Micromechanics of high pressure compaction in granular quartz aggregates

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Abstract. The mechanical behaviour of porous sandstones is generally modelled using concepts from granular mechanics, often overlooking the effect of cementation. To probe the key differences between sand and sandstone mechanics, we performed triaxial deformation experiments on Ottawa quartz sand at 5-40 MPa effective confining pressure. At 5 MPa, the samples are able to dilate. At higher confinement, the aggregates show continuous compaction, displaying strain hardening. The stress-strain behaviour is non-linear and the exact onset of inelastic compaction could not be determined accurately. Measured P-wave velocities show the development of anisotropy. With increasing axial strain, the along-axis velocities tend to increase, while velocities perpendicular to the compression axis tend to decrease (at low pressure) or remain constant (at high pressure). In samples deformed under elevated pressure conditions, acoustic emission event locations are diffuse. Microstructural investigations show an increase in grain chipping and crushing with increasing confining pressure, but no evidence of localised compaction could be observed. The nature of the pore-fluid, either decane or water, does not significantly influence the mechanical behaviour at strain rates of 10^{-6} to 10^{-4} s⁻¹. Grain angularity and grain-size distribution also did not significantly change the mechanical behaviour. We infer that our observations indicate that the lack of cementation introduces additional degrees of freedom for grains to slide, rotate, and reorganise at the sample-scale, precluding the existence and sustainability of stress concentrations beyond the grain-scale. This results in progressive compaction and hardening, and lack of compaction localisation.

1. Introduction

Highly porous unconsolidated sands and poorly consolidated sandstones (porosity \geq 20%) constitute important reservoirs targeted for hydrocarbon extraction (e.g. of Mexico [Ostermeier, 1995; Li et al., 2003]; Slochteren Sandstone, Groningen Gas Field, the Netherlands [Hettema et al., 2002]). It is their high porosity which makes such reservoirs suitable for fluid production due to their often high permeability. However, changes in effective overburden stress due to fluid extraction can result in compaction at the reservoir level. Even relatively small amounts of compactive strain (few tenths of a percent) can lead to surface subsidence and induced seismicity [Doornhof et al., 2006; Spetzler and Dost, 2017; Pijnenburg et al., 2018, 2019, while larger strains (several percent) and strain localisation can lead to geotechnical problems, such a wellbore casing collapse [Doornhof et al., 2006; Li et al., 2003], or reduce reservoir permeability impacting production [Olsson et al., 2002]. Compaction is most likely caused by a direct poro-elastic response [Wang, 2000] and time- or rate-independent inelastic compaction, as well as time-dependent compaction creep, which may continue long after production has ceased [Doornhof et al., 2006].

Porous sandstones have been extensively studied and a vast body of work is dedicated to deformation and strain

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localisation in sandstone [see Wong and Baud, 2012, and references therein]. At very low confining pressure, strain localisation in porous sandstones has been shown to occur via dilatant shear localisation moving to compactant shear localisation at low to moderate confinement [Wong and Baud, 2012]. At higher confining pressures, compaction may transition towards strain localisation via discrete [Fortin et al., 2007; Tembe et al., 2008; Louis et al., 2006; Heap et al., 2015; Klein et al., 2001] or diffuse compaction bands [Baud et al., 2004; Olsson and Holcomb, 2000; Olsson et al., 2002] to distributed cataclastic flow under hydrostatic conditions [Baud et al., 2004. While discrete compaction bands (i.e. < 3 grains wide [Baud et al., 2004]) may be formed under both constant strain rate and constant stress conditions [Heap et al., 2015], they have been observed in very few sandstones deformed under laboratory conditions. It is believed that discrete compaction bands are promoted in high porosity $(\geq 20\%)$, well-sorted and spatially homogeneous sandstones Cheung et al., 2012, such as the Bentheim, Bleurswiller and Diemelstadt sandstone [Fortin et al., 2007; Tembe et al., 2008; Louis et al., 2006; Heap et al., 2015]. Furthermore, under wet conditions sandstones generally tend be significantly weaker [Baud et al., 2000a; Duda and Renner, 2012].

Studies on the uniaxial consolidation (porosity loss) behaviour of sands are numerous, focusing on the effect of grain-size and angularity, mineralogy and chemical environment [Brzesowsky et al., 2014a; Chuhan et al., 2003, 2002; Mesri and Vardhanabhuti, 2009; Fawad et al., 2011; Brzesowsky et al., 2014b]. These studies show that higher initial porosity, increasing grain angularity, increasing grain-size and size uniformity, decreasing intrinsic grain strength and the presence of aqueous fluids enhance consolidation [Brzesowsky et al., 2014b, a; Hangx et al., 2010; Chuhan et al., 2003, 2002; Lee and Farhoomand, 1967; Vesíc and Clough, 1968; Zoback and Byerlee, 1976]. Initial consolidation of sand is attributed to tighter packing promoting

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particle locking, while upon higher stresses interparticle slip and particle crushing and fracturing lead to further particle $unlocking \ and \ concomitant \ compaction \ [\textit{Mesri and Vardhan-}$ abhuti, 2009; Muñoz-Ibáñez et al., 2019]. Additionally, many studies exist in the soil mechanics community, regarding triaxial consolidation of sands and clays, though often at low confining pressures [e.g. see Pestana et al., 2002; Alikarami et al., 2015]. Under low confining conditions (confining pressures of 0.1 - 7 MPa), recent experiments using X-ray computed tomography show the formation of dilation and compaction shear bands [Alikarami et al., 2015], accommodated by grain rolling and frictional sliding [Alikarami et al., 2015; Karner et al., 2005]. Grain angularity impacts compaction and strain localisation behaviour by enhancing interlocking in more angular aggregates, which in turn inhibits dilation [Alikarami et al., 2015; Guo and Xubin, 2007]. However, systematic experimental study of the micromechanics of strain localisation in unconsolidated sands at higher confinement has received much less attention [Karner et al., 2005; Skurtveit et al., 2013; Nguyen et al., 2014].

Instead, extensive numerical modelling efforts have been made to predict the behaviour of compacting sands and sandstones, either through constitutive modelling [Issen and Rudnicki, 2000; Pestana and Whittle, 1999; Tengattini et al., 2014; Choo and Lee, 2018; Buscarnera and Laverack, 2014; Einav, 2007] or employing particle models such as Discrete Elements [Potyondy and Cundall, 2004; Wang et al., 2008; Wu et al., 2018; Marketos and Bolton, 2009]. Such numerical models are crucial in providing the means to extrapolate granular material behaviour across spatial and temporal scales. However, they generally assume that rocks can be approximated as grains held together by cement bonds [e.g. see Einav, 2007; Potyondy and Cundall, 2004]. This would imply that once sufficient cement bonds are broken, the material would essentially behave as a loose granular aggregate, as confirmed by low confinement experiments on loose sand and artificially cemented sand [Bernabé et al., 1992. While compaction bands in sand have not been experimentally verified, DE modelling efforts have suggested that it is possible to generate compaction bands in sand packs, though less pronounced than in sandstones [Marketos and Bolton, 2009].

Given the first-order similarities in mechanical behaviour between sands and sandstones, and the effectiveness of granular microphysical models to explain the deformation of sandstones, should imply that unconsolidated sand aggregates behave in the same way as sandstones. This would mean that in sand the onset of inelastic compaction is driven by the onset of grain crushing at the microscale. It also implies that shear bands will develop at low confinement [Wong and Baud, 2012], while compaction bands should be observed at high confining pressure (P_c) if the grainsize is narrowly distributed [Wong and Baud, 2012; Cheung et al., 2012. It should be noted that though this implies the assumption of a double-yield cap model, it has been shown that upon deformation of porous aggregates, and the concomitant compaction and porosity reduction, the initial yield cap can expand [e.g. Wong et al., 1992; Xiao and Evans, 2003; Bedford et al., 2018; Pijnenburg et al., 2019]. Essentially this means that any material has the potential to transition from one side of the yield cap to the other, while the yield cap expands, regardless of how the material is deforming. Experiments have already shown that at low confinement, dilatant shear bands do form in loose sand [Alikarami et al., 2015], similar to porous sandstones [Wong and Baud, 2012]. However, while sand experiments performed at higher confining pressure show significant grain crushing, as also seen seen in sandstones [Karner et al., 2005; Zoback and Byerlee, 1976], the micromechanical characteristics of strain localisation in this regime remain unclear.

