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# Can intergranular force transmission be identified in sand?

## First results of spatially-resolved neutron and x-ray diffraction

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**Abstract** This work tackles the challenge of assessment of force distributions in granular media. Spatially resolved neutron and x-ray diffraction are used to measure internal strains of sand grains under load. These approaches are sensitive to the crystallographic strains of the sand grains (quartz crystals) such that each grain acts as a local 3D strain gauge and so, for elastic deformations, a force gauge. First results are presented from recent experiments that provide tantalising indications of the potential of these techniques in the investigation of the mechanics of granular media.

**Keywords** sand; grain-scale mechanics; grain-strains; neutron diffraction; 3D x-ray diffraction

### 1 Introduction

Ioannis Vardoulakis worked on both the mechanics and the physics of granular materials, bridging both the disciplines and communities. As such, in developing continuum theories, Ioannis always had in mind the mechanisms at the small scale, *i.e.*, the physics behind phenomenological models. This work is very much in this spirit.

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In recent years there has been much discussion of force-chains in granular media and their importance in controlling the mechanics at the larger scale. This phenomenon involves the development of spatially continuous lines of forces between contacting grains, by which the boundary forces are transmitted through granular masses. The buckling of such force-chains has been cited as a key mechanism associated with localised deformation and failure of granular bodies (*e.g.*, [1]). To understand such micro-scale mechanisms in granular materials requires the ability to measure both the kinematics (particle displacements and rotations) plus the force distribution through the granular assembly. Insight can be gained from discrete element simulations (*e.g.*, [2]), but these are just models and can, therefore, only help in the absence of real experimental data. Photoelasticity experiments are also very insightful (*e.g.*, [3]), but, whilst “real”, are highly simplified. For real materials, full grain kinematics characterisation has become possible using x-ray tomography plus particle tracking; *e.g.*, [4]. This current work tackles the second challenge, *i.e.*, measuring force distributions. Unfortunately, forces can not be measured, however they might be inferred from strains. This paper presents first results of spatially resolved neutron and x-ray diffraction measurements of crystallographic strains of sand grains (quartz crystals) in samples of sand under load. With such measurements, each grain essentially acts as a local 3D strain gauge or, for elastic deformations, a force gauge.

### 2 Coherent elastic neutron and x-ray diffraction

Coherent elastic neutron and x-ray diffraction have long been used in the characterisation of crystal structures, texture analysis and assessment of structural variations,

*e.g.*, strain or phase changes associated with pressure or temperature conditions. A brief outline of diffraction is given below; see *e.g.*, [5] for more details.

The interaction of x-rays with the electron cloud around an atom, or of a neutron with the nucleus of an atom, leads to diffraction of waves, which interfere constructively or destructively to produce diffraction patterns that are characteristic of the arrangement of atoms in the scattering crystal. The basic equation describing this coherent elastic scattering is Bragg's law,

$$n\lambda = 2d^{hkl} \sin \theta, \quad (1)$$

where  $n$  is an integer,  $\lambda$  is the incident wavelength,  $d^{hkl}$  is the spacing between planes in the  $hkl$  direction of the crystal lattice ( $hkl$  being Miller indices), and  $\theta$  is the angle between the incident ray and the scattering planes (whilst the interaction of x-rays and neutrons with atoms is different, scattering angles are the same, as  $\lambda$  can be equivalent). Note that measurements are usually made in terms of  $2\theta$ , plus two rotation angles,  $\eta$  and  $\omega$ , defined by the measurement system, which describe the position around spheres of constant  $2\theta$ . Interference of the diffracted waves is constructive for phase shifts of multiples of  $2\pi$  (secondary scattering is assumed negligible), to give Bragg peaks at  $(\eta, \omega, 2\theta)$  positions defined by, and characteristic of, the crystal structure. For a sample with many diffracting crystals at different orientations (*i.e.*, a powder), Bragg peaks, at each crystal-defined  $2\theta$  position, are distributed continuously in  $(\eta, \omega)$  giving "powder" rings.

