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Stress gradient within powder en masse during hydrostatic compression

Hojae Yi *, Virendra M. Puri

Department of Agricultural and Biological Engineering Department, The Pennsylvania State University, Agricultural Engineering Building, University Park, PA 16802, United States

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ABSTRACT

Non-homogeneous density of compressed powder has been known and documented. This phenomenon suggests that the stress inside a powder en masse is not homogeneous during the compaction process. This hypothesized stress distribution inside powder bulk suggests a relationship between the dimension of powder bulk under compaction and the level of internal stress. To elucidate this relationship, stress changes inside a powder bulk during hydrostatic compression were measured using a fundamental mechanical tester, namely, a cubical triaxial tester. We found that the internal stress decreased when the distance from the load-acting surface became larger. For example, along the central line of the sample with 100 kPa of hydrostatic stress, the internal stress decreased 32.2% from 94.7 kPa to 64.2 kPa and down to 55.1 kPa (41.9% decrease), when the distance from the surface increased from the pressured surface to 1.52 cm and to 3.04 cm. To quantify the contribution of distance from the load-acting surface to the decreasing stress, a linear relationship and an exponentially decaying function were tested. It was shown that the exponentially decaying function explains the decreasing stress well. The rationale of the exponential decaying function was discussed with respect to the classical elasticity formulation.

1. Introduction

Manufacturing processes involving powdered materials are known to have many undesirable issues. Compaction of particulate solids is a very common and critical process in many industries. The forming of powder compacts plays a significant role in: subsequent processes, final quality of the product, and the overall efficiency of the processes. One of the important compaction issues originates from the density distribution of compacts, including non-metallic powders.

The nonuniform density of green compacts is thought to be an artifact of nonuniform compaction. In other words, bulk powder mass agglomerate heterogeneously in different regions, which experience different stress conditions during compaction. We hypothesize that this heterogeneity of powder en masse, loose or compacted, is a result of stress gradient which is related to the distance from the surface where stress is applied and the external force itself. This is particularly important in understanding the evolution of powder compact because if stress gradient develops, it will result in nonuniform compacts. In turn, this can be a source of anisotropy, non-linear behavior and so on; all of which are undesirable. Moreover, this stress gradient leads to an important question: what is the right size of test specimen since the stress gradient inside a powder mass under stress implies that the size of test specimen affects the measured mechanical value due to varying consolidation status.

Study on this issue dates back to Train [1], who employed a uniaxial die–punch system to compress a Heavy Magnesium Carbonate B.P. According to Train [1], regardless of the condition of lubrication of die and pressures, a compact develops density distribution, which the author attributed to elastic relaxation along the major principal direction of pressure application. Recent studies also report uneven compacts. The evidences of such uneven powder compaction were presented as density distribution of compacts observed with various analytical methods [2–6].

Even though these studies documented nonuniform compaction due to a hypothesized stress gradient of powder en masse, the actual stress distribution during powder compaction has not been measured directly. This information is of crucial importance in verifying the hypothesis of stress distribution inside a powder system as well as in the development of a quantitative relationship between the actual stress experienced by the powder sample and applied stress.

As the first step to understanding this issue, we attempt to determine the hypothesized stress gradient inside the bulk solid sample during isotropic compaction by measurement of pressure inside a specimen under hydrostatic compaction using a fundamental mechanical tester. A cubical powder test specimen was compressed using an applied isotropic stress boundary condition (i.e., stress magnitude is same in all three principal directions, hydrostatic stress condition) and the resulting stress *inside the powder sample* was measured during the compaction process. The results were studied to examine differences of the measured stress values at various locations inside the powder sample and to determine and explain the relationship between the measured stress inside the sample and the distance from the pressure application surface and applied pressure.

* Corresponding author.
E-mail address: huy1@psu.edu (H. Yi).

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2. Method and material

2.1. Cubical triaxial tester

To apply isotropic (hydrostatic) stress, a low pressure Cubical Triaxial Tester (LPCTT) was used. A cubical triaxial tester is a fundamental mechanical tester that employs flexible pressure application membrane to apply force on the six faces of the cubical test specimen. The LPCTT can apply up to 100 kPa with 6.1 cm by 6.1 cm by 6.1 cm cubical test specimen when powder sample was prepared without sample holding membrane. Fig. 1 is a schematic of LPCTT, which was originally developed by Kamath and Puri [7].

The LPCTT uses two individual pressure controllers (Proportion-Air Inc. QB1TFEE015), which can deliver pressure from 0 to 100 kPa at 0.2% of accuracy over the entire range. Individual pressure controller regulates horizontal and vertical pressures independently via software developed with LabView™ version 8.2 (National Instruments™).

The application of pressure results in reduction in the dimensions. The decrease in dimension is continuously monitored using six linear motion potentiometers that are in constant contact with the opposite surface of pressure application membranes. Albeit a linear motion potentiometer itself has an infinite resolution, the actual measurements were recorded with ± 1 μm of resolution due to the limitation of the data acquisition system built with the LabView™ version 8.2 (National Instruments™). The LPCTT has the capability of displacement as large as 6.1 cm in each of the three principal directions.