We performed a series of triaxial deformation experiments on homogeneous, highly porous, unconsolidated quartz sand aggregates at effective confining pressures in the range 5-100 MPa. During the experiments, the effect of chemical environment (decane versus water), grain angularity (well-rounded versus subrounded/-angular) and grain-size distribution (narrow versus broad) on deformation behaviour is studied. We aim to highlight systematic qualitative and quantitative differences between our results on sand and observations on cemented sandstones, by making a systematic comparison between high-pressure deformation of sand versus sandstone, with a special focus on microstructural controls, strain localisation and acoustic characteristics.

2. Experimental methods

We performed two types of conventional triaxial experiments on fluid-saturated sand aggregates under room temperature conditions:

1. Two hydrostatic tests (OS-04 and OS-17) were done at $P_{\rm c}$ up to 55 MPa and 105 MPa, respectively. Decane-saturated test OS-04 was performed at fixed confining pressure steps of 2.5 MPa, while during each step the sample was allowed to reach a near-steady volumetric strain rate

Table 1. Conventional triaxial compression and hydrostatic experiments performed on fluid-saturated Ottawa sand aggregates ($d=300-400\mu\mathrm{m}$) at 20°C and $P_\mathrm{p}=5$ MPa, plus unloading Young's Modulus obtained in the tests. Additionally, mean grain size values are given when measured.

[%]	1.707	$P_{\mathrm{c}}^{\mathrm{eff}}$								
	[%]	[MPa]	[GPa]	$d_{50} \ [\mu \mathrm{m}]$						
				427-444						
Decane-saturated sand aggregates OS-03										
36.3	33.6	20	6.3	351-375						
36.2	33.5	25	6.8	351-353						
36.0	32.6	30	7.8	318-339						
36.1	36.1^{c}	5 - 50		410-427						
and ag	ggregat	es								
36.3	36.3^{c}	5	3.6							
36.2		10	4.5	438-439						
36.1	34.7	15		364-374						
36.3	34.3	20	5.7							
36.1	34.0	20	5.4							
34.6	32.9	20	5.9							
36.0	33.9	20	6.5	374-386						
36.3	33.8	25	6.7							
36.2	32.7	30	7.3	300						
	-			- 0 0						
	36.3 36.2 36.0 36.1 and ag 36.3 36.2 36.1 36.3 36.1 34.6 36.0 36.3	36.3 33.6 36.2 33.5 36.0 32.6 36.1 36.1° and aggregat 36.3 36.2 36.1 34.7 36.3 34.3 36.1 34.0 34.6 32.9 36.0 33.9 36.3 33.8 36.2 32.7 36.3 31.1	36.3 33.6 20 36.2 33.5 25 36.0 32.6 30 36.1 36.1° 5–50 and aggregates 36.3 36.3° 5 36.2 10 36.1 34.7 15 36.3 34.3 20 36.1 34.0 20 34.6 32.9 20 36.0 33.9 20 36.3 33.8 25 36.2 32.7 30 36.3 31.1 40	36.3 33.6 20 6.3 36.2 33.5 25 6.8 36.0 32.6 30 7.8 36.1 36.1° 5-50 and aggregates 36.3 36.3° 5 3.6 36.2 10 4.5 36.1 34.7 15 36.3 34.3 20 5.7 36.1 34.0 20 5.4 34.6 32.9 20 5.9 36.0 33.9 20 6.5 36.3 33.8 25 6.7 36.2 32.7 30 7.3 36.3 31.1 40 8.1						

Symbols: ϕ_0 is sample porosity prior to set-up, ϕ_i is the estimated sample porosity after application of $P_{\rm c}$ and $P_{\rm p}$, $P_{\rm c}^{\rm eff}$ represents effective confining pressure, $E_{\rm u}$ is the Young's modulus obtained from a linear fit to the linear (elastic) part of the stress-strain curve during unloading, d_{50} is the mean grain size obtained from particle size analysis (two measurements).

- $^{\rm a}$ The estimated porosity after application of confinement does not include any sample volume changes during the pore pressure application phase. Error is $\sim 0.6\%$ (see Appendix A).
- ^b Hydrostatic experiments: OS-04 performed at P_c steps of 2.5 MPa, while during each step the sample was allowed to reach a near-steady volumetric strain rate of $< 10^{-6} \text{ s}^{-1}$. OS-17 performed at a fixed confining pressure step rate of 2.5 MPa/10 mins.
- $^{\rm c}$ Experiment started immediately after application of the pore pressure ($P_{\rm c}^{\rm eff}=5$ MPa).
- ^d Sample experienced jacket leakage, so no pore volume changes could be obtained.
 - ^e Sample deformed up to 1.5% e.
 - $^{\rm f}$ Sample deformed up to 4% e.

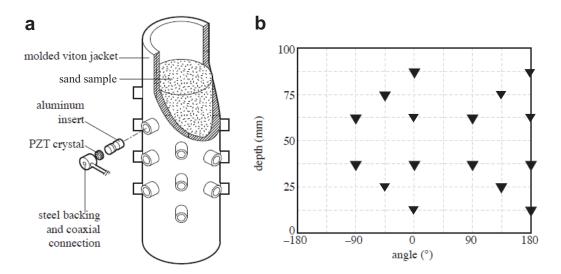


Figure 1. a) Schematic of the sample (diameter is 40 mm and length is 100 mm) and sample jacket. In total 16 acoustic transducers were mounted on molded inserts in the jacket. Unused inserts were blanked with aluminium plugs. b) Map of sensor positions around the sample.

of $<10^{-6}~\rm s^{-1}$. Water-saturated test OS–17 was performed at a fixed confining pressure step rate of 2.5 MPa/10 mins. Both experiments were performed on a grain-size batch of $350\pm50\mu \rm m$.

2. Sixteen triaxial deformation tests were done with applied confining pressures $(P_{\rm c})$ in the range 10-45 MPa, at a constant axial strain rate (é) of $10^{-5}~{\rm s}^{-1}$. After $\sim 5\%$ axial strain, strain rate was cycled several times from $10^{-6}~{\rm s}^{-1}$ to $10^{-4}~{\rm s}^{-1}$, to test the effect of strain rate on deformation. These tests were performed using three different grain-size batches $(350\pm50~\mu{\rm m},\,340\pm160~\mu{\rm m}$ and $240\pm60~\mu{\rm m})$.

The experiments were either performed using decane or water as pore-fluid (pore-fluid pressure, $P_{\rm p}=5$ MPa), to test the effect of chemical environment on deformation behaviour. A summary of the experiments, plus key parameters obtained during the tests, is presented in Tables 1 and 2.

2.1. Sample material and preparation

The sand used in 17 of the experiments was ASTM standard C778 Ottawa sand obtained from US Silica (Ottawa, IL, USA). Specifications indicated a SiO₂ content of 99.7 wt-%, with minor quantities of Al₂O₃ (0.06 wt-%), Fe₂O₃ (0.02 wt-%) and TiO₂ (0.01 wt-%). Scanning electron microscopy (SEM) showed that most sand grains are well-rounded, with smooth surfaces characterised by pressure solution indentations and quartz overgrowths. Fractions of the sand $(350\pm50~\mu\text{m},~340\pm160~\mu\text{m}$ and $240\pm60~\mu\text{m})$ were prepared by sieving twice the material as received. Additionally, one experiment was performed using Beaujean sand, the same as used by Brzesowsky et al. [2011, 2014a, b]. This material was obtained from the Heksenberg Formation, Beaujean quarry, Heerlen, Netherlands, and sieved to a grain-size fraction of $240\pm60~\mu\text{m}$. Compared to Ottawa sand, the Beaujean sand is more subrounded to subangular.

Pre-packed sand aggregates (40 mm diameter, 100 mm length) were prepared by pouring a fixed amount of sand (~ 250 g in mass) into a Viton sample jacket used for the experiments (see Figure 1), followed by 10 mins on a shaking plate with a small weight applied to the sand pack. Starting porosity was calculated using the exact sample dimensions and sample mass, assuming a density of quartz of 2.66 g/cm³. This preparation method resulted in a well-controlled starting porosity within the range 36.0-36.3% for the Ottawa grain-size fraction of $300-400~\mu m$ (Table 1). Note that sample OS-06 had a slightly lower initial porosity of 34.6%, which could affect its mechanical behaviour Brzesowsky et al. [2014a].

