Bragg Institute Neutron Beam Instrument Proposal

ID	**P1458**
Title	**In situ study of stress distribution in a compacting particulate system: 1. Spherical particles.**
Round	**2010-2 Neutron**
Type	**Normal**
Status	**Completed**
Date Created	**17/5/2010**
Date Submitted	**28/5/2010**
Date Completed	**4/3/2011**

Publications

This is a list of publication associated with this proposal.

1. Wensrich, CM; Kisi, EH; Zhang, JF and Kirstein, O, Measurement and analysis of the stress distribution during die compaction using neutron diffraction, *Granul. Matter* **14**(6), 671-680 (2012) (**from Kowari**) DOI

Schedule

The experiment has been scheduled using the following equipment and scientists.

Equipment	Start Date	End Date	Length
Kowari	19/01/2011	25/01/2011	7
Kirstein, Oliver	19/01/2011	25/01/2011	7
Kowari 100kN Load Frame P-2	19/01/2011	25/01/2011	7
User Supplied Sample Environment - Kowari	19/01/2011	25/01/2011	7

Report

Experimental Report

The mechanical interaction of discrete particles is of considerable interest in a wide range of industries including the bulk handling of mineral ores, powder metallurgy and the compaction of pharmaceutical products into pills and capsules. Particle mechanics, the modeling of many body systems of discrete particles under applied load, is a growing area of research and many models for the stress and strain distribution in compacting systems have been developed. This experiment was intended to demonstrate proof of concept for applying neutron diffraction residual stress measurement techniques to a compacting powder using a simplified