

# Mechanical compaction of crustal analogs made of sintered glass beads: the influence of porosity and grain size

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1	Mechanical compaction of crustal analogs made of sintered glass beads: the influence
2	of porosity and grain size
3	
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11	
12	Key Points:
13	The mechanical compaction behavior of monodisperse sintered glass bead samples is
14	similar to that of well-sorted monomineralic sandstones
15	During triaxial compression at high confining pressure, discrete compaction bands
16	developed in a synthetic sample with a porosity of 0.35
17	All else being equal, increasing grain diameter from 0.2 to 1.15 mm decreases the stress
18	to reach C* by more than a factor of two

#### 19 Abstract

The fundamentals of our understanding of the mechanical compaction of porous rocks stem from 20 experimental studies. Yet, many of these studies use natural materials for which microstructural 21 parameters are intrinsically coupled, hampering the diagnosis of relationships between 22 microstructure and bulk sample behavior. To probe the influence of porosity and grain size on 23 the mechanical compaction of granular rocks, we performed experiments on synthetic samples 24 prepared by sintering monodisperse populations of glass beads, which allowed us to 25 26 independently control porosity and grain diameter. We conducted hydrostatic and triaxial compression tests on synthetic samples with grain diameters and porosities in the ranges 0.2-1.15 27 mm and 0.18-0.38, respectively. During hydrostatic compaction, sample porosity decreased 28 29 suddenly and substantially at the onset of inelastic compaction due to contemporaneous and 30 extensive grain crushing, a consequence of the monodisperse grain size. During triaxial tests at 31 high confining pressure, our synthetic samples failed by shear-enhanced compaction and showed evidence for the development of compaction bands. Critical stresses at the onset of inelastic 32 compaction map out linear-shaped yield caps for the porosity-grain diameter combinations for 33 which the critical stress for inelastic hydrostatic compaction is known. Our yield caps reinforce 34 35 the first-order importance of porosity on the compactive yield strength and show, all else being equal, that grain size also exerts a first-order control and should therefore be routinely measured. 36 37 Our study further reveals the suitability of sintered glass beads as analogs for crustal rocks, which facilitate the study of the deconvolved influence of microstructural parameters on their 38 mechanical behavior. 39

40

#### 41 Plain Language Summary

Porous rocks that form the shallow part of the Earth's crust are submitted to pressure conditions under which they deform by compaction, i.e., their porosity is reduced. Our understanding of the compaction stems primarily from laboratory experiments conducted on samples of natural rocks. Yet, because the internal structure of natural materials is complex, studying of the influence of structural parameters in isolation, such as porosity and grain size, on the way rock compacts is limited. To tackle this issue, we conducted compression tests on synthetic samples made of fused glass beads for which we could control porosity and grain size. When the pressure on the 49 synthetic rocks is increased uniformly in all directions, a threshold pressure, characteristic of the rock strength, is reached beyond which they compact suddenly and substantially. When a 50 51 differential stress is imposed on the synthetic rocks, discrete bands of lower porosity are observed. We show that porosity and grain size exert a first-order control on the ability of the 52 synthetic rocks to resist compaction. All else being equal, if the grain size is divided by five, the 53 pressure beyond which the sample will deform by irreversible compaction is multiplied by more 54 than two. Further, our synthetic samples are good analogs for natural rocks such as sandstones 55 and tuffs and our results therefore have broad applications, from reservoir compaction and 56 subsidence to the destabilization of volcanoes. 57

58

#### 59 **1 Introduction**

The mechanical compaction of porous materials is an important process in the Earth's crust. It is 60 61 one of the main deformation mechanisms of lithification, diagenesis, fault growth, and/or sealing, and plays a key role in many processes in sedimentary settings such as reservoirs, aquifers, and 62 basins (Bjørlykke, 2006; Guéguen & Boutéca, 2004; Taylor et al., 2008), and in volcanic settings 63 (Farguharson et al., 2017; Grunder & Russell, 2005; Quane et al., 2009). Understanding the 64 phenomenology and micromechanics of compaction rests upon the ability to characterize the 65 evolution of microstructure through compactant deformation. To predict the occurrence and 66 extent of mechanical compaction, knowledge of the relationship between rock microstructural 67 attributes and bulk mechanical properties is crucial. Indeed, the effective macroscopic properties 68 of heterogeneous materials such as crustal rocks intricately depend on the phases present, their 69 volume fraction, their spatial distribution, and their properties (e.g., Torquato, 2002). Therefore, 70 71 relating microstructural attributes of porous rock to bulk properties has been the focus of numerous studies in the past decades, the majority of which relied on direct experimental 72 measurements or numerical simulations (Blair et al., 1993; Doyen, 1988; Eberhart-Phillips et al., 73 74 1989; Ghazvinian et al., 2014; Schöpfer et al., 2009; Scott & Nielsen, 1991). As early as 1990, it had already been suggested the two principle microstructural controls on the 75

mechanical and hydraulic properties of sedimentary rocks were (1) porosity and (2) grain size

77 (Bourbie & Zinszner, 1985; Paterson & Wong, 2005; Rutter & Glover, 2012; Zhang et al., 1990).

78 Broadly speaking, an increase in rock porosity causes a decrease in strength (e.g., Baud et al.,

79 2014; Chang et al., 2006) and an increase in permeability (Bernabé et al., 2003; Dardis & Mccloskey, 1998). In detail, porosity also exerts an influence on the type of failure that can result 80 81 from the application of crustal stresses (i.e., brittle or ductile). Low-porosity rock will remain brittle even under a wide range of pressure conditions (analogous to depth), whereas high-82 porosity rock will only behave in a brittle manner at relatively low-pressure and will transition to 83 ductile behavior at high-pressure (Wong et al., 1997; Wong & Baud, 2012). Alongside the 84 influence exerted by porosity, grain size has also been the target of an increasing number of 85 experimental studies which demonstrated its controlling influence on mechanical and hydraulic 86 properties (Atapour & Mortazavi, 2018; Schultz et al., 2010; Wasantha et al., 2015). However, 87 when considering porosity and grain size, one usually refers to average values for the bulk 88 volume of samples. As pores and grains are not always homogeneously distributed, a more 89 accurate way to describe microstructure is to use pore and grain size distributions. Grain size 90 distribution in lithified sedimentary rocks is also known to influence the mechanical behavior 91 and failure mode (Guéguen & Fortin, 2013; Weng & Li, 2012; Xu et al., 2020), notably the 92 possible development of compaction localization at high effective pressures (Baud et al., 2004). 93 94 Assuming that grain crushing initiates by Hertzian fracture at grain contacts, Zhang et al. (1990) proposed a micromechanical model predicting the stress required to achieve grain crushing using 95 porosity and grain size. While the model by Zhang et al. (1990) is in basic agreement with the 96 existing data set, Rutter and Glover (2012) suggested that data for sandstones would be better 97 98 described by a different empirical law. Moreover, the scatter on compiled experimental data remains large (Baud et al., 2014; Chang et al., 2006). Indeed, trends in plots of strength as a 99 100 function of porosity or grain size are complicated by the fact that other microstructural parameters, such as pore size and shape, their distributions, or matrix composition, change 101 102 together with porosity and grain size. For example, a decrease in porosity is often associated with an increase in the proportion of cement, that, if located along grain boundaries, can greatly 103 increase the strength of granular materials (Baud et al., 2017; David et al., 1998; de Bono et al., 104 2015; Haeri et al., 2005; Yin & Dvorkin, 1994). These complexities arise from the fact that the 105 106 influence of porosity, grain size, and these other microstructural considerations are all interconnected and that ultimately a fruitful way forward would be to find a method by which they 107 can be deconvolved and studied in isolation. 108

109 The uncertainty ensuing from the strong coupling between microstructural parameters, the inherent variability from sample to sample and the heterogeneity of natural materials limits the 110 111 extent to which experimental studies can draw definitive conclusions about the influence of specific microstructural attributes on the mechanical properties of natural materials. To tackle 112 this issue, several strategies could be considered: experiments using particularly simple materials 113 occurring with a broad range of porosity such as Fontainebleau sandstone (Saadi et al., 2017; 114 Sulem & Ouffroukh, 2006) or Leitha limestone (Baud et al., 2017); simulations using numerical 115 samples (Schöpfer et al., 2009; Weng & Li, 2012); or experiments using synthetic samples 116 (Bouzidi & Schmitt, 2012; Castagna & Backus, 1993; Chapman et al., 2018; David et al., 1998; 117 Plona et al., 1980). In this study, we chose to follow the rationale of Berge et al. (1995), Guyon 118 et al. (1987), and Wadsworth et al. (2016) who proposed sintered soda-lime silica glass beads as 119 suitable analogs for granular crustal rocks, such as sandstones and tuffs. Silicate glass 120 compositions have been extensively studied and data pertaining to their properties have been 121 gathered in handbooks for various applications (e.g., Bansal & Doremus, 2013). The elastic 122 properties of soda-lime silica glass are comparable to those of granular sedimentary and volcanic 123 rocks (Berge et al., 1995; Vasseur et al., 2016). Additionally, in nature, grains that go through the 124 different steps of diagenesis and form variably porous upper crust material are well 125 approximated, to a first order, by sintering beads. Viscous sintering of glass beads then allows 126 for the reproduction of the granular to non-granular transition and the preparation of synthetic 127 128 rocks of pre-determined final porosity and grain size (Wadsworth et al., 2016). In this paper, we present results of a suite of mechanical tests performed on synthetic samples 129 made of monodisperse distributions of glass beads. After describing the preparation technique 130 and the intact microstructure of our synthetic samples, we present the mechanical data and the 131

associated failure mode. These results are discussed, and we will focus on the following

133 questions. Mechanically speaking, how do the sintered glass beads samples compare with natural

134 sandstones? Quantitatively, what is the influence of grain size and porosity on mechanical

135 compaction? Considering the importance of compaction bands in the fields of rock mechanics,

136 hydrology, and geology, could this failure mode be reproduced in synthetic samples?