2.2. Experimental apparatus and procedure

The deformation experiments were performed in a conventional triaxial apparatus at the Rock and Ice Physics Laboratory of University College London [see description in Eccles et al., 2005; Brantut et al., 2014. The machine consists of an oil-filled pressure vessel, with servo-controlled application of axial load and pore pressure, and manual application of confining pressure. After filling of the vessel, confining pressure on the sample was ~ 5 MPa. Confining and pore pressures were then increased in 1 MPa steps, such that the pore pressure never exceeded the confining pressure, up to 10 MPa confining pressure and 5 MPa pore pressure. Subsequently only confining pressure was increased in 5 MPa steps to the target conditions. The sample was then left to equilibrate for about 2 hours. Axial shortening was measured using the average of two linear variable differential transformers (LVDTs), located outside of the pressure vessel, and used to calculate axial strain. All tests were performed under drained, constant pore pressure conditions. After $\sim 5 \%$ axial strain, strain rate-stepping stages were performed by down-stepping the strain rate by one order of magnitude ($\dot{\epsilon} = 10^{-6} \text{ s}^{-1}$), followed by up-stepping the strain rate by one order of magnitude compared to the initial strain rate ($\dot{\epsilon} = 10^{-4} \text{ s}^{-1}$). This sequence was repeated.

For all experiments, total axial strains of $\sim 7.5\%$ were achieved. Pore volume changes were determined by measuring the change in pore-fluid volume during deformation, as determined from the position of the actuator inside the servo-controlled pore pressure intensifier. The triaxial deformation experiments were terminated by halting the loading ram. Subsequently, the sample was unloaded at a strain rate of $10^{-5}~{\rm s}^{-1}$, followed by reducing the confining and pore pressures to room pressure, making sure that at all times $P_{\rm c} > P_{\rm p}$.

2.3. Wave velocity and acoustic emission measurements

During deformation, acoustic emissions (AEs) and the evolution of P-wave velocities with increasing deformation were monitored. The sample jacket is equipped with 16 piezoelectric transducers positioned around, and directly touching, the sample, and connected to 40 dB high-frequency preamplifiers and a 50 MHz digital recording system [e.g., *Brantut et al.*, 2014] (Figure 1). P-wave velocities were determined by regularly and sequentially sending

Table 2. Conventional triaxial compression experiments performed on fluid-saturated aggregates of Ottawa and
Beaujean sand, with different grain angularities and grain-size distributions (GSD), at 20° C and $P_{\rm p}=5$ MPa, plus
unloading Young's Modulus obtained in the tests. Additionally, mean grain size values are given when measured.

Sample	Sand type	d	ϕ_0	ϕ_{i}	E_{u}	d_{50}				
		$[\mu\mathrm{m}]$	[%]	[%]	[GPa]	$[\mu \mathrm{m}]$				
Effect of grain angularity: decane-saturated sand aggregates										
OS starting material 308-309										
BS starting material					294-298					
OS-19	Ottawa	180 – 300	37.4	30.9	8.0	280-283				
	(well-rounded)									
BS-01	Beaujean	180 - 300	38.6	31.6	8.0	198 - 227				
	(subrounded/-angular)									
	, , , , , , , , , , , , , , , , , , , ,									
Effect of grain-size distribu	tion: water-saturated sar	nd aggregates								
starting material (narrow)						308-309				
starting material (broad)						403-450				
OS-07	Ottawa	300 – 400	36.3	33.8	6.7					
		(narrow GSD)								
OS-14	Ottawa	180-500	34.9	32.5	5.5	308-335				
		(broad GSD)								
		, ,								

Symbols: d is grain-size, ϕ_0 is sample porosity prior to set-up, ϕ_i is sample porosity after application of P_c and P_p , P_c^{eff} represents effective confining pressure, E_u is the Young's modulus obtained from a linear fit to the linear (elastic) part of the stress-strain curve during unloading, d_{50} is the mean grain size obtained from particle size analysis (two measurements).

a high frequency (1 MHz) and high voltage (250 V) pulse on each transducer while recording transmitted waves on the remaining sensors. During the time intervals between active wave velocity measurements, we monitored AE activity and recorded incoming signals when a threshold voltage (typically about 100 mV) was detected over five sensors. In addition to wave velocity and triggered AE waveform data, we also monitored the total of AE hits on each individual sensor, with time bins of 5 s.

2.4. Data processing

External axial load, piston displacement, confining pressure, sample temperature, pore-fluid pressure and pore-fluid volume change signals were logged at time intervals of 1 s. The raw data were processed to give differential stress $(\sigma_1 - \sigma_3)$, axial strain (ϵ) , axial strain rate $(\dot{\epsilon})$ and porosity change $(\Delta\phi)$ data versus time. The displacement data were corrected for apparatus distortion using predetermined stiffness calibrations. The change in pore volume was used to calculate porosity change, assuming rigid solid particles.

Using a cross-correlation technique, precise P-wave arrival times were determined [for details, see Brantut et al., 2014]. Assuming that volumetric deformation was uniformly distributed along the sample axis, the sensor positions were corrected from the axial and radial deformation achieved in each experiment. The local positions of the transducers around the sample allow for P-wave speeds to be calculated in four directions relative to the sample axis: 90.0°, 58.0°, 38.7°, and 28.1°, respectively. AEs were located using the arrival times of waveforms to the 16 transducers, assuming a homogenised transversely isotropic P-wave speed model that uses P-wave speeds interpolated in time from the measured values. Due to severe clipping of the wave form, it was not possible to assess the cumulative AE energy.

2.5. Microstructural methods

After the experiments, the jacketed samples were extracted from the pressure vessel. Due to the friable nature of the sand aggregate, intact extraction of the sample was only possible by freezing the sample at $-20^{\circ}\mathrm{C}$ for ~ 12 hrs, while inside the jacket. The frozen sand aggregate was subsequently removed from the sample sleeve and emplaced in a fluorinated ethylene propylene (FEP) jacket of the same dimensions as the frozen sample. The samples were allowed to defrost and dry in an oven at $80^{\circ}\mathrm{C}$ for several weeks, prior to impregnation with LR White epoxy resin. Subsequently, thin sections were made from the material, roughly

at the centre of the aggregate, parallel to the compression axis. Though the samples were impregnated they remained friable and during thin section preparation some grains were plucked from the section.

Using an optical microscope, micrographs of approximately 10 by 10 mm were made. The freezing procedure did not seem to have affected the microstructure, as many grains and their associated grain fragments appear to be undisturbed. From these micrographs, crack maps were constructed (see Figure 2) by manually tracing all throughgoing or partially through-going cracks visible within the sand grains. Small cracks along grain edges, caused by chipping, were not mapped. The resulting crack maps were overlain by a grid of 100 $\mu{\rm m}$ spacing. Within a square of four cells, the number of cracks was counted and attributed to the centre of the square. Each new square was shifted one cell (quadrant) with respect to the previous one (analogous to a Kalsbeek counting net [Kalsbeek, 1963]). The resulting columns and rows of numbers were converted to a crack density contour map.

Due to the low freezing temperature of decane $(-30^{\circ}\mathrm{C})$ it was not possible to freeze the decane-saturated samples, following the same method. Therefore, no attempts were made to preserve the microstructures of the decane-filled samples, and the sand was simply allowed to dry after removal from the sample sleeve. Loose sand grains from such experiments were examined using Scanning Electron Microscopy (SEM), by mounting the powder on double-sided carbon tape on stubs.

Undeformed starting material and selected, post-deformation sand samples were analysed using a Malvern laser particle sizer, to determine changes in the average grain-size (d_{50}) and grain-size distribution of each sample due to deformation. Prior to sampling, each material was well mixed and a 0.5 g sample was taken for particle analysis. Agglomeration effects can appear to lead to Malvern particle size analysis overestimating the mean grain-size by up to ~ 1.5 times [Hangx et al., 2010], as well as to post-experiment distributions with grain-sizes that are larger than those observed in the starting material. However, compared to grain-size analysis via optical methods, this method provides the opportunity to sample a larger number of grains, plus circumvents any stereological uncertainty obtained from 2D thin section analysis.

3. Results

3.1. Fluid-saturated Ottawa sand experiments: effect of chemical environment

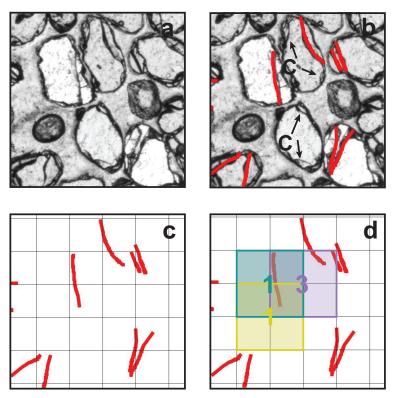


Figure 2. Procedure for processing of the microstructures to produce crack density contour maps. a) Optical micrograph of a portion of a typical sand microstructure. b) Cracks are marked in red, while chipping-related cracks (C) are not taken into account. c) The crack map is overlain by a grid of $100 \ \mu m$ spacing. d) Within squares of four grid cells, the number of cracks is counted and attributed to the centre of the square. The resulting columns and rows are converted to a crack density contour map.