experimental arrangement comprising a single action cylindrical die and spherical particles. The results will be used to verify/modify particle mechanics models of the system and assist in planning future experiments to explore the influence of the many other particle (shape, size etc), mechanical and material (hardness, modulus etc) variables.

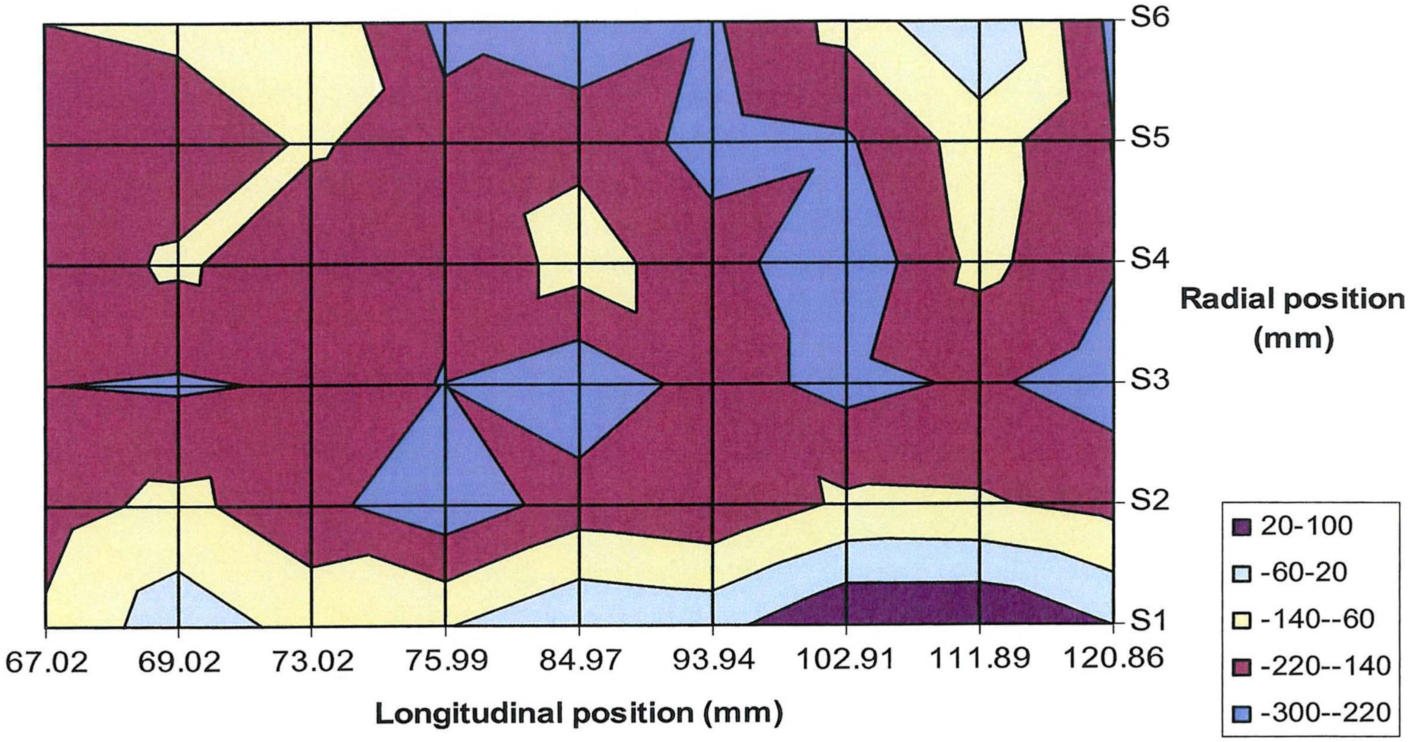
After initial problems with the steel die due to attenuation, a uniaxial 25 mm diameter aluminium die was filled to a depth of 59 mm with spherical copper particles ~110 micons in diameter. Copper was substitued for the original choice (alumina) due to the latter's high modulus, incompatibility with the Al die strength and difficulty obtaining monodisperse powders. Copper has a lower elastic modulus (to give large strains) and much larger scattering length than alumina. We chose the 200 reflection since calculated diffraction elastic constants under teh Reuss approximation gave the largest compliance for these planes. Assuming radial symmetry, diffraction data were recorded in a 2-dimensional section from the centreline of the die to the exterior of the die wall and spanning from the bottom to the top of the die. A gauge volume of 4x4x4 mm gave good diffracted intensity however a 2mm sample scan step was used to improve the spatial resolution. Strain scans were made at 9 positions along the length of the die with 6 sampling positions in each scan. Four 2-d strain scans were made to yield strains in the hoop, radial, axial and one oblique direction - sufficient to extract principal strains.