#### **2** Preparation and characterization of the synthetic samples

#### 139

## 2.1 Viscous sintering of monodisperse populations of glass beads

When heated above the glass transition temperature interval, glass beads become viscous 140 droplets. If these glass beads are packed together when heated then, as they transition from glass 141 142 beads to viscous droplets, they can interact and coalesce in a process referred to as viscous sintering (Frenkel, 1945; Mackenzie & Shuttleworth, 1949; Wadsworth et al., 2016). The 143 144 dominant consequence of viscous sintering is that the porosity is reduced with time. Because it is temperature-activated, this process has advantages for producing idealized porous materials. A 145 146 desired packing arrangement, or grain size distribution can be determined in the cold, room temperature state, and then via heating, viscous sintering processes can be used to evolve that 147 state to lower porosities. 148

149 Viscous sintering is driven by interfacial tension between the glass beads and the interstitial gas 150 between the beads (Kuczynski, 1949). During viscous sintering, an initial system of viscous droplets evolves with time through two main stages: (1) the growth of necks between droplet-151 droplet pairs that share a contact (Frenkel, 1945), and (2) the shrinkage and closure of the pores 152 between the droplets (Mackenzie & Shuttleworth, 1949). The initial system of viscous spherical 153 droplets and interstitial pores evolves into a system of isolated pores within a viscous liquid 154 continuum. That is, the end-state (gas in a liquid) is the inverse of the starting state (liquid in a 155 gas) (Wadsworth et al., 2017). In practice, in order to reach a desired porosity in a desired time 156 for a given grain size of glass bead, we control the temperature of synthesis (Wadsworth et al., 157 2016). The effect of changing the grain size is to change the pore size between the grains; 158 159 therefore the ability to tweak the grain size of the starting glass bead population allows us to 160 control the pore size distribution independently from the porosity, effectively deconvolving these structural controls. 161

Table 1. Chemical composition of glass beads (provided by manufacturer) used in this study.
 N.b. All wt% are recalculated to 100% disregarding the minor effect of loss on ignition.

Oxide	SiLiBeads (wt%)	SpheriGlass (A-Glass)
		(wt%)
SiO <sub>2</sub>	72.95	72.5

Na <sub>2</sub> O	13.08	13.7
CaO	9.06	9.8
MgO	4.25	3.3
Al <sub>2</sub> O <sub>3</sub>	0.58	0.4
FeO/Fe <sub>2</sub> O <sub>3</sub>	-	0.2
K <sub>2</sub> O	-	0.1

We prepared three sets of samples using monodisperse populations of glass spheres of diameter 165 166 between 0.15 and 0.25 mm (Spheriglass A-glass 1922 from Potters Industries Inc.), 0.4 and 0.6 mm (SiLibeads Glass beads type S 5218-7), or 1 and 1.3 mm (SiLibeads Glass beads type S 167 4504) of similar chemical composition (Table 1). The corresponding grain diameter distributions 168 are presented in Figure 1(a). The synthetic rocks were prepared as blocks from which samples 169 170 were cored. For each block, a monodisperse distribution of beads was poured into a ceramic tray of dimensions  $205 \times 125 \times 50$  mm. The tray was then manually shaken to flatten out the beak 171 pack surface and then placed inside an electric box furnace (L9/11/SKM by Nabertherm). The 172 box furnace was set to heat at a constant rate of 3 °C.min-1 to 680 °C, which is above the glass 173 transition onset temperature of 549 °C provided by the manufacturer (similar for both bead type; 174 175 Table 1). The peak sintering temperature was maintained constant for 1 to 12 hours depending on the target final porosity (longer times result in lower porosity). The tray was moved to 180° of its 176 177 initial position halfway through the dwell to reduce the heterogeneity of the sintered block that may arise when the temperature distribution in a furnace is not even. After being held constant 178 179 for a fixed time, the temperature in the furnace was lowered to 500 °C at a cooling rate of 1 °C.min-1 and finally decreased to ambient temperature at a cooling rate of 3 °C/min. This 180 cooling workflow was designed to minimize thermal microcracking. 20 mm-diameter cylindrical 181 samples were cored along the horizontal axis of the resulting sintered block to minimize gravity 182 183 induced porosity gradients along the axis of the samples. These samples were then cut and precision-ground to a nominal length of 40 mm. 184

185 2.2 Description of the sintered glass beads samples

A photograph of one of the synthetic samples is provided as Figure 1(b). Insights into the 186 microstructure of the synthetic samples were gained using polished thin sections observed under 187 a scanning electron microscope (SEM) (Figure 1(c)(d)). The SEM images were acquired on a 188 polished slice of a sample with a porosity of 0.35 and a mean grain diameter of 0.2 mm. As 189 predicted by the Frenkel model, adjacent beads are connected by a neck and pore space remains 190 191 between the bonded grains (Figure 1(d)). The lowest porosity obtained in this study is 0.22. 192 Therefore, all our samples are in the range between high porosity – close to the initial packing 193 porosity where incipient sintering has only formed necks – and intermediate porosity – where sintering has progressed and begun to close the pore network. Our study does not encompass low 194 porosity synthetic rocks which would exhibit pore structures close to isolated pores in a glass 195 medium (the equilibrium porosity at the end of sintering is 0.03; Wadsworth et al., 2016) . 196 Thus, all our samples are in the upper range of porosity encompassed by natural granular rocks. 197 While a material is only truly granular when the individual grains can move relative to one 198 199 another (which is the case for the glass beads prior to sintering), our synthetic rocks are close to that granular end member case. The microstructure of an assembly of soda-lime silica glass 200 spheres that underwent densification by sintering to a more advanced stage have been imaged in 201 2D by Vasseur et al. (2016) and continuously in 3D by Wadsworth et al. (2017), who performed 202 203 X-ray computed-tomography. These studies showed that the topological inversion of the viscous system takes place continuously through sintering, hence allowing for the construction of a 204 205 unified physical description for the evolution of porosity and permeability during viscous sintering. 206

207 Although statistically homogeneous, the granular materials prepared here by sintering glass spheres present heterogeneities on the scale of 2-3 grains. Indeed, SEM images of intact samples 208 209 reveal small heterogeneities in the pore size distribution (Figure 1(c)). As our preparation workflow allows for controlling the diameter of the grains and the degree of polydispersivity but 210 211 not for designing the exact nature and structure of the porous space, some porous patches, whose width can reach 0.4 mm, can be observed in our intact samples (Figure 1(c)). Using square 212 windows of 0.8 mm of edge-length and of 2mm of edge-length, 2D porosity measurements were 213 performed on SEM images of intact samples (using ImageJ). The square window with an edge-214 215 length of 2 mm ensures that the measured area contains at least 10 glass beads in any one

direction, to ensure a representative element volume (REV), and the square window with an 216 edge-length of 0.8 mm allows us to better understand whether there are variations in porosity on 217 218 a smaller scale. Figure 1(e) presents histograms of the distributions of 2D porosity measurements obtained for the zoomed-out SEM image in Figure 1(c). While porosity measurements performed 219 in the larger window provide a monomodal distribution closely-clustered around 0.35, porosity 220 measurements performed in the smaller window yield local values up to 0.42 in a sample with an 221 average porosity of 0.35. We refer to these volumes as porosity clusters. In addition, the absence 222 of cement is accompanied by heterogeneities in the local grain contacts geometry, as previously 223 reported by den Brok et al. (1997). Sintered porous materials are random heterogeneous but 224 isotropic porous media in terms of their microstructure, where we use 'heterogeneous' to refer to 225 the lack of structural order. However, our samples are homogeneous in the sense that the random 226 variation in the microstructure occurs on length scales much less than the sample lengths. 227

#### **3 Experimental procedures**

To study mechanical compaction using synthetic samples, we conducted a suite of mechanical 229 tests on sintered glass bead samples. We performed hydrostatic and conventional triaxial 230 compression experiments. During hydrostatic experiments the principal stresses are equal in all 231 directions, i.e., the state of stress is  $\sigma_1 = \sigma_2 = \sigma_3$ . During triaxial experiments, an axial stress is 232 superimposed onto a hydrostatic pressure. The principal stress parallel to the axis of the sample 233 is higher than the principal stresses normal to this axis, i.e., the state of stress is  $\sigma_1 > \sigma_2 = \sigma_3$ . 234 Although we focus here on compaction, we performed a few triaxial tests at relatively low 235 pressure to identify the brittle-ductile transition. A summary of all our experiments is provided in 236 Table 2. The solid density of glass beads was determined using a helium pycnometer 237 (Micromeritics AccuPyc II 1340) prior to and after sintering at 680°C. We found that the solid 238 density of the glass beads, 2.49 g/cm3, was unchanged following exposure to 680°C. As we 239 know the solid density of the glass beads, porosity was derived from the dimensions and the 240 241 mass of the samples. The mean error associated with the porosity measurement is 0.005 (Table 2). The permeability of the samples was measured prior to deformation using a benchtop gaz 242 (nitrogen) permeameter. Permeability was measured under a confining pressure of 1 MPa using 243 the steady-state method (following the method detailed in Heap et al., 2017). All experiments 244 245 were conducted at the Ecole et Observatoire des Sciences de la Terre (EOST) in Strasbourg