3.1.1. Mechanical data

In this paper, we adopt the convention that compressive stresses, compressive axial strains and porosity change are measured positive. The principal compressive stresses are denoted as σ_i , with $\sigma_1 > \sigma_2 = \sigma_3 = P_c$. The effective principal stresses are denoted $\sigma_i^{\rm eff}$, and are defined as $\sigma_i^{\rm eff} = \sigma_i - P_{\rm p}$, where $P_{\rm p}$ is pore pressure. Porosity changes during each experiment are measured from the point that pore-fluid pressure is applied ($P_{\rm c}^{\rm eff} = 5$ MPa). Note that no porosity change can be measured for the increase in effective confining pressure to 5 MPa, as up to that point the sample is dry. Extrapolation of porosity- $P_{\rm c}^{\rm eff}$ data obtained during the hydrostatic stage (shown in Figure A1, see Appendix A) predict that neglecting the porosity change that will occur during the dry stage will lead to an overestimation of the actual sample porosity, or an underestimation of the porosity change, by $\sim 0.6\%$, which is negligible compared to the total porosity change measured during the experiments ($\Delta \phi$ up to 10%).

Differential stress-axial strain and mean stress-porosity change curves for hydrostatic and triaxial deformation experiments at effective pressures $P_c^{\rm eff}$ of 5 to 40 MPa under decane- and water-saturated conditions (pore-fluid pressure, $P_{\rm p}=5$ MPa) are presented in Figure 3. Overall, all experiments show an initial stage of quasi-linear stress-strain behaviour (Figure 3a-b). Only at very low effective confining pressure ($P_c^{\rm eff}=5$ MPa; cf. OS-15, Figure 3d) dilation is observed, while at higher confinement the Ottawa sand aggregates compact. After $\sim 1\%$ strain, the stress-strain curves become non-linear and the samples transition from strain-neutral towards strain-hardening behaviour with increasing effective confining pressure (cf. Figure 3b). More porosity change is achieved at higher confinement, reaching near-uniaxial compaction (i.e. $\Delta \phi \simeq \epsilon$) at effective confining pressures of 40 MPa. Note that the slightly lower porosity

sample OS–06 showed also slightly lower differential stresses at the same amount of axial deformation (Figure 3e). Overall, little effect of pore-fluid chemistry (decane vs. water) is observed in terms of aggregate strength and volumetric behaviour.

The hydrostats (cf. OS-04 and OS-17 in Figure 3c and d, respectively) show a slightly sigmoidal shape with an initial concave upward trend between mean stress and porosity change, becoming quasi-linear after approximately 2% porosity change and concave downward again after 5%porosity change. At an effective mean stress of 50 MPa, 5.7% and 4.9% porosity change is achieved for the decane- and water-saturated hydrostatic experiment, respectively. The decane hydrostat follows the volumetric behaviour seen in the hydrostatic stages of the decane-saturated triaxial deformation experiments. While the water hydrostat follows the hydrostatic stages of the water-saturated triaxial experiments up to effective confining pressures of 25 MPa, it underestimates compaction at higher confinement (Figure 3b). This may be related to sample preparation or to the pressurisation procedure employed during the hydrostatic experiment. It should be noted that within the pressure range tested, the hydrostatic experiments lack any significant inflection point, generally associated with the departure from elastic behaviour and the onset of inelastic compaction (P^* $[Zhang\ et\ al.,\ 1990]).$

In our triaxial deformation experiments, the non-linearity of the stress-strain curves during loading makes the determination of a specific onset of inelastic behaviour (commonly denoted C^* in sandstones [Wong et al., 1997]) very elusive and was therefore not done. Upon unloading, the stress-strain data is near-linear and shows that 80-95% of the total axial strain is permanent deformation, at all stages of the experiment (Figure 3e). We use the linear portions of

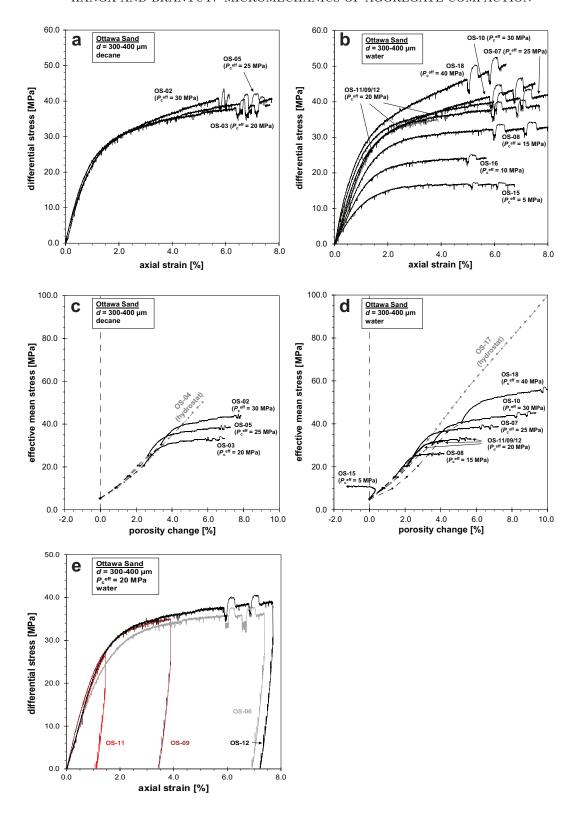


Figure 3. a-b) Differential stress versus axial strain and c-d) effective mean stress versus porosity change curves for triaxial experiments performed on decane-saturated and water-saturated Ottawa sand aggregates, at room temperature and fluid pressures of 5 MPa. Solid lines indicate triaxial deformation stages. Dotted lines show hydrostatic experiments or experiment stages. Strain rate-stepping stages resulted in up and down steps in differential (a-b) and mean stress (c-d) at the end of each experiment. e) Differential stress versus axial strain curves for selected experiments, illustrating the experimental reproducibility, the impact of porosity on mechanical behaviour (cf. OS-06 and OS-12) and typical unloading behaviour.

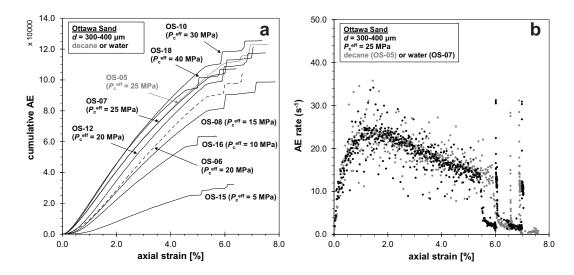


Figure 4. Acoustical data obtained for the decane- and water-saturated experiments ($d=300-400~\mu m$). a) Cumulative AE and b) AE rate versus axial strain. Note that the strain rate-stepping stages were initiated after $\sim 5\%$ axial strain.

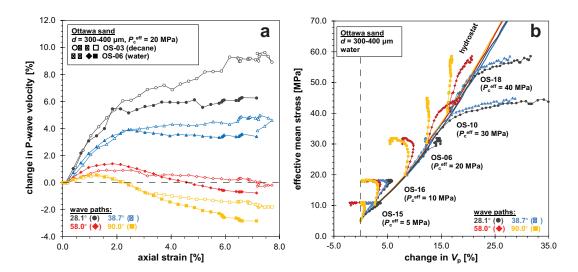


Figure 5. a) Changes in P-wave velocity along the four wave path directions versus axial strain for experiments OS-03 (decane; triangles) and OS-06 (water; circles) at $P_c^{eff}=20$ MPa. Strain rate-stepping was initiated at $\sim 5\%$ axial strain. b) Effective mean stress versus change in P-wave velocity at different effective confining pressures for water-saturated Ottawa sand aggregates. The triaxial deformation experiments are superimposed onto the hydrostat. Note that strain rate-stepping stages were initiated near the end of each experiment.

 $et\ al.,\ 2000b]$ - see Figure 3e; full data set available from the UK National Geoscience Data Centre). As reported in Table 1, Young's Modulus ranges from 3.6 to 8.1 GPa in the effective confining pressure range 5 to 40 MPa, increasing with $P_{\rm c}^{\rm eff}$. At constant confining pressure, $E_{\rm u}$ remains roughly constant throughout the experiment (cf. OS-11, -09, -06 and -12; Table 1 and Figure 3e). Pore-fluid does not affect the static elastic stiffness.