Peak position was then used to establish the d-spacing and strain at each position in the four 2-d strain maps. Figure 1 shows the strain distribution in the hoop scan and Figure 2 in the axial scan as examples. Note that the hoop strain is fairly uniform other than falling towards zero at the die wall (at S1) as expected. In figure 2 there is a continuous reduction in the axial stress on moving further from the consolidating pressure of the ram (at position 120.86mm). These observations can be seen more clearly in Figure 3 which shows the average hoop strain as a function of distance die-centre (S6) to die-edge (S1) and Figure 4, the average axial strain (black squares) as a function of distance along the die from the base (67.02 mm) to the ram (120.86 mm).

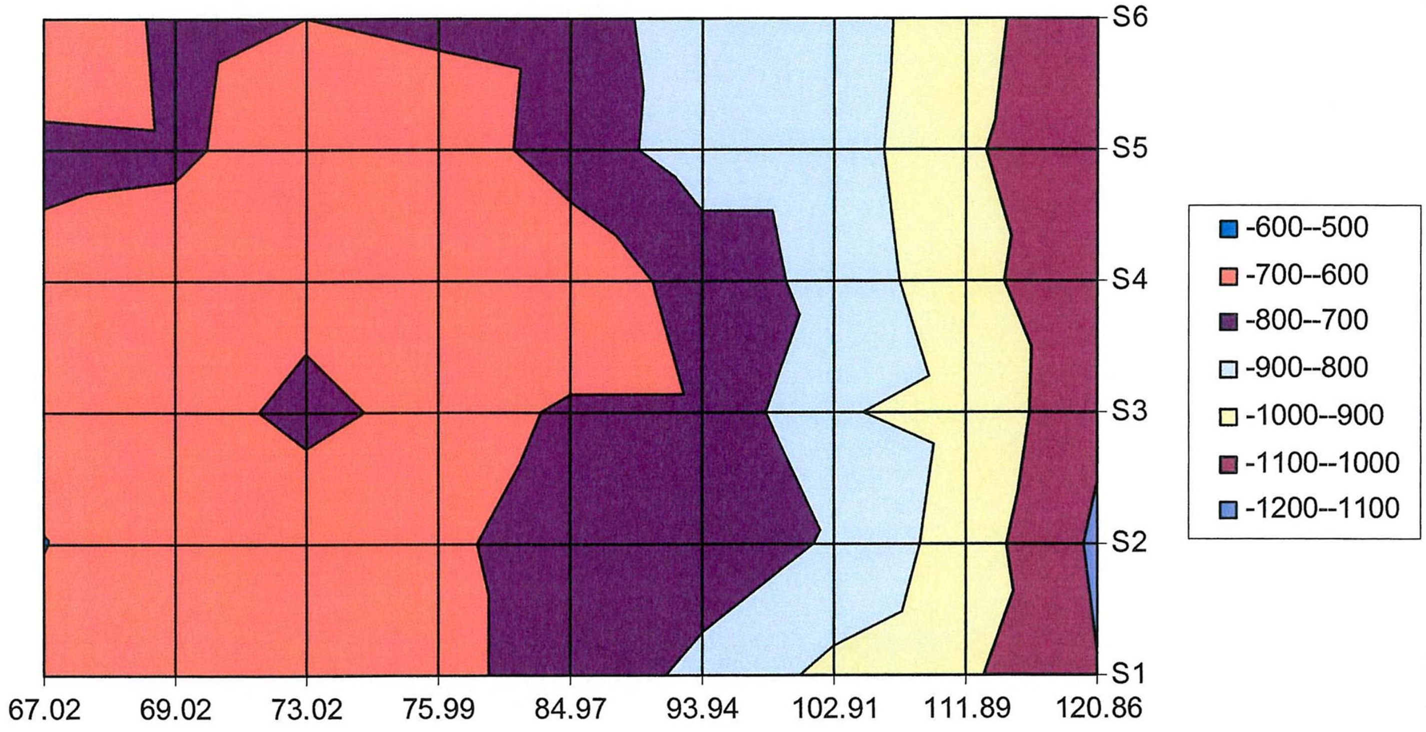
The strains have been converted to stress under the assumption of Reuss-like behaviour i.e. relatively free particles and the resulting data are being analysed and compared with various particle mechanics models. It is expected that they will be submitted for publication late in 2011.

