d_{max} .

References

- [1] Alzebedeh K, Al-Ostaz A, Jasiuk I, Ostoja-Starzewski M. *Int. J. Solids Structures* 1998, 35, 2537–2566.
- [2] Sutherland LS, Shenoi RA, Lewis SM. *Compos. Sci. Technol.* 1999, 59, 209–220.
- [3] Sutherland LS, Shenoi RA, Lewis SM. *Compos. Sci. Technol.* 1999, 59, 221–233.
- [4] Sutherland LS, Shenoi RA, Lewis SM. *Compos. Sci. Technol.* 1999, 59, 235–251.
- [5] Bieniawski ZT. *Int. J. Rock Mech. Min. Sci.* 1968, 5, 325–335.
- [6] Hodgson K, Cook NGW. In *Proceedings of the Second International Congress Rock Mechanics*, Belgrade, Yugoslavia, 1970.
- [7] Hudson JA, Brown ET, Fairhurst C. In *Proceedings of the 13th Symposium on Rock Mechanics*, University of Illinois, Urbana, IL, 1971.
- [8] Pratt HR, Black AD, Brown WS, Brace WF. *Int. J. Rock Mech. Min. Sci.* 1972, 9, 513–529.
- [9] Bazant Z, Chen EP. *Appl. Mech. Rev.* 1997, 50, 593–627.
- [10] Jirasek M, Rolshoven S, Grassl P. *Int. J. Numer. Anal. Meth. Geomech.* 2004, 28, 653–670.
- [11] Pisarenko D, Gland N. *Phys. Chem. Earth (A)*, 2001, 26, 83–88.
- [12] Pan PZ, Feng XT, Hudson JA. *Int. J. Rock Mech. Min. Sci.* 2009, 46, 674–685.
- [13] Zhang Q, Zhu H, Zhang L, Ding X. *Int. J. Rock Mech. Min. Sci.* 2011, 48, 1320–1328.
- [14] Poulsen BA, Adhikary DP. *Int. J. Rock Mech. Min. Sci.* 2013, 63, 62–71.
- [15] Scholtes L, Donze FV, Khanal M. *J. Mech. Phys. Solids* 2011, 59, 1131–1146.
- [16] Bažant ZP. *J. Eng. Mech.* 1984, 110, 518–535.
- [17] Bažant ZP, Planas J. *Fracture and Size Effect in Concrete and Other Quasibrittle Materials*. Vol. 16. CRC Press: Boca Raton, FL, 1997.
- [18] Bažant ZP, Drahomír N. *J. Eng. Mech.* 2000, 126, 166–174.
- [19] Carpinteri A, Ferro G, Monetto I. *Mag. Concrete Res.* 1999, 51, 217–225.
- [20] Carpinteri A, Ed. *Size-Scale Effects in the Failure Mechanisms of Materials and Structures*. CRC Press, London, UK, 2002.
- [21] Van Mier JGM, Van Vliet MRA. *Eng. Fract. Mech.* 2003, 70, 2281–2306.
- [22] Rossi P, Wu X, Le Maou F, Belloc A. *Mater. Struct.* 1994 27, 437–444.
- [23] European Standard EN 13813-2003.
- [24] European Standard EN 13279-1-A1.
- [25] Gitman I, Askes H, Sluys L. *Eng. Fract. Mech.* 2007, 74, 2518–2534.
- [26] Shigang A, Xiaolei Z, Yiqi M, Yongmao P, Daining F. *Appl. Compos. Mater.* 2014, 21, 603–614.
- [27] Cnudde V, Wirzen A, Masschaele B, Jacobs PJS. *Eng. Geol.* 2009 103, 76–83.
- [28] Miettinen A, Luengo Hendriks CL, Chinga-Carrasco G, Gamstedt EK, Kataja M. *Compos. Sci. Technol.* 2012, 72, 1901–1908.
- [29] Gouze P, Luquot L. *J. Contam. Hydrol.* 2011, 120, 45–55.

- [30] Salvo L, Suéry M, Marmottant A, Limodin N, Bernard D. C. R. Phys. 2010, 11, 641–649.
- [31] Epstein CL. *Introduction to the Mathematics of Medical Imaging*. Siam, Philadelphia, USA, 2008.
- [32] Landis EN, Keane DT. X-ray microtomography. *Mater. Charact.* 2010, 61, 1305–1316.
- [33] Feldkamp LA, Davis LC, Kress JW. *J. Op. Soc. Am.* 1984, 1, 612–619.
- [34] A. S. T. M. Designation: “D 3148-02” Standard test method for elastic moduli of intact rock core specimens in uniaxial compression. American Society for Testing and Materials, 2005.
- [35] Łydzba D, Róžański A, Rajczakowska M, Sobótka M, Serwicki A, Stefaniuk D. In *Proceedings of the TC105 ISSMGE International Symposium on Geomechanics from Micro to Macro*. Soga K, Ed., IS-Cambridge, 2014.
- [36] Otsu N. *Automatica*, 1975, 11, 23–27.
- [37] Manual for Bruker-microCT CT-Analyser v. 1.13.
- [38] Feller W. *An Introduction to Probability Theory and Its Applications: Volume One*. John Wiley & Sons, New York, USA, 1950.
- [39] Hoek E, Brown ET. *Underground Excavations in Rock*. Inst. Min. Metall: London, 1980.
- [40] Kim JK, Yi ST. *Sadhana* 2002, 27, 467–484.