3.1.2. Acoustical data

The evolution of the cumulative acoustic emission count and AE rates as a function of axial strain are shown in Figure 4a and b, respectively. For all experiments, cumulative AEs increase with increasing strain, showing concave upward behaviour up to approximately 1 % axial strain followed by concave downward behaviour at larger deformation. Furthermore, more AEs are counted with increasing confining pressure. Note that for experiment OS-06, which had a

slightly lower starting porosity, fewer cumulative AEs were counted during compaction.

Acoustic emission rate, i.e. the time derivative of the cumulative AEs, shows a rapid initial increase in rate with axial strain, until it reaches a peak AE rate at an axial strain of $\sim 1\%$, after which the rate slows down again. With increasing confining pressure, the peak AE rate increases in magnitude ($\sim 6.5 s^{-1}$ at 5 MPa $P_{\rm c}^{\rm eff}$ to $\sim 25 s^{-1}$ at $P_{\rm c}^{\rm eff}$ by APa) and occurs at lower axial strain with increasing confining pressure ($\sim 1.8\%$ at 5 MPa $P_{\rm c}^{\rm eff}$ to $\sim 1\%$ at $P_{\rm c}^{\rm eff}$ >25 MPa). pore-fluid composition does not affect the cumulative number of AEs or the AE rate (cf. Figure 4b).

Absolute P-wave velocities during the decane- and watersaturated experiments, measured along the four wave paths, range from 1800 to 2250 m/s prior to deformation, increasing with increasing confinement, i.e. with decreasing porosity at the start of the triaxial deformation stage. The velocities measured through the lower half of the samples were consistently higher, most likely due to a slightly denser packing of the aggregate resulting from the preparation method. Changes in P-wave velocity with respect to the pre-deformation value are shown in Figure 5a as a function of axial strain for decane- and water saturated sand (P_c^{eff} = 20 MPa). For all experiments, P-wave velocity increases rapidly for the initial 1-2% strain. At low confining pressure $(P_{\rm c}^{\rm eff}$ <20 MPa), P-wave velocity decreases along all four wave paths, and eventually becomes less than the starting value for the (sub-)horizontal wave paths (58.0° and 90.0°). By contrast, at higher confining pressure, after the initial rapid increase, P-wave velocity stays constant or increases at a slower rate along the steeper wave paths (28.1° and 38.7° , and 58.0° at the highest P_c^{eff}). Upon unloading Pwave velocity remains nearly constant for all experiments, suggesting that most of the measured deformation is permanent in line with the mechanical data (cf. Section 3.1.1). The evolution of $V_{\rm p}$ is not significantly affected by the porefluid type (Figure 5a).

For water-saturated aggregates, the evolution of P-wave velocity with effective mean stress, superimposed onto the hydrostat, is shown in Figure 5b. For the selected experiments, changes in P-wave velocity are shown along all four wave paths, to illustrate the development of azimuthal anisotropy of damage as a function of confining pressure. For all triaxial deformation experiments, the change in Pwave velocity initially increases linearly with effective mean stress and shows similar or slightly lower values compared to those measured under hydrostatic conditions. The initial rate of P-wave velocity increase is the lowest for the horizontal wave path, increasing as wave path angle rotates to sub-vertical, though no dependence on confinement is seen. As effective mean stress increases P-wave velocity decreases with increasing mean stress along all wave paths for effective confining pressures up to 30 MPa. By contrast, at high confinement ($P_c^{\rm eff} \geq$ 30 MPa) P-wave velocities increase rapidly with increasing stress, and thus strain, except along the horizontal wave path. The increase in wave velocity is most rapid in the sub-vertical direction, i.e. sub-parallel to the compression direction. All experiments display an increase in wave-anisotropy (i.e. $V_p^{28.1}^{\circ}/V_p^{90.0}^{\circ}$) due to deformation, which becomes stronger with higher confinement (see Figure 5b). At low $P_{\rm c}^{\rm eff}$, anisotropy is slightly above one, at 1.05 to 1.1. As confining pressure increases to 30-40 MPa, anisotropy increases to 1.2 - 1.3.

3.1.3. Microstructural analysis and located AE events

Undeformed Ottawa sand ($d = 300 - 400 \mu m$) consists of well-rounded grains with relative smooth surfaces (Figure 6a). After deformation, the grains have a more ragged appearance, most likely due to grain edge chipping such as seen in Figure 6b. Chipping is evident at low confinement (Figure 6b), where little grain crushing occurs. At higher confinement, fracturing and crushing becomes more dominant, shattering grains into angular fragments and small angular flakes (Figure 6c and d). These microstructural observations are confirmed by grain-size analyses (Figure 6e), which show the increased formation of finer particulates with increasing confinement. Very little grain-size reduction is observed in hydrostatic experiment OS-04 (decane), though compaction is following the same trend as the hydrostatic phases of the triaxial deformation experiments. No grainsize analysis could be performed on the water-saturated hydrostatic experiment (OS-17).

AE events indicate that localisation at low confining pressure ($P_{\rm c}^{\rm eff} \leq 5$ MPa) occurs along a shear plane. Though not evident from the microstructure, the volumetric data suggests that this plane is likely a dilation band. In line with this, pervasive grain failure is absent in the microstructure (Figure 6e), while grain edge chipping is prevalent, facilitating the required grain rotation and rearrangement needed

for strain localisation. At higher confining pressures ($P_{\rm c}^{\rm eff} \geq 10$ MPa) deformation appears to be more diffuse, with no clear indication of localisation. Furthermore, the degree of grain fracturing and crushing increases with axial strain from 5.3 cracks/mm² at 1.5% strain (OS-09) to 5.6 cracks/mm² at 4.5% strain (OS-11) and 11.4 cracks/mm² at 7.5% strain (OS-06; $P_{\rm c}^{\rm eff} = 20$ MPa), as well as with confining pressure (e.g. from 5.0 cracks/mm² at 5 MPa to 16.9 cracks/mm² at 40 MPa $P_{\rm c}^{\rm eff}$). The microstructures do not show clear indications for strain localisation (cf. Figure 6f-h).

3.2. Effect of strain rate

After $\sim 5\%$ axial strain, strain rate-stepping stages were performed by stepping the strain rate by one order of magnitude down and up. During strain rate down-steps the differential stress, and hence also the mean stress, measured across the sample decreased. Similarly for strain up-steps the differential and mean stress increased (cf. Figure 3). The magnitude of these stress changes appears to increase with increasing confining pressure.

The strain rate steps are also clearly reflected in the cumulative acoustic emission counts and AE rates. Decreasing the strain rate significantly reduces the AEs produced by the sample, down to a rate of $<3~s^{-1}$. Conversely, increasing the strain rate resulted in a rapid increase in the number of AEs, to a rate of $\sim 30~s^{-1}$ (Figure 4). However, P-wave velocities are not significantly impacted by the strain rate-stepping sequence, which is in accordance with the observation that strain rate-stepping does not affect sample volume changes.

3.3. Experiments varying grain angularity and grainsize distribution

In addition to the experiments on well-rounded Ottawa sand with a narrow grain-size, we also performed experiments on sub-rounded/sub-angular Beaujean sand ($d=240\pm60~\mu\mathrm{m}$) and Ottawa sand with a broader grain-size distribution ($d=340\pm160~\mu\mathrm{m}$). These experiments aimed at studying the effect of grain angularity (OS-19 and BS-01, $P_{\rm c}^{\rm eff}=45~\mathrm{MPa}$, decane) and grain-size distribution (OS-07 and OS-14, $P_{\rm c}^{\rm eff}=25~\mathrm{MPa}$, water) on compaction behaviour (Table 2).

Differential stress-axial strain and porosity change-axial strain curves are presented in Figure 7. All four aggregates show behaviour similar to the Ottawa sand described in Section 3.1 with quasi-linear loading and near-uniaxial compaction behaviour. All four experiments show significant strain-hardening, which is more pronounced at high confining pressure. In terms of the effect of grain angularity, the mechanical data suggest that there is no significant difference in behaviour between the well-rounded Ottawa sand and the more subrounded/-angular Beaujean sand. It is evident from Malvern grain-size analysis that the Beaujean sand underwent significant grain crushing, with d_{50} values reducing by $\sim 28\%$ from 296 μ m to 212 μ m (compared to only $\sim 8\%$ reduction for Ottawa sand, from 308 μm to $282 \mu m$). Similarly, varying grain-size distribution, i.e. narrow vs. broad, does not affect the mechanical behaviour of the Ottawa sand aggregates. Microstructural observations show that the coarser grains within the aggregate remain largely intact during deformation, while the finer grains have been crushed (Figure 8). Compared to the narrow GSD Ottawa sand deformed at 25 MPa (OS-05), the average grainsize has been significantly reduced, as a result of the formation of fines (d_{50} is 321 μ m and 352 μ m, respectively; see Figure 6e). No indications for strain localisation are observed.