(France) following the procedure detailed by Baud et al. (2015). All samples underwent the same 246 preparation before the experiments. First, the samples were encased in very thin (< 1 mm-thick) 247 copper foil jackets to preserve bulk sample cohesion following deformation (so that thin sections 248 could be prepared) and to avoid disking. The samples were then dried in a vacuum oven at 40 °C 249 for at least 48 hours and then vacuum-saturated with deionized water. Before each test, the 250 sample to be deformed was positioned between two steel end-caps - the bottom one of which has 251 a concentric hole at the center for fluid access to the pore pressure system. In addition, the 252 bottom endcap was separated from the sample by a thin highly permeable filter, made from 253 coffee filter paper, to prevent broken beads from infiltrating the pore pressure piping during the 254 experiments. Viton® tubing was used to separate the sample from the confining pressure system. 255 All experiments were performed at room temperature on water-saturated samples. Computer-256 controlled stepping motors were used to independently regulate the confining pressure, pore fluid 257 pressure and axial stress. Data were acquired with a sampling period of 10s for hydrostatic 258 experiments and 1s for triaxial compression experiments. 259

260	Table 2. Experimental conditions and mechanical data of the synthetic samples tested in this
261	study. Triaxial tests were conducted at nominal strain rates of 10 <sup>-5</sup> s <sup>-1</sup> .

	D	+/-	Confining	Pore pressure	Effective pressure	Peak stress $\sigma_v$		Yield stress C*	
Sample	Porosity	(mean error = 0.005)	pressure P <sub>c</sub> (MPa)	P <sub>p</sub> (MPa)	$P_{eff} = P_c - P_p$ (MPa)	P (MPa)	Q (MPa)	P (MPa)	Q (MPa)
			mea	n grain diameter	r 1.15 mm				
1814	0.181	0.004	_	-	0	10	30	-	-
1812	0.183	0.003	40	10	30	-	-	92	185
1816	0.189	0.004	60	10	50	-	-	115	194
1412	0.271	0.005	30	10	20	38	55	-	-
1414	0.265	0.005	70	10	60	-	-	80	59
1419	0.262	0.005	100	10	90	-	-	105	45
1413	0.269	0.005	130	10	120	-	-	132	35
1411	0.271	0.005	160	10	150	-	-	159	28
114012	0.294	0.006	-	-	0	1.4	4.3	_	-

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114010	0.294	0.000	-	10	nydrostatie	-	-	156	0
114016	0 294	0.006	_	10	hydrostatic	_	_	Р	*
1313	0.303	0.006	130	10	120	-	-	128	23
1314	0.294	0.006	100	10	90	-	-	102	35
114011	0.308	0.006	70	10	60	74	43	-	-
114013	0.302	0.006	40	10	30	36	17	-	-

mean grain diameter 0.5 mm

2317	0.255	0.005	-	-	0	10	31	-	-
23111	0.249	0.005	40	10	30	74	132	-	-
2316	0.256	0.005	70	10	60	-	-	92	97
2318	0.258	0.005	110	10	100	-	-	133	100
2313	0.263	0.005	160	10	150	-	-	177	80
2314	0.256	0.005	190	10	180	-	-	200	60
22114	0.299	0.006	-	-	0	20	7	-	-
2218	0.302	0.006	30	10	20	-	-	40	59
27312	0.304	0.006	50	10	40	-	-	61	63
22111	0.292	0.006	70	10	60	-	-	83	70
27314	0.295	0.006	90	10	80	-	-	99	58
27315	0.294	0.006	110	10	100	-	-	117	52
22213	0.301	0.006	120	10	110	-	-	125	45
27313	0.299	0.006	130	10	120	-	-	133	39
2213	0.299	0.006	150	10	140	-	-	152	36
2217	0.296	0.006	170	10	160	-	-	168	23
27211	0.205	0.000		10	harden statis			P	*
2/311	0.295	0.006	-	10	nyarostatic	-	-	173	0

mean grain diameter 0.2 mm

31012	0.225	0.005	-	-	0	20	59	-	-
31713	0.220	0.004	40	10	30	-	-	87	172
31013	0.222	0.004	70	10	60	-	-	130	212
37	0.226	0.005	90	10	80	-	-	150	212
31113	0.256	0.005	70	10	60	-	-	109	147
31110	0.266	0.006	130	10	120	-	-	177	170
311	0.262	0.005	160	10	150	-	-	200	150
31114	0.255	0.005	190	10	180	-	-	225	135
3813	0.300	0.005	40	10	30	68	115	-	-
3814	0.307	0.006	70	10	60	-	-	92	95
3413	0.305	0.006	130	10	120	-	-	146	77
3411	0.299	0.006	190	10	180	-	-	200	61
3311	0.346	0,007	30	10	20	-	-	35	46
3612	0.353	0.008	50	10	40	-	-	58	55
339	0.351	0.008	70	10	60	-	-	74	42
3314	0.356	0.007	90	10	80	-	-	91	33
2212	0.057	0.000		10	1 1			Р	*
3312	0.357	0.008	-	10	nydrostatic	-	_	118	0
3710	0.385	0.009	40	10	30	-	-	37	22
379	0.387	0.009	50	10	40	-	-	46	19
3711	0.385	0.007	60	10	50	-	-	54	13
2712	0.282	0.007		10	budrostatio			P	*
5/12	0.382	0.007	-	10	nyurostatic	-	_	69	0

<sup>262</sup> 

For both hydrostatic and triaxial compression tests, the confining pressure  $P_c$  (kerosene) and the pore fluid pressure  $P_p$  (deionized water) were first slowly increased to their target values using servo-controlled pumps (see Table 2 for values). A fixed pore fluid pressure  $P_p$  of 10 MPa was used for all experiments and we assume a simple effective pressure law  $P_{eff} = P_c - P_p$ , operative

for rock failure under static conditions, as shown for sandstones by Baud et al. (2015) in the 267 brittle and ductile regimes. For the hydrostatic tests, the effective pressure P<sub>eff</sub> was increased in 268 269 small steps from its initial value of 2 MPa until the critical stress state for the onset of grain crushing P\* (Zhang et al., 1990), or the upper pressure limit of the press ( $P_c = 200 \text{ MPa}$ ), was 270 reached. We waited for microstructural equilibrium at each step before increasing the confining 271 pressure further. To do this, we assume that microstructural equilibrium was achieved when the 272 rate of the pore volume change (recorded by monitoring the displacement of the piston in the 273 pore pressure intensifier) was lower than 10<sup>-2</sup> s<sup>-1</sup>. The amount by which P<sub>eff</sub> was increased at each 274 step varied from 1 to 10 MPa depending on the time necessary to reach microstructural 275 equilibrium at the previous step. For triaxial experiments, once the targeted effective pressure P<sub>eff</sub> 276 was reached (i.e., hydrostatic pressurization, see Table 2 for values), the system was left to 277 equilibrate until the pore fluid change was lower than 10<sup>-2</sup> s<sup>-1</sup>. Then, at constant P<sub>eff</sub>, the sample 278 was loaded as the upper piston was lowered at a fixed servo-controlled rate corresponding to a 279 nominal strain rate of 10<sup>-5</sup> s<sup>-1</sup>. Considering the range of permeabilities of the synthetics (from 10<sup>-</sup> 280  $^{13}$  to  $10^{-11}$  m<sup>2</sup>, measured at a P<sub>c</sub> of 1 MPa), the strain rate applied during triaxial compression was 281 282 low enough to ensure drained conditions (i.e., the product of the strain rate and the Darcy timescale  $Da = t_D \epsilon = \mu_f L^2 / (k \Delta P)$  is much less than unity (Heap & Wadsworth, 2016)). 283 During all tests, a linear variable differential transformer (LVDT) monitored the position of the 284 285 upper piston with an accuracy of 0.2 µm, thus giving access to displacement, and a pressure probe in the axial pressure circuit provided a measurement of the applied axial force. Using the 286 initial dimensions of the sample, axial stress and strain were obtained. Porosity change was 287 provided by the conversion of the pore volume change given by the displacement of the piston in 288 the pore pressure intensifier (the system was calibrated to take into account the compressibility 289 of the pore fluid (water)). Finally, acoustic emission (AE) activity was recorded using a USB AE 290 Node from Physical Acoustics and a piezoelectric transducer (a micro80 sensor from Physical 291 292 Acoustic with a bandwidth of 200-900 kHz) attached to the lower piston. AE activity was monitored using the software AEwin and we set the detection threshold for an AE hit at 28 dB. 293 The AE energy is determined by AEwin as the area under the received waveform. Experiments 294 295 were stopped after the samples were unloaded at the same servo-controlled rate as loading and the pressures removed slowly so as not to damage the samples. For experiments conducted in the 296

brittle regime, samples were unloaded following macroscopic failure. In the ductile regime,

samples were deformed up to a 4% axial strain if mechanical data indicated strain localization

299 (i.e., if there were small stress drops in the mechanical data) and up to 6% if not. Based on

300 previous studies on natural rocks (Baud et al., 2004), these strains are considered suitable for

301 subsequent observation of microstructural deformation features. To gain insights into the

302 microstructure, polished thin sections were prepared using selected deformed samples and

303 micrographs of the thin sections were obtained using a SEM.