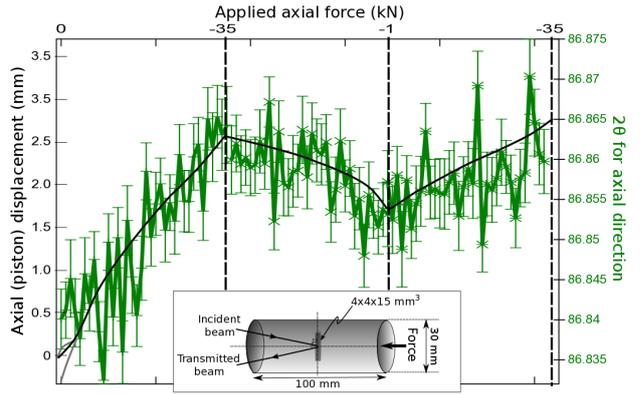
Variations of Bragg peak positions in  $\eta, \omega$  provide orientation information, whereas deviations in  $2\theta$  are associated with lattice distortions, *e.g.*, due to deformation. Measurement of strain from diffraction is thus given by changes in  $2\theta$ , from reference values  $2\theta_0$  and  $d_0^{hkl}$ ,

$$\epsilon_{crystal}^{hkl} = \frac{d^{hkl} - d_0^{hkl}}{d_0^{hkl}} = \frac{\sin \theta_0}{\sin \theta} - 1, \quad (2)$$

which defines positive values for increasing  $d$ , *i.e.*, expansion is positive. For a granular material comprising individual crystalline grains and inter-granular porosity, diffraction measurements are sensitive to the grains themselves and are not associated with porosity changes.

### 3 Neutron diffraction experiments and results

Neutron diffraction measurements have been made for sand under 1D (oedometric) compression using the monochromatic neutron diffraction instrument SALSA, at the Institut Laue-Langevin, which is optimised for residual stress determination in engineering components (*e.g.*,



**Fig. 1** Macroscopic axial displacement (initial loading cycle in grey) and change in  $2\theta$  for grain-strain in the axial direction (average over the gauge volume see in-set) as functions of the applied axial force. Inset - measurement and loading set-up.

[6]). Precise beam-defining optics allow small "gauge volumes" to be defined from where diffraction measurements are made. The vector that bisects the incident and diffracted beams ( $Q$ ) defines the direction of lattice strain measurement. Typical uncertainties in strain measures are about  $5 \times 10^{-5}$ .

Cylindrical samples (diameter 30 mm, length 100 mm) of Ottawa 50-70 sand, a well-rounded silica ( $SiO_2$ ) sand with an average grain size of about  $250 \mu\text{m}$ , were loaded in an aluminium oedometer *in-situ*, *i.e.*, whilst mounted in the diffraction set-up. Loading was force-controlled over a load-unload-load cycle of  $0 \rightarrow -35 \rightarrow -1 \rightarrow -35$  kN in steps of -1 kN (after an initial small loading cycle); compressive forces are negative. At each loading step the force was held whilst a diffraction measurement was made (60 s count time) for a gauge volume of  $4 \times 4 \times 15 \text{ mm}^3$  in the centre of the sample; the large gauge volume produced stronger scattering thus allowing faster measurements over a range of loads, at the expense of spatial resolution. A Bragg peak at  $2\theta \approx 86.8^\circ$  ( $\lambda = 1.64 \text{ \AA}$ ) was measured with a  $Q$ -vector (strain measurement direction) along the sample axis.

Figure 1 shows the piston displacement (indicating macro-strain) and the axial  $2\theta$  values (indicating grain-strain), averaged over the gauge-volume, as functions of the applied axial force. A reasonably uniform trend is observed in the macro-strain curve up to the first maximum load ( $\epsilon_{axial} \approx -2.5 \times 10^{-2}$ ) and the unloading follows a reduced gradient to a residual  $\epsilon_{axial}$  of about  $-1.75 \times 10^{-2}$  (recovered strain  $\approx 0.75 \times 10^{-2}$ ). Reloading follows a similar gradient to the preceding unloading (*i.e.*, the material response is stiffer than during the first loading) and a slightly increased axial strain is achieved. Globally, the grain-strain follows a remarkably similar trend, although with significant