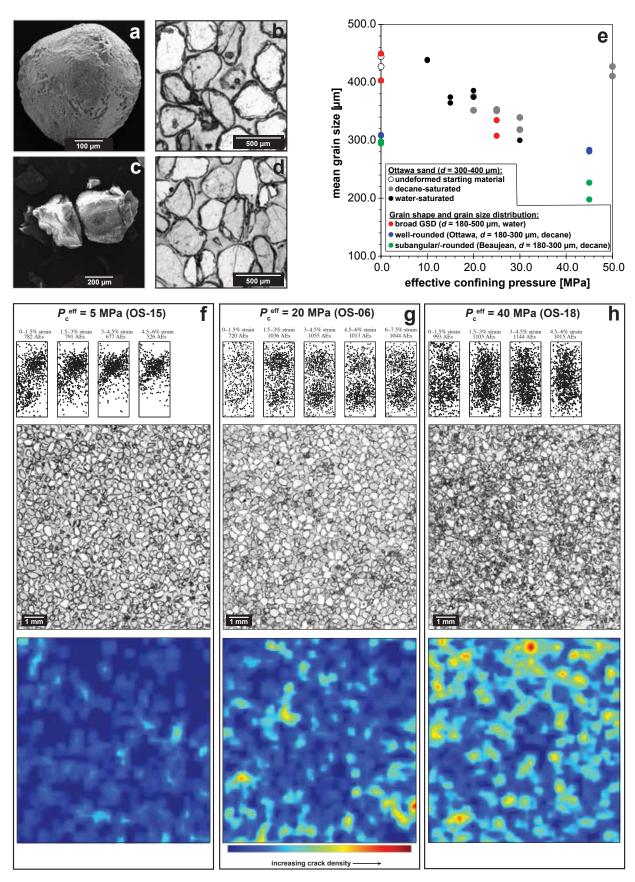


Figure 6. Scanning Electron and optical micrographs of Ottawa sand grains, which are a) undeformed, b) compacted at low confining pressure (OS-15; $P_c^{eff}=5$ MPa) and c-d) compacted at high confining pressure (OS-12 (c) and OS-06 (d); $P_c^{eff}=20$ MPa). e) Mean grain-size (d_{50}) versus confining pressure, showing a reduction in mean grain-size with increasing confinement, indicative of the chipping and fracturing seen in (b-d). Located AE events for the whole sample (40 by 100 mm), optical micrographs of sections taken at the centre of the sample (10 by 10 mm) and corresponding crack density contour maps obtained from water-saturated Ottawa sand aggregates, deformed at e) 5 MPa (OS-15), f) 20 MPa (OS-06) and g) 40 MPa (OS-18) effective confining pressure. Note that the micrographs are not taken from the same slice through the sample as the AE locations.

Goodwin, 2003] and in the laboratory [Alikarami et al., 2015]. Structures seen in the Aztec sandstone are inferred to suggest that compaction bands may also form in unconsolidated sands [Sternlof et al., 2005], though to date no such laboratory observations have been made. By contrast, discrete compaction bands have been observed in sandstones in both nature [Mollema and Antonellini, 1996; Sternlof et al., 2006] and in the laboratory [see Wong and Baud, 2012, for an overview. While there may be differences in their characteristics, both natural and lab-induced compaction bands indeed show locally intense microcracking, grain comminution and porosity reduction. However, not all sandstones are prone to compaction band formation. Porosity and rock homogeneity (mineralogy, grain-size distribution) play a significant role in controlling this type of strain localisation [Cheung et al., 2012; Mollema and Antonellini, 1996], though their interplay is not yet well understood. Our results suggest that there is no evidence for compaction localisation in unconsolidated sand deformed in the compactant regime.

4.2. Effect of water, grain angularity and grain-size distribution on behaviour

In the presence of chemically active fluids like water, sandstones generally tend be significantly weaker [Baud et al., 2000a; Duda and Renner, 2012; Baud et al., 2015; Tembe et al., 2008]. Such physico-chemical weakening of sandstones is attributed to the presence of altered feldspars and clay minerals [Baud et al., 2000a], which reduce the proportion of strong, quartz-cemented grain-to-grain contacts. Furthermore, altered feldspars may promote crack nucleation and growth, while clays may promote slip through a reduction in frictional resistance when wet [Reviron et al., 2009; Baud et al., 2000a]. By contrast, very pure sandstones, like Fontainebleau and Bentheim sandstone, show no significant water-weakening [Reviron et al., 2009; Tembe et al., 2008]. Similarly, our experiments on decane- and watersaturated quartz sand ($d = 300 - 400 \mu m$) show no clear effect of a chemically active fluid on stress-strain and strain- V_p behaviour, and grain-size reduction within the range of strain rates investigated (cf. Figures 3, 5 and 6). These observations are in accordance with the behaviour seen during time-independent uniaxial compaction of pure quartz sand, which is not significantly affected by chemical environment [Brzesowsky et al., 2014a]. This underpins the suggestion that the presence of other mineralogical phases is needed to promote water-weakening. Recent work on impure sandstone inferred that intergranular slip at clay-coated grain boundaries may play an important role in controlling deformation, especially at stress conditions below the yield point [Pijnenburg et al., 2018, 2019].

Grain angularity is known to lead to sharper contact points and hence higher contact stresses, leading to more grain-breakage [Chuhan et al., 2003; Zhang et al., 1990], i.e. the potential to create an instability. At the same time, in sandstones with a broad grain-size distribution, large grains tend to inhibit the propagation of instabilities, thereby hindering the formation of compaction bands [Cheung et al., 2012]. We performed two experiments aimed at investigating the effect of grain angularity (Ottawa sand OS-19 vs. Beaujean sand BS-01, $P_c^{\text{eff}} = 45$ MPa, decane). Previous triaxial experiments on Ottawa and Hostun sand under lower confining conditions ($P_c = 0.1 - 7 \text{ MPa}$; see Alikarami et al. [2015]) showed comparable stress-strain behaviour for the two sands, though the angular Hostun sand displayed more pronounced dilatation and grain-breakage. While our experiments are performed at higher confining pressure conditions, we also observe very similar stress-strain behaviour between the Ottawa and Beaujean sand. Furthermore, the more angular Beaujean sand shows significant grain failure

and a smaller average grain-size compared to the rounded Ottawa sand. We speculate that the more angular Beaujean sand has a higher tendency to strong interlocking, which would require the 'chipping off' of larger fragments to unlock the aggregate and promote grain rearrangement, compared to the smoother, well-rounded Ottawa sand grains.

It should also be noted that overall finer-grained Ottawa sand $(d = 180 - 300 \mu m)$ showed less grain-size reduction

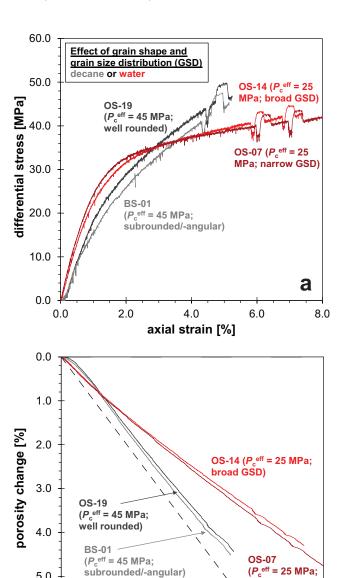


Figure 7. a) Differential stress versus axial strain and b) porosity change versus axial strain curves for triaxial experiments performed to test the effect of grain angularity (grey curves) and grain-size distribution (GSD red curves) on mechanical behaviour. Note that experiments OS-19 and BS-01 were performed on decanesaturated aggregates, while OS-07 and OS-14 were performed on water-saturated aggregates, at room temperature and fluid pressures of 5 MPa. Strain rate-stepping stages resulted in up and down steps in differential stress at the end of each experiment.