#### 304 4 Mechanical data

In this study, compressive stress and compactive strain, i.e., shortening for axial strain and decreasing volume for volumetric strain, will be conventionally taken as positive. The maximum and minimum applied principal compressive stresses are referred to as  $\sigma_1$  and  $\sigma_3$ , respectively, the differential stress as  $Q = \sigma_1 - \sigma_3$  and the effective mean stress as  $P = (\sigma_1 + 2\sigma_3)/3 - P_p$ .

309 4.1 Results of the hydrostatic and triaxial tests

Representative data for the mechanical response of sintered glass bead samples to hydrostatic 310 loading are presented in Figure 2. The hydrostatic experiment was conducted on a synthetic 311 312 sample with a porosity and mean grain diameter of 0.38 and 0.2 mm (3714 in Table 2) and the mechanical data are plotted alongside the corresponding AE activity (dashed purple). The gray 313 314 dashed curve in Figure 2 presents the mechanical data from an experiment performed on a sample with a very similar porosity and the same grain diameter to show the reproducibility 315 (3712 in Table 2). The porosity reduction in pourcentage corresponds to the absolute loss of 316 porosity. In Figure 2, the hydrostats present the following characteristic phases. (1) The initial 317 318 evolution of porosity with increasing effective pressure is non-linear. The duration of this first 319 stage varies from sample to sample, as demonstrated by the difference between the black and the gray curves, and is positively correlated to porosity. (2) The second phase consists of a linear 320 decrease of porosity as a function of increasing effective mean stress, which is characteristic of 321 poroelastic behavior. Almost no AEs are recorded during the initial non-linear and linear 322 portions of the hydrostatic experiment (Figure 2). However, a sudden increase in cumulative AE, 323 associated with a sharp breaking point in the mechanical data, indicated as P\*, marks the 324 transition to (3) a third phase characterized by a large decrease in porosity (of about 0.1) at 325 constant effective pressure. For siliciclastic rock, this inflection on the hydrostat followed by a 326

large porosity reduction is characteristic of inelastic compaction by delocalized grain crushing 327 (Zhang et al., 1990), P\* therefore represents the critical stress for the onset of grain crushing. 328 329 After equilibrium of the system has been reached for the critical state of stress P\*, further increase of the effective stress is accompanied by hardening. Samples submitted to hydrostatic 330 loading (up to the maximum capability of the pressure cell; i.e.,  $P_c = 200$  MPa) presented 331 effective pressure-porosity reduction curves similar to the hydrostats presented in Figure 2. 332 Triaxial compression experiments were conducted at effective pressures Peff ranging from 20 to 333 334 180 MPa and, depending on the effective pressure P<sub>eff</sub>, led to either brittle or ductile failure. A representative curve for the mechanical data and AE activity corresponding to failure by 335 dilatancy and shear fracture formation (i.e., brittle behavior) is presented in Figure 3. The stress-336 strain curve can be divided into three parts. (1) First, the axial strain increases linearly with the 337 differential stress and very few AEs are recorded. (2) Second, a sudden increase in the AEs 338 339 accompanies a small decrease in the slope of the stress-strain curve, which corresponds to the onset of dilatancy (Figure 3). (3) Finally, as the AE bursts, the differential stress reaches a peak 340 (marked as  $\sigma_v$ ) and then drops to a residual value. Among the synthetic samples deformed under 341 triaxial conditions, samples of porosity below 0.26 demonstrated brittle behavior up to effective 342 pressures of 30 to 60 MPa, depending on their grain diameter. The peak stresses  $\sigma_v$  for samples 343 deformed in the brittle regime are compiled in Table 2. 344

Figure 4 shows the third type of mechanical data obtained in this study, i.e., mechanical data for 345 triaxial tests conducted on synthetic samples at relatively high confinement and which failed by 346 347 shear-enhanced compaction. On Figure 4, the stress-strain curve can be delimited into two main portions. (1) As we first load the sample, axial strain increases linearly with differential stress 348 and no AEs are recorded. (2) Then, a subtle decrease in the slope of the stress-strain curve takes 349 place as the AEs start to increase at the onset of shear-enhanced compaction C\* (Wong et al., 350 1997). Finally, the stress-strain curve reaches a plateau punctuated by stress drops that correlate 351 to spikes in the AEs. These stress drops are suggestive of the formation of compaction bands 352 (Baud et al., 2004). All samples deformed in the range of effective pressures corresponding to 353 shear-enhanced compaction demonstrated such stress drops, sometimes accompanied by strain 354 hardening. The critical stresses C\* for samples deformed in the ductile regime are compiled in 355 Table 2. 356

An overview of the mechanical data collected for this study is presented in Figure 5. Mechanical 357 data for triaxial experiments are compiled with their corresponding hydrostatic pressurization 358 359 curves, for samples of porosity ranging from 0.18 to 0.38 and for mean grain diameters of 1.15 (blue), 0.5 (green) and 0.2 (orange) mm. At low and high effective pressures, the mechanical 360 data present the phases described for Figure 3 and Figure 4, respectively. The mechanical data 361 for triaxial compression follow the hydrostat in the poroelastic domain. The deviation from the 362 hydrostat marks the transition to inelastic deformation, either by dilatancy (increase in porosity) 363 or by shear-enhanced compaction (porosity reduction). Some samples deformed in a mode that 364 cannot be easily defined as "brittle" or "ductile". The mechanical data for the experiments 365 performed at effective pressures of 30, 60, and 90 MPa in Figure 5(c) are representative of this 366 hard-to-define failure mode, which we will refer to as transitional. For all the experiments, peak 367 stresses and critical stresses were identified to map out the failure envelopes of our synthetic 368 samples (Table 2). 369

370

#### 4.2 Critical stress states: effect of porosity and grain size

For all experiments, critical stress values were identified in accordance with the failure mode (Figures 2, 3 and 4). Regarding experiments conducted in the brittle and transitional regime, critical stresses P and Q were respectively identified at the peak and at the first stress drop. For experiments conducted in the ductile regime, the stresses P and Q were identified at the deviation from the hydrostat, i.e., at the onset of shear-enhanced compaction C\* (Figure 5). Table 2 includes all experiments for which the critical stresses could be clearly identified using mechanical data and AE measurements.

When plotted in the effective mean stress P - differential stress Q space, peak stresses map out 378 the brittle failure envelope (open symbols) and C\* values map out the compactive yield envelope 379 (solid symbols). When it could be measured, P\* anchors the yield envelope on the x-axis (P\* 380 381 could not be measured for all combinations of porosity and grain size due to the pressure 382 limitations of the triaxial press). The experiments that exhibited a transitional failure mode exist where the brittle envelope meets the yield cap. Figure 6 presents a compilation of failure 383 envelopes for our synthetic samples. Overall, several common features of the envelopes should 384 385 be noted. First, brittle failure of these porous materials is restricted to a small area of the stress space. Second, regarding compactive yield caps, P and Q are linearly correlated, which is 386

387 particularly clear for the caps on Figure 6(c). Third, shear-enhanced compaction occurs over a

- 388 wide range of stress states. For a given grain diameter, porosity is seen to influence compactive
- 389 yield behavior. Broadly speaking, the higher porosity, the lower the stress that required for
- inelastic yield. For example, samples of mean grain diameter of 1.15 mm (Figure 6(a)) submitted
- to triaxial compression under an effective pressure of 120 MPa yielded at 23 and 35 MPa of
- differential stress for initial porosities of  $\varphi = 0.30$  and  $\varphi = 0.26$ , respectively. For samples of
- 393 mean grain diameter of 0.5 mm (Figure 6(b)), triaxial compression under an effective pressure of
- 100 MPa resulted in critical differential stresses of 100 MPa for  $\varphi = 0.26$  compared to 52 MPa
- for  $\phi = 0.30$ . Finally, for synthetic samples of mean grain diameter of 0.2 mm (Figure 6(c))
- deformed at  $P_{eff} = 60$  MPa, inelastic yielding took place at 223 MPa when  $\varphi = 0.22$ , 147 MPa
- 397 when  $\varphi = 0.26$ , 95 MPa when  $\varphi = 0.30$ , and 42 MPa when  $\varphi = 0.35$ . In summary, increasing the
- porosity from 0.22 to 0.35 decreased the stress required for C\* by more than a factor of five
- 399 (Table 2).
- 400 All else being equal, grain diameter also exerts an important influence on the compactive
- 401 behavior. Figure 7(a) and Figure 7(b) presents a compilation of caps for  $\varphi = 0.30$  and 0.25-0.26,
- 402 respectively, for three different mean grain diameters (1.15 mm in blue, 0.5 mm in green, and 0.2
- 403 mm in orange). For  $\varphi = 0.25-0.26$  and  $P_{eff} = 60$  MPa, shear-enhanced compaction started at 59,
- 404 97 and 147 MPa for monodisperse samples of 1.15, 0.5 and 0.2 mm of mean grain diameter
- 405 respectively. For  $\varphi = 0.30$  and  $P_{eff} = 120$  MPa, C\* was reached at 23, 39 and 77 MPa for samples
- of 1.15, 0.5 and 0.2 mm of mean grain diameter respectively. In summary, increasing the mean
  grain diameter from 0.2 to 1.15 mm decreased the stress required for C\* by more than a factor of
- 408 two (Table 2).