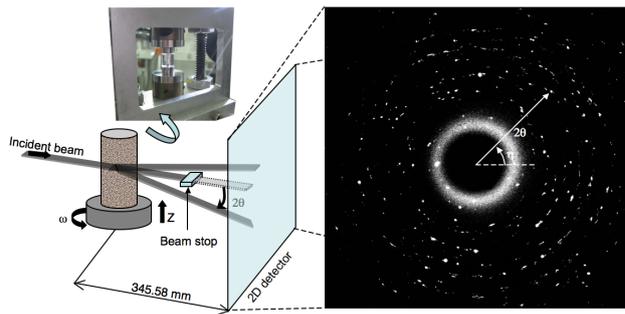
variation around it (which appears not to be just noise, but might indicate the occurrence of load transfer in and out of this volume). The first key observation is this correspondence. Another interesting observation is that the grain-strain at first increases more slowly, than the macro-strain, until about -10 kN, after which the gradient increases; this appears to correspond to a reduction in the macro-curve gradient (*i.e.*, a stiffer response). These trends perhaps indicate a change in the general deformation mechanism from porosity reduction to grain strain. The grain-strain after the first loading is about  $-1.9 \times 10^{-4}$  and the residual after unloading about  $-0.87 \times 10^{-4}$  (recovered strain  $\approx 1 \times 10^{-4}$ ). Thus, whilst there is a significant non-recovered macro-strain (predominantly porosity loss) as would be expected, there is also a significant residual grain-strain. This suggests that the grains remain confined between themselves (“locked”) after removal of the load. Other features exist in these curves, which will be investigated in the future.

#### 4 3D x-ray diffraction experiments and results

3DXRD microscopy, an extension of classical x-ray diffraction, is a relatively new tool for characterisation of position, volume, orientation and elastic strain of hundreds of individual grains and sub-grains inside bulk materials (powders or polycrystals); see [7]. In this work 3DXRD has been carried out on beamline ID11 at the European Synchrotron Radiation Facility where grain-strains can be measured with a resolution of about  $10^{-4}$ .

1D compression was carried out *in-situ* in the 3DXRD set-up (Figure 2) using quartz-glass oedometers (the container has to be non-crystalline, *i.e.*, amorphous, to avoid scattering that could interfere with the analysis). Displacement-controlled axial loading from both ends maintained the sample centred throughout the tests. Two oedometers were used, one of 1.5 mm internal diameter, to test the principle in simplified conditions, and the other having 10 mm internal diameter (external diameters were 10 mm and 50 mm, respectively). A coarser grading of Ottawa sand (20-30 - average grain size  $\approx 720 \mu\text{m}$ ) was used in this case, to have larger grains that might be more easily characterised.

In applying 3DXRD to sand, an assumption is made that the individual grains are themselves single crystals. This assumption has been tested using “extinction analysis” for a single grain and single diffraction angle. This involves identifying a single diffraction angle associated with a particular diffraction peak and acquiring radiographs for small increments in angle traversing the peak position. At the diffraction angle of the crystal the maximum amount of energy is diffracted away from the

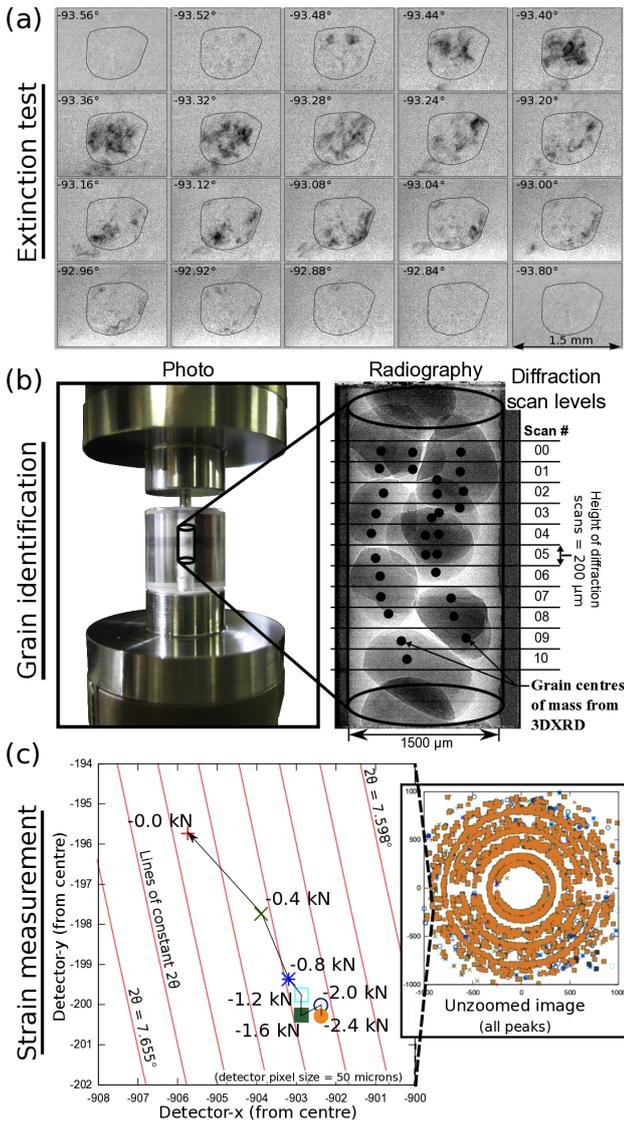


**Fig. 2** 3DXRD set-up and (to right) example diffraction pattern from a single  $\omega$  angle for the 10 mm oedometer.