4.0

axial strain [%]

narrow GSD)

6.0

b

8.0

5.0

6.0

0.0

2.0

than coarser-grained Ottawa sand ($d = 300 - 400 \mu m$), even when deformed at higher effective confining pressure. This is inferred to be related to the tendency for larger grains to have a lower yield stress, making them more prone to grain failure [Chuhan et al., 2003; Zhang et al., 1990]. Regarding grain-size distribution, stress-strain behaviour is very comparable between the two end-members (OS-07 vs. OS-14, $P_c^{\text{eff}} = 25 \text{ MPa}$, water). Looking at the microstructures, the broad GSD experiment displays shielding of the larger grains by the smaller grains, leading to less breakage of the coarser grains, supporting the observations made for Boise sandstone [Cheung et al., 2012]. Overall, our experiments on Ottawa sand show that in terms of chemical environment, grain angularity and grain-size distribution, sands tend to show qualitatively similar behaviour compared to sandstones.

4.3. Hardening envelopes for sand in PQ-space

For sandstones, the yield point is typically defined as the stress conditions at which the material is no longer behaving poro-elastically [Wong and Baud, 2012]. This point is generally accompanied with a marked increase in acoustic emission activity [e.g. Tembe et al., 2007; Fortin et al., 2006]. However, in the case of unconsolidated sand, large permanent deformation is seen during loading from the onset and elastic behaviour is only observed upon unloading (Figure 3e). As such, no clear yield point can be pinpointed on the basis of the mechanical data, the acoustic emission activity or the P-wave velocities (cf. Figures 3b, 4a and 5b). Therefore, to generate a meaningful description of the deformation behaviour of this material, we propose it is more appropriate to represent the stress-strain data in terms of expanding hardening envelopes, each representing the stress supported at a specific porosity value. Since the sand aggregates continuously compact, this means that the hardening envelopes gradually grow as aggregate porosity decreases [Karner et al., 2005]. A single loading path through these hardening envelopes then gives the stressstrain behaviour of the material. As the mechanical behaviour of Ottawa sand is reproducible and its material properties are near-constant throughout each experiment,

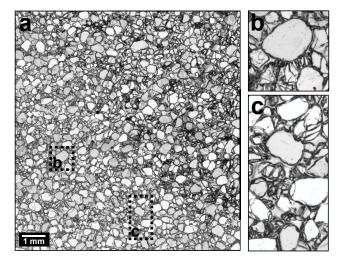


Figure 8. a) Optical micrograph of Ottawa sand aggregates with a broad grain-size distribution (d=180-500 μ m), deformed at 25 MPa (OS-14), with b) and c) insets showing large surviving grains surrounded by crushed particles, suggesting shielding of the coarser grains by the finer grains present within the grain-size fraction.

delineating the mechanical behaviour in terms of hardening envelopes is warranted (cf. compare OS-09, -11 and -12, see Table 1 and Figure 3e). For both the decane- and water-saturated aggregates, these hardening envelopes are drawn in differential stress, $Q=(\sigma_1-\sigma_3)$, versus mean stress, $P=(\sigma_1+2\sigma_3)/3-P_{\rm p}$, space (Figure 9).

As is clear from the hardening envelopes, Ottawa sand shows significant porosity reduction during the hydrostatic stage of the experiment (up to $\sim 5\%$ at 40 MPa $P_{\rm c}^{\rm eff}$, cf. Figure 3), which has also been observed for St. Peter sand [Karner et al., 2003]. However, the most striking feature of the contours is that they are near-vertical up to ~ 30 MPa differential stress, suggesting that porosity change is mainly controlled by mean stress, and not significantly by differential stress. Similar behaviour has been observed before in St. Peter sand aggregates [Karner et al., 2005], porous bassanite [Bedford et al., 2018] and even in some highly porous sandstones [Tembe et al., 2007; Pijnenburg et al., 2019].

In essence, the hardening envelope for sand appears to consist of three linear parts: a shear failure line at low P, a narrow mean stress-range during which deformation appears to be pressure-insensitive, followed by a rapid transition to a near-vertical end cap, which increases in size as porosity decreases (see Figure 9). The bifurcation analysis of Issen and Rudnicki [2000] predicts that localised compaction bands form when the sum of the dilatancy factor β (the ratio of inelastic volumetric over shear strain increments) and friction parameter μ is $(\beta + \mu) < -\sqrt{3}$. Analysis of our data to tease out these constitutive parameters see Table 3) suggests that at least in the highest confining pressure experiments conditions were favourable to promote compaction band formation. However, this prediction is at odds with our observations. Such a discrepancy between the prediction from bifurcation theory and experimental observations is not uncommon for sandstones, and is likely due to the inadequacy of the constitutive description [Baud et al., 2006]. Furthermore, it should be noted that we do not have access to the yield envelope sensu stricto, but to hardening envelopes, which might develop a different slope from the conventional yield envelope. This also inhibits a meaningful comparison between sand and sandstones. Given this discrepancy in behaviour between sand and sandstone, the question now is: what are the underlying microphysical processes controlling this behaviour?

4.4. Mechanisms driving compaction and potential for strain localisation in sands

In our lowest confinement experiment ($P_c^{\rm eff}=5$ MPa), we observed a dilating shear band, based on AE locations (cf. Figure 6f), similar to what is observed in Ottawa sand experiments under similar conditions using X-ray tomography imaging [Alikarami et al., 2015]. However, no strain localisation was observed at higher confinement. If strain localisation had occurred, this would have been evident in the form of stress drops in the stress-strain behaviour (Figure 3; Baud et al. [2004]), or visible in the AE locations (Figure 6g-h; Fortin et al. [2006]; Olsson and Holcomb [2000]), or crack density variations within the samples (crack density contour maps in Figure 6g-h; Baud et al. [2004]; Fortin et al. [2006]). Instead, under these conditions cataclastic flow appeared to control deformation, as also suggested by the near-vertical shape of our hardening envelopes (see Section 4.3).

As summarised by Rudnicki [2007], compaction bands grow due to high stress concentrations at their tips. In other words, the failure of a grain should lead to the grain no longer supporting any stress and the subsequent redistribution of the stress to the surrounding grains, i.e., it needs to lead to strain-weakening/-softening. Throughout all experiments, Ottawa sand shows near-strain-neutral to

Table 3.	Constitutive	parameters	obtained for	conventional	triaxial	compression	experiments	performed	on
	ated Ottawa s								

Sample	$P_{\rm c}^{\rm eff}$	$\beta_{33\%}$	$\mu_{33\%}$	$h_{\text{tan,33\%}}$	$\beta_{32\%}$	$\mu_{32\%}$	$h_{\tan,32\%}$	$\beta_{31\%}$	$\mu_{31\%}$	$h_{ an,31\%}$
	[MPa]	[-]	[-]	[GPa]	[-]	[-]	[GPa]	[-]	[-]	[GPa]
$\overline{\text{OS-08}}$	15	0.13	1.83	0.01	0.19	0.67	0.10			
$OS-09^a$	20	0.23	0.86	0.80	0.20	0.31	0.08			
OS-12	20	0.27	0.92	0.87	0.23	0.16	0.09			
OS-07	25	0.39	-0.30	1.19	0.30	-0.82	0.37	0.29	-1.02	0.09
OS-10	30	0.39	-0.30	1.19	0.38	-1.73	1.53	0.33	-1.93	0.47
OS-18	40							0.35	-12.11	3.88

Symbols: $P_{\rm c}^{\rm eff}$ represents effective confining pressure, β , μ and $h_{\rm tan}$ are the constitutive parameters as needed for bifurcation analysis [Rudnicki and Rice, 1975; Issen and Rudnicki, 2000], obtained at sample porosities of 33%, 32% and 31%, respectively. Here β is the dilatancy factor, μ is the internal friction coefficient and $h_{\rm tan}$ is the tangent hardening modulus, obtained from the slope of a differential stress-axial strain plot (see Figure 3b).

^a Sample deformed up to 4% e.

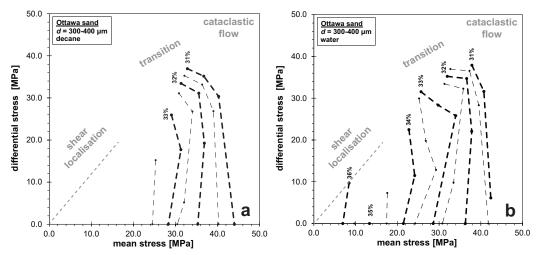


Figure 9. Hardening envelopes for specific aggregate porosity-values plotted in differential stress versus mean stress space for a) decane-saturated and b) water-saturated Ottawa sand ($d = 300 - 400 \mu \text{m}$) aggregates, at room temperature and fluid pressures of 5 MPa. Thick and thin dashed black lines separate porosity changes of 0.5%.

strain-hardening behaviour (Figure 3a and b, Table 1). The progressive hardening of the material is also evidenced by the AE locations, which appear to propagate from the sample edge towards the centre, and the continuously increasing P-wave velocities, suggesting a stronger, denser aggregate is being formed (cf. Figure 5). Therefore, the condition required for compaction band propagation, the creation of an instability, does not appear to occur.