Figures

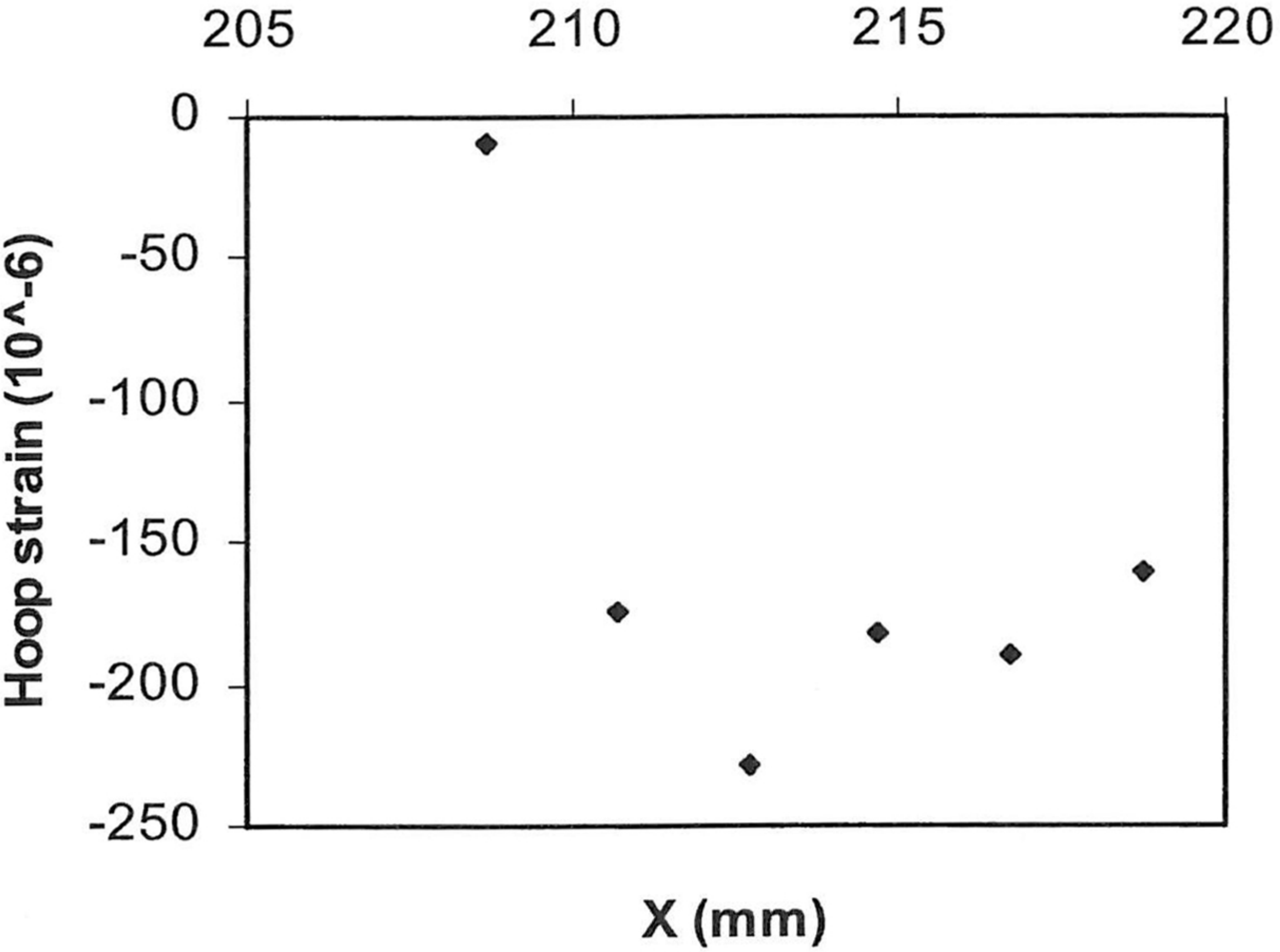
Hoop strain distribution in compacting Cu powder at 60 MPa consolidation pressure.