#### 409 **5 Microstructural observations**

- 410 Representative SEM micrographs for the microstructure of a synthetic sample after hydrostatic
- 411 compression to beyond P\* are presented in Figure 8. The images correspond to a sample with an
- 412 initial porosity and mean grain diameter of 0.357 and 0.2 mm (sample 3312; see Table 2),
- 413 respectively, deformed up to a porosity reduction of 0.16. The corresponding mechanical data are
- 414 presented in Figure 5(i). At the lowest magnification, the thin section shows extensive
- 415 delocalized grain crushing. Zooms into the microstructure confirm that most grains were entirely
- 416 crushed and that the resulting shards progressively filled the porosity as the sample compacted.

417 Uncrushed glass beads allow for the observation of cross-cutting microfractures propagating
418 from grain to grain. On the basis of 2D image analysis (using ImageJ), the local final porosity

was estimated. The least and most damaged areas yielded porosity values around 0.30 and 0.11,respectively.

Representative SEM for the microstructure of a synthetic sample triaxially deformed to beyond 421 C\* are presented in Figure 9. The images correspond to a sample with an initial porosity and 422 mean grain diameter of 0.35 and 0.2 mm (sample 3314; see Table 2), respectively, triaxially 423 424 deformed at an effective pressure of 80 MPa up to an axial strain of 3.5%. The corresponding mechanical data are presented in Figure 5(i). As suggested by the small stress drops punctuating 425 the stress-strain curve beyond  $C^*$  and the corresponding bursts of AE activity (Figure 5(i)), the 426 sample contains evidence of compaction localization. Several discrete bands were observed in 427 the upper and lower parts of the thin section, i.e., at the extremities of the sample, and one cross-428 429 cutting discrete band was observed in the middle (Figure 9). The compaction band in the middle is 2-5 grains wide -i.e., thickness of 0.4 to 1 mm - and is oriented normal to the direction to the 430 431 maximum principal stress  $\sigma_1$ . Note that the band appears to avoid the porosity patches, and thus slaloms between them. A zoom on the band shows extensively fractured and compacted glass 432 beads (Figure 9). Shards resulting from the fracturing and crushing fill the porosity within the 433 band, reducing the porosity from 0.35 to approximately 0.27 (estimation based on 2D 434 435 measurements on the SEM images using ImageJ). The grains are unaffected outside the compaction band, and the porosity was estimated using ImageJ to be similar to that measured in 436 437 the laboratory (0.36).

#### 438 6 Discussion

#### 439 6.1 Suitability of sintered glass beads as crustal analogues

440 Synthetic granular rocks such as our sintered soda-lime silica glass beads provide a well-

441 characterized two-phase medium for investigating mechanical processes in siliciclastic rock. Our

- 442 motivation for using synthetic samples was to quantify the influence of individual
- 443 microstructural parameters (e.g., porosity and grain diameter) on the mechanical behavior of
- 444 granular rock by keeping all other parameters constant. In natural sandstones, for example,
- samples with different porosities may also be characterized by different grain and pore sizes and

distributions. However, understanding the mechanical behavior of sandstones using fused glass 446 bead synthetics hinges on the comparability of natural and synthetic sandstones. Before 447 discussing the suitability of sintered glass beads as analogs for crustal rocks, we will briefly 448 mention the differences between the microstructure of our synthetic samples and natural crustal 449 rocks. First, the grain size distribution of all the synthetic samples is monomodal and closely-450 clustered (Figure 1a). Natural sandstones, for example, can be characterized by polydisperse 451 grain size distributions. Second, the grains in our synthetic samples are spherical and have 452 identical physical and mechanical properties, while natural sandstones often contain non-453 spherical grains and different types of grains (e.g., quartz and feldspar). Finally, natural 454 sandstones can contain cement (e.g., clay cement between grains). Our synthetic samples do not 455 contain cement (Figure 1c and 1d). To compare our synthetic samples to natural sandstones, we 456 457 selected sandstones whose porosity and grain diameter lie in the range covered by our synthetic samples (our porosity range is 0.18-0.38 and our grain diameter range is 0.2-1.2 mm). We chose 458 Boise sandstone (porosity of 0.35 and average grain radius 0.46 mm; Bedford et al., 2019; Zhang 459 et al., 1990), Idaho Gray sandstone (porosity of 0.36 and average grain radius  $0.7 \pm 0.2$  mm; 460 461 Bedford et al., 2019) and Bentheim sandstone (porosity of 0.23 and average grain radius 0.3 mm; Klein et al., 2001). Bentheim sandstone is a monomineralic sandstone with a narrow grain size 462 463 distribution. Due to its homogeneous mineralogy and well sorted grain size, it has been used in many rock deformation studies, notably on strain localization (Baud et al., 2004; Tembe et al., 464 465 2006, 2008; Wong et al., 2001). It is therefore an ideal sandstone to compare with our synthetic samples. We compiled mechanical data from hydrostatic and triaxial experiments conducted in 466 conditions similar to those imposed during experiments on the synthetic samples, i.e., at room 467 temperature on water-saturated samples at a fixed pore pressure of 10 MPa. In Figure 10, we plot 468 469 selected mechanical data from our database alongside mechanical data from hydrostatic experiments (a) and triaxial experiments (b) conducted on Boise, Idaho Gray and Bentheim 470 sandstones. 471

Regarding the hydrostatic behavior. (Figure 10(a)), we first note that, during the initial loading
and increase of the effective pressure up to P\*, Boise, Idaho Gray, and the synthetic sample with
a porosity of 0.35 present porosity reduction curves that are almost identical. The characteristic
"tail" at the beginning of the hydrostat is typically attributed to the closure of microcracks
(Walsh, 1965). Assuming our sintered glass beads do not contain microfractures at the beginning

of the hydrostatic pressurization, as indicated from our microstructural analysis of the intact 477 material, we attribute the non-linear initial portion of the hydrostat to grain rotations and 478 479 rearrangements, which is corroborated by the positive correlation between the size of the tail (i.e., the amount of compaction) and the porosity of the sample. Qualitatively speaking, the 480 compaction curves evolve differently beyond P\*. While a progressive inflection and strain 481 hardening is observed for both Boise and Idaho Gray sandstones, P\* manifests as a sharp 482 breaking point beyond which the synthetic sample undergoes a porosity reduction of about 0.1 483 without hardening. Zhang et al. (1990) demonstrated that the first inflection in the hydrostat 484 corresponds to the inception of grain crushing and that increasing the effective pressure beyond 485 this point exacerbates the deformation. This gradual behavior is absent for the synthetic sample, 486 which experiences extensive grain crushing and porosity loss at the state of stress just higher 487 than P\*. The observation of extensive grain crushing at a stress just above P\* is similar to that 488 reported for Bentheim sandstone, a rock that also contains a closely-clustered monomodal grain 489 size distribution (Baud et al., 2006)(Figure 10(a)(c)). Examination of the microstructure showed 490 that very few areas in the sample remained uncrushed (Figure 8). Contrary to most natural 491 492 sandstones (e.g., Caruso et al., 1985), our synthetic rocks are composed of monomodal distributions of uniform grains of identical elastic properties. Thus, the force chains induced in 493 494 the granular framework during loading are expected to be more homogeneously distributed in our monodisperse synthetic samples (Guéguen & Boutéca, 2004; Papadopoulos et al., 2018). As 495 496 a result of this homogeneity, when the externally applied effective pressure reaches the critical value P\*, the normal forces induced at the grain contacts must reach the critical value at the same 497 498 time, and most grains are thus crushed at the same state of stress. Quantitatively, the effective stress at which the onset of grain crushing (P\*) occurs is higher in our synthetic rock (120 MPa) 499 500 than it is in Boise (75 MPa) and Idaho Gray (55 MPa) sandstones. Several differences between the synthetic and natural samples could be considered to explain the higher P\* in the synthetic 501 samples. First, Boise and Idaho Gray sandstone have a larger average grain diameter. Second, 502 Boise and Idaho Gray sandstone contain minerals other than guartz that are characterized by 503 504 lower values of fracture toughness, such as feldspar (Atkinson & Meredith, 1987). However, although the mineral composition of the two sandstones is very close, the P\* of Boise sandstone 505 is about 25 MPa higher than that of Idaho Gray sandstone (Figure 10(a)). Therefore, we 506

speculate that the much higher P\* for the synthetic samples could arise from the difference in
grain diameter (the smaller the grains, the stronger the sample).