Instead, we speculate that our data suggest that even at high confining pressure, grain-breakage, and concomitant grain rotation and rearrangement controls compaction. It is difficult to differentiate between AE events emerging from grain-breakage and grain rotation [Wong et al., 1992], meaning that the acoustic emission locations could be derived from either (see Figure 6f-h), as also suggested by Menéndez et al. [1996]. However, significantly fewer AEs are observed at low confinement (Figure 4a), compared to high $P_{\rm c}^{\rm eff}$ experiments. Microstructural evidence suggests that grain chipping (Figure 6b) is prevalent at these low confinement conditions, while progressively more grain-breakage occurs as confinement increases (see crack density contour maps in Figure 6f-h).

This change in microstructural behaviour with confining pressure is also suggested from the strain rate stepping data. As shown in Figures 10a and b, upon a change in axial strain rate, there is a change in differential stress supported by the sample. Overall, the differential stress change increases in magnitude when confining pressure increases, i.e. when ag-

gregate porosity goes down. Following Brantut et al. [2014], given that the strain rate is related to the differential stress as $\sigma^* \ln(\dot{\epsilon}/\dot{\epsilon_0}) \approx \Delta Q$, where σ^* is the activation stress, shows that at low $P_{\rm c}^{\rm eff}$ (high porosity), the activation stress is very low (< 0.5 MPa), while at higher confinement (lower porosity) the value is high (> 1 MPa). High activation stresses are generally associated with cataclastic behaviour in sandstones, e.g. $\sigma^* = 4 - 5$ MPa for compaction band formation in Bleurswiller sandstone [Heap et al., 2015]. Similarly, looking at the cumulative AE data, we can see that fewer AEs are produced per unit axial strain at low confining pressure. The rate of AEs with unit axial strain (AE efficiency, see Wong et al. [1992]) increases with confinement (Figure 10c). It has been suggested that this is generally associated with a transition from compaction by predominantly grain rotation/rearrangement, associated with minor grain fracturing (chipping), to predominantly grain failure plus grain rotation [Wong et al., 1992], in line with the observed strain rate dependency.

The above hypothesis for the grain-scale behaviour is supported by the wave velocity data, which do not show significant changes in horizontal V_p , indicative of the opening of axial cracks. Furthermore, the decrease in P-wave velocities as failure approaches, such as observed for sandstones reflecting enhanced microcracking and pore collapse [Fortin et al., 2006], is also not observed. Though our samples experience significant grain failure, as evidenced by the grain-size and thin section analyses, the P-wave velocity appears not

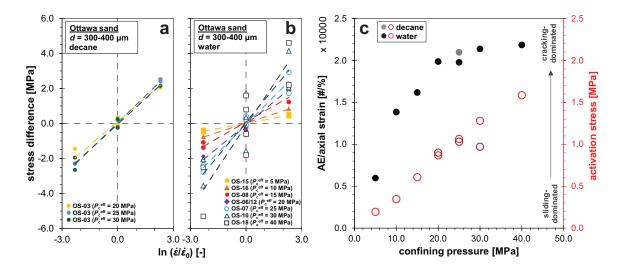


Figure 10. Differential stress difference measured upon a change in axial strain rate versus normalised strain rate for a) decane-saturated and b) water-saturated aggregates. c) Cumulative number of AEs per unit axial strain and activation energy versus effective confining pressure for decane- and water-saturated aggregates.

to be controlled by the formation of cracks. Instead, it is inferred that the data reflect that the acoustic behaviour of Ottawa sand is associated with the contact area between grains [Digby, 1981; Walton, 1987].

Overall, we speculate that for our sand aggregates grain failure is easily accommodated by the rearrangement of surrounding grains, thereby preventing stress concentrations to arise within the aggregate. As the aggregate is subjected to higher confining pressures, it achieves a lower porosity and a more locked microstructure. Furthermore, higher confinement promotes more lateral constraint. So, while grain chipping is sufficient to promote rearrangement at low P_c^{eff} , grain crushing becomes more important at higher P_c^{eff} , as the grains experience less freedom to move around, as reflected in our microstructural, AE and strain rate data. However, even at high confinement, grain rearrangement is still possible meaning that no stress concentration, and hence no strain localisation, will occur within the aggregate. The potential for compaction band formation therefore seems to be hinging on the degree of cementation at the grain contact, though it is unclear what the minimum degree of cementation should be and more research is needed on this [e.g. Bernabé et al., 1992; Yin and Dvorkin, 1994].

5. Conclusions

To probe the key differences between the mechanical behaviour of unconsolidated sand and cemented porous sandstones, we performed systematic triaxial compression experiments on well-rounded Ottawa quartz sand at room temperature, strain rate of $10^{-6}-10^{-4}\,\mathrm{s}^{-1}$ and effective confining pressures of 5-40 MPa. All samples had a well controlled starting porosity of 36.0-36.3%, a narrow grain-size distribution ($d = 300 - 400 \mu m$) and were either decane (inert fluid) or water (chemically active fluid) saturated. Additionally, a limited number of experiments were performed to assess the effect of grain angularity (Beaujean sand) and a broader grain-size distribution ($d = 180 - 500 \mu \text{m}$) on behaviour. The observed mechanical, acoustical and microstructural behaviour was compared to that seen in highly porous sandstones, in order to elucidate whether strain localisation, in the form of shear and compaction bands, can occur in sands as well. We observed the following:

- 1. Pure quartz Ottawa sand initially shows quasi-linear stress-strain behaviour going to strain-neutral and strain-hardening behaviour with increasing effective confining pressure. Overall, only at very low $P_{\rm c}^{\rm eff}$ (5 MPa) dilatant behaviour is seen, while compacting increases with effective confining pressure. This behaviour is also reflected in the P-wave velocities, with initial V_p -values of 1800 2300 m/s that increase by up to 20% as compacting progresses.
- 2. The presence of an inert or chemically active fluid did not significantly affect the mechanical and acoustical behaviour of Ottawa sand. These observations are in line with what is observed for pure quartz sandstones.
- 3. Grain angularity and grain-size distribution also did not affect the stress-strain behaviour of the sand. More angular Beaujean quartz sand shows more grain crushing, which is expected due to the presence of sharper grain-to-grain contacts, leading to higher contact stresses. For aggregates with a broader grain-size distribution, large grains tend to be shielded by smaller grains, which end up being crushed. Qualitatively this behaviour is also seen in sand-stones, such as the Boise sandstone.
- 4. As our sand aggregates do not show truly elastic and yield behaviour, the mechanical behaviour is better described using hardening envelopes. In PQ-space, these hardening envelopes appear to consist of a linear shear failure line and a rapid transition to a near-vertical end cap. At low confining pressure, we see the formation of a dilation shear band, while at high confinement cataclastic flow dominates compaction behaviour, in line with the behaviour expected from the hardening envelopes. No compaction bands are observed.
- 5. The lack of cementation is inferred to lead to relatively easy grain rotation and rearrangement upon grain chipping and failure. As a result, no instability or strain localisation can occur, which would lead to the formation of compaction bands. It is believed that cementation plays a key role in controlling strain localisation via compaction bands, though it is unclear how exactly and more research is needed.

Appendix A: Sample porosity after hydrostatic phase

Volumetric changes cannot be measured during the initial stage of the hydrostatic phase, up to $P_{\rm c}=5$ MPa, as no pore-fluid is introduced into the sample yet and hence sample porosity cannot be calculated. After the introduction of pore-fluid, the volumetric changes are measured during the subsequent application of hydrostatic pressure, which is used to calculate sample porosity. The dependence of starting porosity on confining pressure is shown in Figure A1.

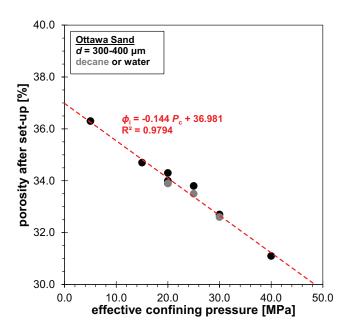


Figure A1. Porosity at the end of the hydrostatic set-up phase as a function of effective confining pressure.

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