direct path and so the grain should “extinguish” (*i.e.*, go dark on the radiogram); this is the basis of a similar method known as Diffraction Contrast Tomography [8]. If the grain being imaged is in fact a single crystal then the whole grain should show extinction at the same angle. Figure 3a shows the result of such a test. In fact the grain does not quite extinguish fully at a single discrete diffraction angle, but the difference in extinction angles is very small (about  $0.6^\circ$  from first to last extinction). The variations in scattering angle across the grain are likely due to small variations in crystal growth, or, more likely, plastic deformation through the long life-history of the sand grain.

Figure 2b shows an example diffraction pattern for a single  $\omega$  angle in the 10 mm oedometer specimen. Clear, isolated diffraction peaks can be seen. Using algorithms described in [7], the peaks in such diffraction images over a range of  $\omega$  can be indexed and associated with individual crystals, based on *a priori* knowledge of the quartz grains’ expected crystallographic structure. Figure 3b shows a comparison of the centres-of-mass of the grains identified in 3DXRD scans for the 1.5 mm oedometer with a radiograph (x-ray absorption) image of the specimen. A good correspondence of the positions of the grain centres (in each slice) is seen.

So, can grain-strains be measured? To address this question, full 3DXRD scans were performed (using the 10 mm diameter oedometer) during the unloading leg of a loading-unloading cycle (to avoid large grain rotations/displacements expected with loading). The loading/unloading was displacement controlled and the piston was locked during each set of 3DXRD measurements. The scans covered 7 positions across the sample width at 4 elevations about the centre of the sample (the beam was about 1.5 mm wide and 0.5 mm high); this total set of 28 scans took about 11 hours. Initial results indicate that strains within grains and very small grain rotations can be measured. Figure 3c shows the movement, as a function of the axial force,



**Fig. 3** (a) Extinction test: radiographs after subtraction of the average of the first and last in sequence (to remove approximately direct beam image) at  $0.04^\circ$  steps from  $-93.56^\circ$  to  $-92.80^\circ$ . (b) Comparison of centres-of-mass from 3DXRD and grain positions from radiography (1.5 mm oedometer), (c) Movement of a selected Bragg peak, for a grain at the centre of the 10 mm oedometer sample, as a function of decreasing load.

of a single identified peak position (one of very many peaks) for a grain within the central part of the specimen. This diffraction peak movement indicates a grain rotation in  $\eta$  and, more importantly, an increasing  $2\theta$  angle, *i.e.*, decreasing  $d$ -spacing and so shortening of this grain in this direction (roughly horizontal) corresponding to about  $-2.4 \times 10^{-3}$ . This is a key result as it indicates that the internal grains-strain can be measured using 3DXRD. Work is continuing to track all the peaks for all the identified grains and map grain-strain distributions that might be related to force chains.

## 5 Conclusion

Two methods, exploiting the diffraction of x-rays and neutrons, have been used to measure internal grain-strains of sand particles during compression. With such measurements, each grain can be thought of as acting as a local (3D) strain gauge. For elastic deformations, the strains inside the grains might be used to infer forces transmitted between contacting grains. Analysis of the data is continuing and further experiments are to be carried out, but the first results presented herein provide tantalising indications of the potential of these methods. The proof-of-concept has been provided and grain-strains have been measured (of the order of  $10^{-3}$  and  $10^{-4}$ , for neutron and x-ray measurements respectively). Spatially-resolved neutron diffraction has been shown to provide good resolution of strains, in large samples, as a volume average over many grains. Further detail on force distributions and load transfer will be gained from 3D mapping at different load levels using smaller gauge volumes (*e.g.*,  $2 \times 2 \times 2 \text{ mm}^3$ ). 3DXRD has a lower strain resolution, but, as *individual* grain-strains can (and have been) measured, this could allow direct observation of force-chain structures. In addition individual grain (and sub-grain) kinematics can be observed. When linked with tomography imaging (underway), datasets of full grain kinematics and force transfer (from the grain strains) will be possible.

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