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# Stress distributions in compacted powders in convergent and stepped dies

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#### 1. Introduction

Granular materials are ubiquitous and have unique mechanical properties. They can act like a fluid or solid depending on the situation and show a wide range of behaviours at various length scales (both microand macroscopic). The mobility of particles tends to mean that mechanical interactions within a granular assembly are far more complex than those within solid engineering materials. High stress compaction processes lie within the transition between these two states of matter and have particular relevance in various geological systems as well as industries such as powder metallurgy, pharmaceutical pill production and ceramic processing. Improvements in our understanding of compaction are therefore beneficial in a wide range of engineering and natural systems.

The diffraction of thermal neutrons ( $\lambda \approx 1-3$  Å) is a widely used experimental technique for studying the internal structure and mechanical behaviour of solid materials [1]. Through the measurement of changes in interatomic plane spacing,  $d_{hkl}$ , detailed information on the strain (and stress) state within crystalline solids can be obtained. This approach has become very useful in the scientific study of the mechanics of solids as well as the assessment of externally applied or residual stress (due to plasticity or thermal processes) in an engineering context [1,2,3,4]. Compared to X-ray diffraction, neutrons penetrate relatively large distances into most materials and allow the measurement of the strain deep within samples.

Some recent work has adapted neutron diffraction techniques to the study of the mechanics of granular materials [5,6,7,8]. In much

#### ABSTRACT

Neutron diffraction techniques are used to measure a series of bulk stress distributions in granular materials undergoing high stress die compaction. Two different granular materials are studied: a ductile material in the form of iron powder and a brittle material in the form of quartz sand. The behaviour of these two materials is examined in terms of the resulting stress distribution within two dies of different geometry: an axisymmetric converging die and a stepped cylindrical die. These measured distributions are examined and discussed to highlight both similarities and differences in the mechanical response of these contrasting materials at high consolidating loads. The potential for neutron diffraction techniques to provide full-field information on the mechanical response and stress within granular samples is demonstrated.

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the same way as solid materials, neutrons are diffracted by crystal structures within particles in a granular assembly. Corresponding diffraction based strain measurements made in these systems have been shown to provide information on both the bulk [7,6] and discrete [8] stress states within these systems. This approach is able to provide a wealth of information for both scientific study as well as model validation.

In this paper we apply neutron diffraction to the measurement of stress distributions during the compaction of two granular materials; a ductile iron powder and a brittle quartz sand. These powders were compacted in two dies of different geometry: a convergent axisymmetric die and a cylindrical stepped die. The aim of this work was to develop some understanding of the influence of material properties and die geometry on the stress distribution within a powder compact.

### 2. Strain measurement and stress reconstruction from neutron diffraction

Measurement of strain using neutron (or X-ray) diffraction is well developed experimental technique for which there is a wealth of literature (e.g. [2,3,4]). A detailed description can be found elsewhere, however a brief overview of the technique is as follows;

Neutrons are scattered by direct interactions with the nuclei of atoms within a sample. Constructive interference between neutrons scattered from a crystal lattice occur in directions satisfying Bragg's law:

 $n\lambda = 2d\sin\theta.$ 

(1)





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In a constant wavelength experiment (see Fig. 1), a narrow beam of fixed wavelength neutrons is passed through a sample with a selected diffracted beam viewed through a slit or collimator system. Through this arrangement, a very accurate measurement of a scattering angle can be made for a given set of crystal planes within a small, well-defined region, known as the gauge volume. Any strain within the sample (residual, thermal or applied in situ) can then be detected as a small shift in this scattering angle. In terms of the measured lattice spacing *d* (inferred through Bragg's law), the strain within the gauge volume can then be calculated as:

$$\langle \varepsilon_{\kappa} \rangle = \frac{d - d_0}{d_0},\tag{2}$$

where  $d_0$  represents a reference lattice spacing measured in the absence of load.

This strain measurement is not only confined to the gauge volume, but is also specific to a single direction defined by the scattering vector,  $\kappa$ , which bisects the incident and diffracted beams. At each point within a sample it is possible to measure sufficient strain components to reconstruct the full triaxial strain tensor,  $\varepsilon_{ij}$ . This process relies upon least squares fitting of the strain tensor to a set of individual measurements made from different directions within the sample. A detailed explanation of this procedure can be found in [8]. Through exhaustive measurement over a mesh of points, it is possible to map out the detailed spatial distribution of the triaxial strain tensor throughout a sample.

Given that such a measurement only refers to the elastic variation in lattice spacing, it is possible to reconstruct the stress tensor from measured strain using Hooke's law, regardless of any plasticity in the sample (see [6] for a detailed discussion of this point). That is, stress can be calculated from strain via:

$$\left\langle \sigma_{ij} \right\rangle = K \left\langle \varepsilon_{kk} \right\rangle \delta_{ij} + 2G \left\langle \varepsilon_{ij}^* \right\rangle, \tag{3}$$

where *K* and *G* are (respectively) the bulk and shear moduli, and  $\langle \mathcal{E}_{ij}^* \rangle = \langle \mathcal{E}_{ij} \rangle - \frac{1}{3} \langle \mathcal{E}_{kk} \rangle \delta_{ij}$  is the deviatoric component of strain. Note that the effects of single crystal anisotropy play a role in this relationship and elastic constants must be chosen accordingly (known as 'diffraction elastic constants' [4]). However through a careful choice of crystal planes with a normal compliance close to the polycrystalline average, these effects can be minimised.

In some respects, the application of these techniques to granular materials is unremarkable; diffraction methods provide average elastic strains within the solid particles in an assembly. However there is to be some interpretation when translating this information to the 'granular continuum'. Bulk stress in this setting refers to a spacial average of



Fig. 1. Top: Neutrons (as waves) are scattered by a crystal lattice, forming diffraction peaks at angles satisfying Bragg's law. Bottom: In a fixed wavelength instrument, shifts in a single diffraction peak are measured and interpreted as lattice strain. Adapted from [6].

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