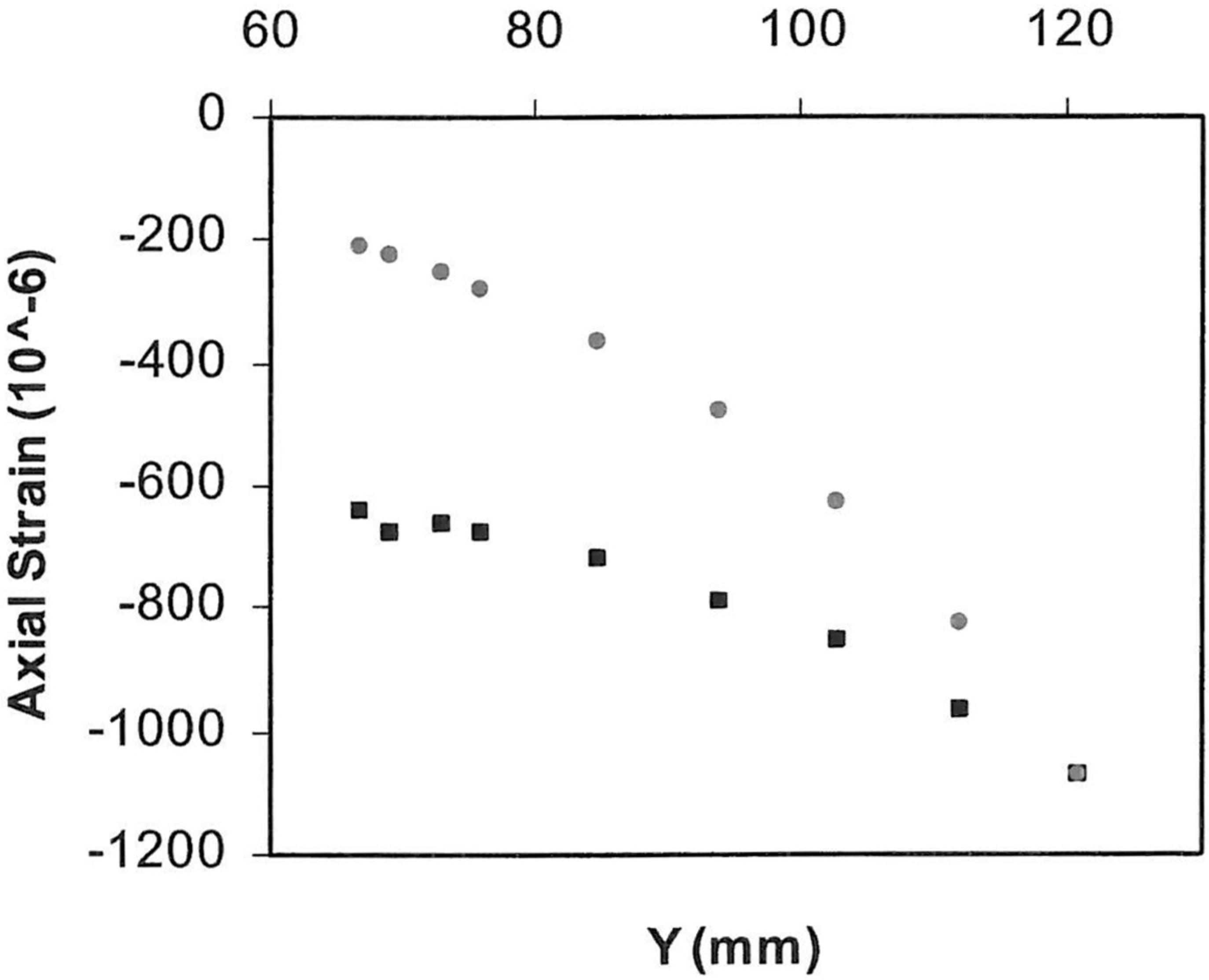
Axial strain distribution in compacting Cu powder at 60 MPa consolidation pressure.



Average hoop strain from die wall (laft) to die centre (right)



Average axial strain from die wall (laft) to die centre (right)



Researchers

Name	Role	Attending	First Time User	Position (at submit)
Erich Kisi (Uni Newcastle, AU)	Principal Scientist	Yes	No	Staff
Oliver Kirstein (European Spallation Source)	Co-proposer (editor)	Yes	No	Staff
Christopher Wensrich (Uni Newcastle, AU)	Co-proposer (editor)	Yes	No	Staff
Jian Feng Zhang (Uni Newcastle, AU)	Co-proposer (editor)	Yes	No	Post Graduate

Instruments

Name	Desired Date	No. days requested
Kowari	13/09/2010	8

Name	Desired Date	No. days requested

Name	Requested
Kirstein, Oliver	Yes
Kowari 100kN Load Frame P-2	Yes
User Supplied Sample Environment - Kowari	Yes

Experimental Setup: Strain Scanning with user supplied uniaxial die and press.

Details

Scientific Area **Materials**

Impossible Dates **Please note, preferred dates in the period 8th November - 26th November. Prefer two blocks of 4 days rather than a single block of 8 days.**
Impossible dates are 24th September - 11th October 2010 (Kisi and Kirstein).

Proposed Research (Text):

Scientific Background

The mechanical interaction of discrete particles is of considerable interest in a wide range of industries including the bulk handling of mineral ores, powder metallurgy and the compaction of pharmaceutical products into pills and capsules. Particle mechanics, the modeling of many body systems of discrete particles under applied load, is a growing area of research and many models for the stress and strain distribution in compacting systems have been developed. There is broad intersection with fields such as geotechnical engineering (the modeling of compacting soils and clays) and through the various micromechanical models used, with the analysis of residual stress in engineering materials.