We will now compare the behavior of natural sandstones with the one of our synthetic samples 509 when subject to triaxial compression. Figure 10(b) presents mechanical data from triaxial tests 510 conducted on a synthetic sample with a porosity of 0.3 (orange line) and on Bentheim sandstone 511 (Baud et al., 2004) (black line) under an effective pressure of 120 MPa. Qualitatively, the stress-512 strain curves are very similar. Quantitatively, C\* is about 50 MPa higher in Bentheim sandstone 513 514 than in the synthetic sample and is likely the result of the difference in porosity and grain size (both higher for the synthetic sample). Beyond C\*, the mechanical data for both samples show 515 small stress drops, suggesting that the samples failed by development of compaction localization, 516 as shown by Baud et al. (2004). 517

518 Although studies on the mechanical behavior of tuffs under hydrostatic and triaxial compression are comparatively rare (e.g., Heap et al., 2015a; Zhu et al., 2011), our new data for sintered 519 520 synthetic samples are also relevant to welded granular materials. Indeed, in the case of welded tuffs – the product of the deposition of hot volcanic ash and lapilli – our samples are an exact 521 522 analog, where volcanic welding and glass sintering are fundamentally the same dynamic process (Wadsworth et al., 2019). We note that in nature, welding of tuff can be associated with 523 internally porous clasts, vesiculation or resorption, viscosity or temperature gradients, and shear, 524 all of which can conspire to complicate microstructure relative to sintered glass beads, but that 525 nevertheless, the broad theme of mechanical results given here are relevant in volcanic 526 527 environments as well as other crustal scenarios.

#### 528 6.2 Deconvolution of microstructural parameters

In nature, porosity is often related to grain diameter. However, numerous other parameters such 529 530 as grain sorting, shape, orientation, location of cements, and the extent of compaction, can influence the bulk porosity of a porous rock (Rogers & Head, 1961; Scherer, 1987). One of the 531 532 results of this multi-component control on porosity is that crustal rocks that show a low porosity are not necessarily composed of small grains and vice versa. In fact, crustal rocks span a wide 533 range of porosity-grain size combinations and can demonstrate complex porosity-grain size 534 relationships. For illustration, we have compiled the porosity and the mean grain diameter of 19 535 natural sandstones that have repeatedly been used in laboratory studies (Figure 11). Porosity 536

values are in the range 0.03-0.38 and mean grain diameter values are in the range 0.075-0.92 mm. 537 If we consider only this subset of natural sandstones, several observations can be made: (1) for 538 539 the few sandstones with a porosity higher than 0.25, the mean grain diameter varies over a range twice as large as sandstones of lower porosity (0.2-0.9) and (2) for sandstones of porosity lower 540 or equal to 0.25, mean grain diameter is lower than 0.5 mm and clusters around 0.28 mm; such 541 that the grain size effect on porosity becomes attenuated as diagenesis progresses as pore and 542 pore throats are compacted. By compiling these data, we can conclude that (3) sandstones that 543 come from a single formation (see for example Buntsandstein, Fontainebleau or Boise 544 sandstones) can cover a large range in grain diameter and porosity, within which variations in 545 grain diameter appear to occur independently from variations in porosity and vice versa. For 546 example, the porosity of Fontainebleau sandstone can vary greatly (0.03-0.28), while the mean 547 grain diameter (0.250 mm) remains constant (Bourbie & Zinszner, 1985; Lindquist et al., 2000; 548 Louis et al., 2007). It is important to note that there is some bias in sample selection for 549 laboratory studies, such that crustal rocks with a low variability within a unit are favored so that 550 repeat measurements can be made (e.g., Menéndez et al., 1996). We can find that field studies 551 552 reported a much wider range of average grain size and porosity for sandstones, which can be encountered as very fine-grained (0.0625 mm; Selley, 2004) and can grade up to very coarse-553 554 grained and pebbly (2 mm; Selley, 2004), with well to poorly-sorted distributions and porosity ranging over more than one order of magnitude 0.02-0.30 (e.g., Morrow, Nugget, Bartlesville, 555 556 and Grimsby sandstone; Nelson & Kibler, 2003). For instance, anomalously high porosities were reported in a significant number of deeply-buried (> 4 km) reservoir sandstones worldwide (e.g., 557 558 porosity in the range 0.24-0.40 in the Tertiary channel-fill sandstone, offshore west Africa; Bloch et al., 2002). 559

Deconvolving structural parameters such as porosity and grain size is necessary to derive 560 definitive constraints on the micromechanics of compaction from experimental studies. Indeed, 561 while the importance of porosity in controlling yield strength is well-established for crustal rocks 562 (e.g., Wong & Baud, 2012), the independent effect of a change of grain or pore size is only 563 poorly investigated (Atapour & Mortazavi, 2018). As revealed when compiling grain diameter 564 565 and porosity for laboratory sandstones (Figure 11), it is possible that the approximate consistency in the grain diameter has meant that its influence on compactive yield strength has been masked 566 in rock mechanics study thus far. Sintering glass beads has allowed us to effectively deconvolve 567

the effect of porosity and grain diameter and other microstructural factors, to parameterizespecifically for their importance.

570 6.3 Influence of porosity and grain size on compactive yield

Our synthetic samples were designed and prepared to maximize microstructural homogeneity. 571 572 Yet, they present heterogeneities in the porosity distribution and in the geometry of grain-tograin contacts (Figure 1). Similar porosity clusters have been reported in Bleurswiller sandstone, 573 574 the mechanical compaction of which has been investigated in several studies (Baud et al., 2015; Fortin et al., 2005, 2006; Tembe et al., 2008). These published works have demonstrated the 575 576 importance of porosity clusters on the micromechanical processes leading to inelastic compaction. Indeed, while yield envelopes reported for natural sandstones are typically elliptical 577 578 in shape (Baud et al., 2006; Guéguen & Fortin, 2013; Wong et al., 1997), Bleurswiller sandstone presents an approximately linear yield cap (Baud et al., 2015). The linear yield envelope of 579 580 Bleurswiller was fitted by Baud et al. (2015) using a dual-porosity micromechanical model for cataclastic pore collapse. The pore collapse model, initially developed for dual-porosity 581 carbonates, treats the pore size distribution as bimodal with the pore space divided into 582 microporosity and macroporosity (i.e., a porosity cluster) (Zhu et al., 2010). Assuming the matrix 583 into which porosity clusters are embedded fails according to the Coulomb criterion, the pore 584 collapse model predicts that a porosity cluster collapses when the stress field in its vicinity 585 attains the critical state according to the Coulomb criterion, which results into a linear 586 dependence of the differential stress O at the yield point C\* with the effective stress P (Baud et 587 al., 2015). Although our yield caps that include P\* appear linear, further microstructural analysis 588 need to be done to identify the micromechanical process and clarify the role of pore collapse in 589 590 the failure of our synthetic samples.

The pore space heterogeneities of our synthetic samples appear to influence the micromechanics of failure. Yet, we believe they do not prevent us from discussing the relative influence of bulk porosity on the compactive yield behavior. A compilation of six yield envelopes for synthetic samples of porosity of 0.25 and 0.30 and of mean grain diameter of 0.2, 0.5 and 1.15 mm is presented in Figure 12. All of the compactive yield caps are linearly shaped with a negative slope. Overall, we observed that, for a given grain diameter, increasing porosity decreases the stress at which C\* occurs and that a difference in porosity of 0.01 results in a difference in C\* of approximately 8% + 5%. This appears to apply whatever the grain diameter is in the range

599 0.15-1.3 mm. Indeed, at an effective pressure of 60 MPa, an increase of porosity from 0.26 to

600 0.30 (+0.04), decreases the stress at which C\* occurs from 59 to 43 MPa (-28%), 97 to 70 MPa (-

- 601 28%) and from 147 to 95 MPa (-35%) for mean grain diameter of 1.15, 0.5 and 0.2 mm,
- 602 respectively.

As for porosity, grain size was experimentally identified to have a first-order control on 603 604 compactive yield of porous siliciclastic rock (Wong, 1990; Wong et al., 1992, 1997; Zhang et al., 605 1990) and has been included as a parameter in micromechanical models (e.g., Sammis & Ashby, 1986; Zhang et al., 1990). In the Hertzian fracture model of Zhang et al. (1990), average grain 606 radius acts as a scaling parameter for the critical pressure P\* with an equal weight than porosity. 607 This model was successfully applied to a number of natural and synthetic sandstones and 608 609 unconsolidated materials (David et al., 1998; Wong et al., 1997) with some scatter. However, 610 although a consensus on the key influence of grain size has been reached, compactive yield caps compilations for sandstones often only discuss the influence of porosity. Figure 13 shows a 611 compilation of compactant failure caps for Boise and Bleurswiller sandstones (data from Cheung 612 et al., 2012) and sintered samples that only differ from one another in terms of their average 613 grain diameter. If we consider only the yield caps of the synthetic samples, we observe that, all 614 else being equal, an increase in average grain diameter from 0.2 to 0.5 mm (+130%) or from 0.5 615 to 1.15 mm (+150%) shifts the stress at which C\* occurs to values approximately 2 times lower 616 (-50%) (Figure 13). Moreover, the difference in C\* that results from a change in grain diameter 617 remains approximately the same whatever the porosity. Thus, our data show that an increase in 618 average grain diameter by a factor of 2 results in a decrease in the stress to reach C\* of 619 approximately 50 +/- 5%. As for Boise and Bleurswiller sandstone, they are similar in 620 mineralogy and porosity but, although both their grain diameter distributions present a peak at 621 125 µm, the former has a wider sorting that extends up to 725 µm. Despite significantly different 622 grain sorting, their compactive yield caps for the onset of shear-enhanced compaction are very 623 similar, albeit slightly different in shape with a more linear cap for Bleurswiller sandstone. The 624 caps of our synthetics are similar in shape to those of the natural sandstones but are mapped out 625 626 at very different stress states (Figure 11). Tembe et al. (2008) reported that Bentheim sandstone, although similar to Boise and Bleurswiller sandstones in terms of porosity, presents a compactive 627 yield cap characterized by higher stresses. Indeed, the abundance of secondary minerals (feldspar, 628

oxide and mica) in Boise and Bleurswiller compared to Bentheim (>99% guartz) likely causes 629 the decrease in the compactive yield stresses for Boise and Bleurswiller sandstones. Similarly, 630 631 since Bleurswiller and Boise sandstones present a grain diameter distribution with a peak at 125 μm, we would expect their compactive yield caps to be mapped out at higher stresses than the 632 caps for the synthetic samples that have grain diameter distributions with peaks at 200, 500 and 633 1150 µm (Figure 13). The discrepancy in compactive yield stresses between Boise and 634 Bleurswiller sandstones and the synthetic samples can be attributed neither to porosity nor to 635 grain size, but possibly to the presence of cement and of secondary minerals other than quartz 636 (feldspar, oxide and mica). 637