Data that are measured with the LPCTT include applied pressures and resulting displacements in the three orthogonal directions, i.e., vertical (gravity) direction and two perpendicular horizontal directions. This feature has a substantial advantage over the conventional cylindrical triaxial tester, with which usually the displacement only in the vertical (gravity) direction can be measured readily. A few researchers have modified the conventional triaxial tester to measure redial displacements, but this requires substantive changes to the conventional test set up (VMP to give HY two/three references here). The versatility of the LPCTT in determination of parameters of various constitutive stress–strain relationships has been well documented [8–11].

Because the LPCTT can control pressure in the three principal directions independently, numerous stress paths and compaction rates can be investigated. In this research, only isotropic stress condition was applied following the Hydrostatic Triaxial Compression (HTC) stress path. In HTC, identical stresses were applied to six faces (bounding surfaces) of the cubical test specimen. While the pressures in the three principal directions increase by the same amount of increment, the compressed test specimen's dimensions are measured. In this study, pressure was increased from 0 to 100 kPa at 15 kPa/min rate. The slow rate is to minimize any time-dependent mechanical behavior effects. Five replications were performed to estimate descriptive statistics parameters of the measured mechanical properties of powder specimens.

2.2. Stress measurement inside a cubical test specimen

For the measurement of stress developed at various locations inside a cubical powder specimen, precision miniature pressure sensor (Model 105S, Precision Measurement Company, Inc. Ann Arbor, MI) was used. The 105S sensor can measure pressure ranging from 0 to 200 kPa with 0.5 kPa of resolution. The sensor is a small and thin disk whose dimensions are 2.5 mm in diameter and 0.5 mm of thickness. Because of its small dimensions, the interference of the embedded sensor on the compaction of powder expected to be minimal, i.e., the volume of the sensor is less than 0.008% of the test specimen. To measure the internal stress of powder sample, the miniature pressure sensor was placed at a specific location inside the powder en masse during the deposition of powder in the test chamber using the spoon-by-spoon filling method. The active surface of pressure sensor was oriented to be consistent with vertical principal stress direction. During test, the location and orientation of the sensor should remain unchanged as the applied stress is increased. Even though the sample experiences volumetric compression, the relative location of the miniature pressure sensor had not changed based on careful visual observations made before and after the test. During the powder sample preparation, the pressure sensor is placed at the locations along centerline as well as a diagonal line including the surface and the geometric center of the test specimen as illustrated in Fig. 2 (sensor locations are given in Tables 2 and 3). One

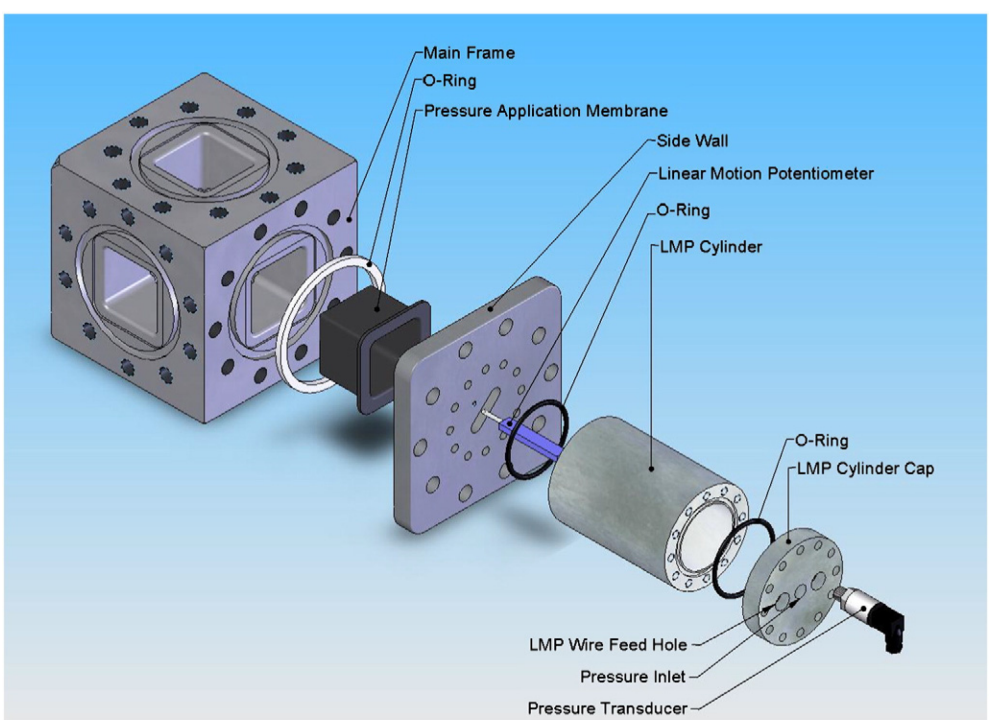

Fig. 1. Exploded view of one side of cubical triaxial tester.