When a mechanical load is applied to a system of particles, the system response depends on many factors including: the particle size, shape, size distribution, surface roughness and surface adsorbed species; geometric factors such as the breadth and depth of the particle bed; mechanical factors such as inter-particle friction, wall friction and the stiffness of the wall. Consider the simple case of a cylindrical die filled with powder particles and acted upon by a close-tolerance ram. As a result of the factors outlined above, the stress (and strain) distribution in the compacting powder will be mechanically inhomogeneous with wide variation in the direction and magnitude of the stresses experienced by the powder. This inhomogeneity causes problems such as cracking in green ceramic and pharmaceutical compacts and uneven shrinkage in sintered metal products. Although great inroads have been made with modeling, verification of model predictions has been very difficult, usually relying on the average behaviour of the entire system *after* removal of the load. Conventional experiments lack the ability to probe the behaviour of the system while under load and also have limited capacity for spatially resolving the stress (strain) distribution in the particulate system.

The mechanics of granular materials can be examined using a variety of approaches. These approaches vary from simple continuum models that make generalised assumptions regarding the structure of the stress within the material (typically from critical state soil mechanics), to sophisticated models involving computational continuum mechanics (e.g. [1]) or the Discrete Element Method (DEM) (e.g. [2][3]). Simple models still provide valuable information on the overall behaviour of the material, while the finer details of the inhomogeneity of the stress state and density can be examined using the more sophisticated approaches. At all levels there are questions to be answered. For example, the simple continuum models rely on the assumption that the material is approaching a critical or asymptotic state of stress (often expressed as a coefficient of lateral stress). The actual stress state is entirely dependent on the loading history and these critical stress states may never be achieved. To what level is the critical state assumption valid during die compaction?

DEM is by far the most recent approach in the modelling of granular materials. DEM refers to a technique where a granular material is modelled at the level of individual particle interactions. Each particle in a discrete element model carries with it its own equations of motion that are based upon the forces of interaction between the particle and its neighbours and whatever body forces are present. These forces are calculated using a variety of models such as such as Hertz-Mindlin contact, Coulomb friction, electrostatics, and the behaviour of liquid bridges (and whatever else the case may be). At each time step in a simulation, the equation of motion of each particle is updated and integrated to determine the new position and velocity (translational and angular) for the next time step. By simulating each particle in an assembly in this way, the bulk behaviour of a material can be determined.

This technique has an apparent simplicity, but in reality it is a massive computational task. This is evident in the fact that the pioneering work in the area by Cundall and Strack [4] is over 30 years old, while practical application is a much more recent phenomenon. The acceptance of DEM has closely mirrored the availability of affordable computational power, with simulations of several hundred thousand particles now being commonplace.

Since the very beginning, the most important issue surrounding DEM has been the validation of models. By their very nature, DEM models contain many parameters, and so the level of rigor associated with validation is directly dependant on the level of detail in the experimental data used. Sophisticated models can easily be 'tuned' to replicate simple measurements. More sophisticated models require much more detailed information for validation. Detailed stress and strain information within these materials is very difficult to measure by traditional means. Boundary stresses can be measured in some situations (e.g. [5]), however, these measurements are typically restricted to averaged quantities over quite large areas. From this perspective, there is a great need for detailed measurements of the distribution of stress within granular solids under load.

The penetrating power of neutrons is widely used to probe strain and stress distributions within solids and it is a logical step to extend this application of neutron diffraction to granular systems. By using a dedicated residual stress diffractometer like KOWARI, it will be possible to measure tri-axial, 3-d spatially resolved strains *in situ* while granular samples are held under loads in various configurations of interest. From this data, elastic moduli will be used to infer the stress distribution in the system for comparison with the results of the various models available. The stress distribution in the die walls can also be investigated as part of the same series of experiments as well as the onset of plasticity. It is anticipated that the work may open up an entirely new way to experimentally investigate particulate systems under load. This in turn may assist a transition in process-design methodology away from trial and error methods to methods based upon verifiable mathematical models.

Aim

This experiment is intended to demonstrate proof of concept for the technique using a simplified experimental arrangement comprising a single action cylindrical die and spherical particles. The results will be used to verify/modify particle mechanics models of the system and assist in planning future experiments to explore the influence of the many other particle, mechanical and material variables.

In terms the scientific value, there will be two direct outcomes of this experiment;

- The direct examination of the structure of stress within a compacted granular assembly will answer important questions regarding the nature of the effects of boundary friction within die compaction, and in a broader sense the nature of the development of critical stress states (both active and passive).