Overall, we varied grain diameter by one order of magnitude (Figure 11) and we see a large 638 effect of that variation on the yield compactive strength of our synthetic samples. Grain 639 diameters of natural sandstones can also vary by more than one order magnitude (Nelson & 640 641 Kibler, 2003), but the effect of that variation has not hitherto been deconvolved from other microstructural factors. We thus conclude that, if grain size were to be accounted for 642 643 quantitatively, its effect would be similar to that of porosity. However, we observe, for the range of porosity and grain diameter used herein, that the influence of porosity on compactive yield is 644 higher than the influence of grain diameter. Indeed, to cover a similar range in the stress space, 645 the average grain diameter of our synthetic samples was increased by up to 600 % (relative to the 646 647 lowest range we used, 0.15-0.25 mm) while bulk porosity was only increased by up to 120% (relative to the lowest porosity we used, 0.18). 648

## 649 6.4 Compaction localization

650 For all of our synthetic samples deformed in the regime of shear-enhanced compaction, 651 mechanical data show stress drops of variable amplitude (Figure 5), suggesting that compaction localization took place (Baud et al., 2004; Heap et al., 2015b; Louis et al., 2006). This 652 observation concurs with the general consensus that microstructural homogeneity promote strain 653 654 localization in granular materials (Katsman et al., 2005; Louis et al., 2009; Wang et al., 2005). Indeed, Cheung et al. (2012) demonstrated that uniform grain size distribution promotes the 655 development of discrete compaction bands. As our synthetic samples are characterized by a 656 657 monodisperse distribution of grain size, we expected compactant deformation to localize in the form of compaction bands. In Figure 10(c) and Figure 10(d), we juxtaposed a SEM micrograph 658

of a discrete compaction band in a synthetic sample and a micrograph of a discrete compaction 659 band in Bentheim (from Baud et al., 2004), respectively. The compaction bands present very 660 661 similar microstructural attributes (Figure 10(c)(d)). An important difference is that our micrograph has been obtained on a sample of porosity of 0.35. To our knowledge, the range of 662 porosity over which compaction bands were reported in sandstones is approximately 0.13; 0.29 663 (Fossen et al., 2011; Schultz et al., 2010; Tembe et al., 2008). The mechanical behavior of 664 sandstones with a porosity higher than 0.29 at or near the brittle-ductile transition has been 665 studied (e.g., Bedford et al., 2019; Cheung et al., 2012; Wong et al., 1997) but high-porosity 666 sandstones typically used in laboratory often have polydisperse distributions of grain size (see 667 for example, Boise sandstone), which has been recognized to inhibit strain localization. 668 Therefore, the effect of porosity on the propensity for compaction localization may have been 669 masked by the influence of other structural parameters. Our new data therefore extend the upper 670 limit of porosity for which compaction localization has been observed to 0.35 and suggest that 671 compaction localization can occur in samples with a porosity up to 0.38 (the highest porosity of 672 our set of samples). 673

However, although numerical simulations suggested compaction localization could occur in sand 674 packs (Marketos & Bolton, 2009), experimental validation for the formation of compaction 675 bands in high-porosity granular aggregates such as unconsolidated sands has not been reported. 676 677 For example, Hangx and Brantut (2019) performed triaxial experiments on Ottawa quartz sand with a porosity of 0.36 and did not observe strain localization in the compactant regime of 678 deformation. These authors proposed that the possibility for grain rotation and rearrangement – 679 permitted by the lack of cementation – allows grain failure to be accommodated and prevents 680 stress concentration to occur. Although our high-porosity synthetic samples do not have cement, 681 we show that they can develop compaction bands in the regime of shear-enhanced compaction. 682 Therefore, we speculate that the necks formed at grain contacts during sintering in our synthetic 683 samples act as the cement in consolidated sandstones and play a key role in controlling 684 compaction localization. To a first-order, the potential for compaction localization appears to be 685 controlled not by porosity, but by the granular/non-granular and/or unconsolidated/consolidated 686 687 nature of rock, which is intimately related to the degree of cementation at grain contacts and by extension, in some cases, to porosity (Lemée & Guéguen, 1996). Additionally, if we consider 688 that the porosity of a loose packing of grains is approximately  $0.38 \pm 0.01$  (Johnson & Plona, 689

690 1982), the observation of discrete compaction bands in a synthetic sample of porosity of 0.35

691 suggests that even a small proportion of consolidated/cemented grain contacts could be sufficient

692 to trigger stress concentrations within aggregates and the formation of compaction bands.

#### 693 7 Crustal implications and concluding remarks

694 Crustal rocks such as sandstones and tuffs, the primary microstructural elements of which are comparable with our synthetic samples, occur as geological units in reservoirs, aquifers, fault 695 696 zones and in volcanic environments; settings where they typically undergo structural changes due to geologic processes. Therefore, implications – and applications – of our results for natural 697 698 systems are broad. For example, in the context of hydrocarbon and/or geothermal reservoirs, depletion-induced reservoir compaction is an ubiquitous phenomenon that eventually leads to 699 700 surface subsidence (Gambolati et al., 2006; Nagel, 2001). On assessing which sedimentary layer compacts first and/or to the highest extent, unconsolidated upper formations and clay-rich 701 702 formations are usually considered as the best candidates. However, reservoir formations are often only vaguely described as coarse- or fine-grained and grain size is rarely considered in numerical 703 terms (Sun et al., 2018b), even in geotechnical models predicting the extent of irreversible 704 compaction for the bulk reservoir (Buscarnera et al., 2020; Hol et al., 2018). Our new data 705 706 suggest that formations with large grain diameters, alongside those with a high porosity, could be prime candidates for mechanical compaction and should therefore be considered when assessing 707 reservoir subsidence. 708

In volcanic contexts, inelastic compaction of edifice-forming rock (including non-volcanic 709 basement rocks) presumably acts as a driving force in the growth and destruction life-cycle of 710 large volcanoes (Bakker et al., 2015; Concha-Dimas et al., 2005; Heap et al., 2015c; Van Wyk 711 712 De Vries & Borgia, 1996), which involves episodes of spreading that eventually leads to catastrophic collapses (Van Wyk De Vries & Francis, 1997). Since flank and/or edifice collapse 713 714 models often invoke a weak/ductile internal or basal unit to explain instability and collapse 715 (Ablay & Hürlimann, 2000; Morgan & McGovern, 2005; Voight, 2000), it is important to 716 understand what controls the mechanical behavior of porous rocks, especially considering that porous volcanic rocks can also develop compaction bands (Heap et al., 2015b, 2020). Our 717 718 synthetic materials could help understand whether simple empirical or theoretical models can 719 effectively describe the relationship between grain size, porosity and compactive yield strength,

and thus give accurate predictions for the evolution of inelastic compaction and subsequent

subsidence and/or edifice spreading. Moreover, since our synthetic samples consist of a very

simplified two-phase medium, such laws can be easily tested against discrete element method

simulations of reservoir compaction (Alassi et al., 2006; Sun et al., 2018a) or volcanic collapses

724 (Harnett et al., 2018) for example.

Our approach has allowed us to study the influence of deconvolved microstructural attributes on 725 mechanical compaction. The set of mechanical and microstructural data we present show that the 726 727 failure mode of analog samples made of sintered glass beads transit from brittle at low 728 confinement to ductile with shear-enhanced compaction at high confinement. Compactive yield caps are mapped out on a range of stress states comparable to those for natural crustal rocks (the 729 porosity and grain diameter of which are similar to those of our synthetic samples, i.e., 0.18-0.38 730 731 and 0.2-1.15 mm, respectively) and are linearly shaped when P\* is known and are likely linearly shaped for the porosity-grain diameter combinations for which P\* could not be measured (due to 732 the pressure limit of the triaxial apparatus). Qualitatively speaking, mechanical and 733 734 microstructural data are very similar between the natural and synthetic samples. Regarding the influence of porosity and grain size, we arrived at the following main conclusions. First, 735 increasing only porosity or only grain diameter decreases the stress at which the onset of shear-736 enhanced compaction C\* occurs. Second, to increase the stress at C\* by 50%, porosity has to be 737 738 decreased – in isolation – by 0.06 (30% relative to the range 0.18-0.38) whereas average grain diameter has to be decreased – in isolation – by 0.50 mm (53% relative to the 0.2-1.15 mm). 739 Although the influence of porosity can be regarded as higher than the influence of grain size, our 740 study demonstrates that, over the investigated range of porosity and grain diameter, they both 741 exert a first-order control on the mechanical compaction of natural crustal rocks, which can span 742 over a much broader range of porosity and grain diameters. Therefore, alongside porosity, grain 743 diameter should become a routinely measured structural parameter when dealing with the 744 mechanical compaction of natural crustal rocks. 745

Overall, we believe our study demonstrates the great suitability of sintered glass beads as crustal rock analogs and the great opportunity they embody for studying microstructural parameters such as porosity and average grain diameter in isolation. Since mixtures of glass beads of different diameters can be prepared, variably polydisperse sintered samples can be synthesized and used to investigate the influence of grain size distribution and polydispersivity on

- 751 mechanical and hydraulic behavior. Further, the addition of cement and/or other materials to the
- 752 glass bead mixtures could also be considered in order to sharpen our understanding of the
- deconvolved influence of microstructural parameters on the mechanical and hydraulic properties
- of crustal rocks.