- The experiment will provide a remarkable test bed for DEM in terms of the amount and quality of data available for validation.

The University of Newcastle hosts the Centre for Bulk Solids and Particulate Technologies (CBSPT), arguably the world's premier bulk solids handling research laboratory and is hence well placed to integrate the results of the research with existing experimental and computational studies.

Experimental details

A uniaxial 20 mm diameter steel die will be filled to a depth of 40 mm with spherical alumina particles 250 mm in diameter. Alumina was chosen as the powder material due to a good ratio of compressive strength to elastic modulus (to give large strains), no plasticity mechanism and availability. Assuming radial symmetry, diffraction data will be recorded in a 2-dimensional section from the centreline of the die to the exterior of the die wall and spanning from the bottom to the top of the die. We anticipate that 4 strain scans should suffice. The first is a fairly sparse scan to establish the appropriate d_0 values for the powder and die walls. Next, higher positional resolution strain scans will be used to map the strain distributions at two values of the consolidating pressure and again at the lower pressure during unloading (which has built in hysteresis due to changes in the granular body at the higher pressure). We anticipate a gauge volume of 2 x 4 x 4mm (radial, hoop and axial directions) scanned in 2 mm steps should give the required compromise between diffracted intensity and spatial resolution. At each position, the longitudinal, radial and hoop components of strain will be determined from the d-spacings in the particles determined by diffraction. These data will be publishable in their own right following which they will be used to improve both algebraic continuum models and DEM models of this granular system

Preliminary work

The working powder will be fully characterised in the CBSPT's Newcastle laboratory before the experiment. This will include the frictional characteristics as a function of consolidating pressure, consolidation ratio, particle size and size distribution etc. XRD and SEM will also be used to characterise the spheroids.

Choice of instrument

KOWARI is designed for investigating residual stresses in solids and as such is purpose built for experiments of this kind. We have based our beam time request upon a preliminary scan, then 3 loads and a 20 x 40 matrix of diffraction patterns repeated 3 times (for radial, hoop and axial components of strain). Assuming each diffraction pattern to take 5 minutes for sample positioning and data collection, plus a small allowance for changes or orientation, we estimate that a minimum of 8 days are required.

Experimental Needs:

Special Requirements:

Hazards:

References: [1]AJ Abbo and SW Sloan, A comparison of integration schemes for elastoplastic constitutive laws Research report, University of Newcastle, Department of Civil Engineering and Surveying ; no. 091.02.1994, 1994.
[2]GC Barker, Computer simulations of granular materials, in: A Mehta (Ed.), Granular Matter: An Inter-Disciplinary Approach, Springer, Berlin, 1994.
[3]AB Yu, Discrete element method - an effective method for particle scale research of particulate matter, Engineering Computations, v21 pp205-214, 2004.
[4]PA Cundall, and O Strack, A discrete numerical model for granular assemblies. Geotechnique v29, pp47-65, 1979.
[5]T Krull, MG Jones and S Keys, Stress-field modeling and pressure drop prediction for slug-flow pneumatic conveying in an aerated radial stress chamber, Particulate Science and Technology, v22 129-138, 2004.

Part of a Thesis: No

Samples

Sample Desc / Name	Form	Hazardous	Prepared
Alumina microspheres	Powder		2010-07-14

Comments: Very inert, commonly used material. Not fine enough to cause dust hazard.

Composition:

Hazards: Not hazardous

Crystal Info:

A: 4.76 B: 4.76 C: 12.992 Alpha: 90 Beta: 90 Gamma: 120 At Temperature: 295 Space Group: R -3 c (H)

Name	Formula	CAS	Amount	Hazards
Aluminum oxide	Al2-O3	1344-28-1	40g	Toxicity: 2 Chronic: 2

Sample Desc / Name	Form	Hazardous	Prepared
Cu microspheres	Powder		Yes

Comments:

Composition:

Hazards: Not hazardous

Name	Formula	CAS	Amount	Hazards
Copper	Cu	7440-50-8	50g	Toxicity: 2 Body Contact: 2 Reactivity: 2 Chronic: 2