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1127 Figure 2. Representative mechanical data (black line) and cumulative acoustic emission energy (purple dashed line) for hydrostatic tests performed on the synthetic samples. These data were 1128 obtained on synthetic samples of mean grain diameter of 0.2 mm and initial porosity of 0.38. The 1129 critical stress for the onset of grain crushing P\* is indicated by an arrow. The porosity reduction 1130 in percentage corresponds to the absolute loss of porosity, i.e., a porosity reduction of 14% refers 1131 to a drop from 0.38 to 0.24. (1) When Peff is first increased, porosity decreases non-linearly as a 1132 result of grains rearrangements. (2) As Peff is increased further, the sample undergoes elastic 1133 1134 deformation until P<sub>eff</sub> reaches the critical value P\* beyond which (3) the sample porosity decreases suddenly and significantly by grain crushing (inelastic deformation). 1135



1137 Figure 3. Representative mechanical data (black lines) and cumulative acoustic emission energy (purple dashed line) for triaxial tests performed in the brittle regime. The triaxial test presented 1138 1139 was performed at  $P_{eff} = 30$  MPa on a synthetic sample of mean grain diameter of 0.2 mm and initial porosity of 0.22. The peak stress  $\sigma_v$  is indicated by an arrow. The porosity reduction in 1140 percentage corresponds to the absolute loss of porosity, i.e., a porosity reduction of 0.4% refers 1141 to a drop from 0.22 to 0.216. (1) When loading is first applied, the sample undergoes elastic axial 1142 strain and porosity decreases linearly. (2) The transition to the inelastic stage of deformation 1143 takes place at the onset of dilatancy and, as Q is increased further, it eventually reaches (3) a 1144 critical peak stress  $\sigma_v$  at which point brittle failure takes place and the stress drops to a residual 1145 value. 1146



Figure 4. Representative mechanical data (black lines) and cumulative acoustic emission energy 1148 (purple dashed line) for triaxial tests performed in the regime of shear-enhanced compaction. The 1149 triaxial test presented was performed at  $P_{eff} = 180$  MPa on a synthetic sample of mean grain 1150 diameter of 0.2 mm and initial porosity of 0.30. The critical stress for the onset of shear-1151 enhanced compaction C\* is indicated by an arrow. The porosity reduction in percentage 1152 corresponds to the absolute loss of porosity, i.e., a porosity reduction of 2% refers to a drop from 1153 0.30 to 0.28. (1) Axial strain increases and porosity decreases linearly as loading is first applied. 1154 (2) The transition to the inelastic stage of deformation takes place as Q reaches the critical value 1155 C\* for the onset of shear-enhanced compaction. 1156





- 1158 **Figure 5.** Compilations of mechanical data from hydrostatic loading (dashed colored) and
- triaxial tests (black) for samples with a mean grain diameter of 1.15 mm (blue) and an initial
- porosity of (a) 0.18, (b) 0.26 and (c) 0.30; a mean grain diameter of 0.5 mm (green) and an initial
- porosity of (d) 0.25 and (e) 0.30; and a mean grain diameter of 0.2 mm (orange) and an initial
- 1162 porosity of (**f**) 0.22, (**g**) 0.25, (**h**) 0.30, (**i**) 0.35 and (**j**) 0.38. Effective pressures at which triaxial
- 1163 tests were conducted are indicated at the end of the corresponding curves. The onset for inelastic
- deformation corresponds to the departure of the effective mean stress porosity reduction curve
- 1165 from the hydrostat. For illustration, the onset of shear-enhanced compaction is indicated as C\* by
- black arrows. The critical stresses P\* for the onset of grain crushing are indicated by colored
- 1167 arrows on the hydrostats.



- 1169 **Figure 6.** Compilations of failure envelopes for synthetic samples of mean grain diameter of (a)
- 1170 1.15 mm, (**b**) 0.5 mm and (**c**) 0.2 mm. Initial porosity of the synthetic samples is indicated in the
- legend. Failure envelopes are mapped out by critical stresses  $\sigma_v$  (brittle triaxial test), C\* (ductile
- 1172 triaxial test) and P\* (hydrostatic test). Open symbols correspond to peak stress values and solid
- 1173 symbols to C\* values. P\* (also a solid symbol) anchors the envelope to the x-axis.





- 1175 **Figure 7.** Compilations of failure envelopes for synthetic samples with a porosity of (**a**) 0.30 and
- (**b**) 0.25. Mean grain diameter of the synthetic samples is indicated in the legend. Failure
- 1177 envelopes are mapped out by critical stresses  $\sigma_v$  (brittle triaxial test), C\* (ductile triaxial test) and
- 1178 P\* (hydrostatic test). Open symbols correspond to peak stress values and solid symbols to C\*
- 1179 values. P\* (also a solid symbol) anchors the envelope to the x-axis.



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Figure 8. Representative scanning electron micrograph of the (a) microstructure of a synthetic
sample deformed under hydrostatic loading up to an effective stress beyond P\*. (b)(c) Zooms in

showing extensive grain crushing. Black: porosity, gray: glass.



Figure 9. Scanning electron micrograph of the microstructure of a synthetic sample that failed by development of discrete compaction bands. Sample 3314, with a porosity of 0.35 and a mean grain diameter of 0.2 mm, was deformed under 80 MPa (Table 2). Overview of the thin section allows for the observation of a discrete compaction band in the middle, formed in a direction normal to the maximum principal stress  $\sigma 1$ . (a) and (b) show micrographs of a discrete 2-5

- 1192 grain-thick band within which most grains are crushed. The microstructure outside of the band is
- almost intact. Black: porosity, gray: glass.



Figure 10. Data from tests performed on synthetic samples compared to data for sandstones from the literature. (a) Comparison of the hydrostatic loading curve of a synthetic sample (green dashed) with a porosity of 0.35 and a mean grain diameter of 0.5 mm and of the hydrostatic loading curve of a synthetic sample (orange curve) with a porosity of 0.22 and a mean grain size of 0.2 mm, with the hydrostat of Boise sandstone (black line), with a porosity of 0.35 and a mean grain diameter of 0.92 mm (Zhang et al., 1990), the hydrostat of Idaho Gray sandstone (dashed black) with a porosity of 0.363 and a mean grain diameter of 0.7 mm (Bedford et al. 2019) and

- 1204 the hydrostat of Bentheim sandstone with a porosity of 0.228 and a grain diameter of 0.3 mm
- 1205 (Baud et al., 2006). The onset of grain crushing is indicated as P\*. (b) Comparison of stress-
- strain curves obtained during a triaxial test at an effective pressure of 120 MPa performed on a
- 1207 synthetic sample (orange line) with a porosity of 0.25 and a mean grain diameter of 0.2 mm and
- 1208 on Bentheim sandstone (black line)(Baud et al., 2004). The onset of shear-enhanced compaction
- 1209 is indicated as C\*. (c) For reference, the smallest grain size distribution used in this study is
- 1210 presented along the grain size distribution of Bentheim sandstone (data from Cheung et al. 2012).
- 1211 (d) Comparison of a scanning electron micrograph of a discrete compaction band observed in a
- 1212 synthetic sample ( $\varphi = 0.35$ ) and (e) an optical microscope image of a discrete compaction band
- 1213 in Bentheim sandstone ( $\varphi = 0.23$ ; Baud et al., 2004).
- 1214



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Figure 11. Compilation of porosity and grain diameter for laboratory sandstones. All data are 1217 from the literature, references are given in the legend. Colored areas correspond to the range of 1218 porosity-grain diameter accessible by sintering glass beads. The dashed areas correspond to the 1219 range we specifically investigated in this study. The error bars give the standard deviation of the 1220 grain diameter distribution, when it has been reported. 1221



**Figure 12.** Influence of porosity on the compactive yield strength. Compactive yield envelopes

- 1224 for synthetic samples with a porosity of 0.26 (round solid symbol) or 0.30 (triangle solid symbol)
- and a mean grain diameter of 1.15 mm (blue), 0.5 mm (green) or 0.2 mm (orange) are compiled.



1227 **Figure 13.** Influence of grain diameter on the compactive yield strength. Compactive yield

1228 envelopes for synthetic samples with a porosity of 0.25 and a mean grain diameter of 0.2 mm

1229 (orange), 0.5 mm (green) and 1.15 mm (blue) are compiled with yield envelopes for Boise (open

1230 cross) and Bleurswiller (open circle) sandstones (data from Cheung et al., 2012).