CAN ONE MAKE A POWDER FORGET ITS HISTORY?

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Abstract

It is shown that computer simulations can qualitatively reproduce experiments, where a powder of cohesive, round, hard particles is periodically deformed at constant volume. Two types of initial configurations are considered: Uniaxially precompacted ballistic deposits and biaxially precompacted DLA-clusters. Both initial configurations had the same volume fraction, but due to the different precompaction procedure completely different principal stresses. After a transient which lasts only less than a period, the stresses follow the same periodic function, i.e. the powder forgot its history.

1. INTRODUCTION

One of the fundamental open questions in the physics of powders is how to characterize their state uniquely, the particle configuration in general depending on the deformation history of the powder. This is in marked contrast to thermal equilibrium systems, which statistically explore all available configurations, so that temporal correlations normally die out quickly. A powder, however, is “jammed” or frozen in, if it is not driven by external forces. Even if it flows, it does not explore its alternative configurations on a time scale which is short compared to the inverse strain rate.

This has macroscopic consequences. For example the stress in a compacted cohesive powder not only depends on the solid fraction but also, by what kind of compaction the solid fraction was reached. It is believed, that at least the fabric tensor is needed in order to determine the stress state. Any microscopic theory of the dynamical evolution of the state and hence of powder flow requires an answer to the question, how to characterize such a state. Therefore it is very important also for macroscopic predictions needed e.g. in process engineering.
In this paper we address only a partial aspect of this question. It is known, that the stress components in a powder, which is quasi-statically deformed at fixed volume, approach values independent of the precompaction history [1]. This stress state depends on the volume fraction, but also on the total deformation at constant volume: As one continues to shear, the stress components increase. The point we want to make in this paper is that there are also other ways to make the powder forget its precompaction history. As an example we investigate the periodic kneading of the powder at constant volume. In contrast to steady state flow the principal axes switch back and forth periodically in this case. This has the advantage that the sample returns to its initial shape periodically. In every period steady state flow properties may be reached or not, depending on how long the period lasts.

Periodic kneading was studied in a different context previously [2]. There it was shown for limestone that the stresses are periodic functions with decreasing amplitude. After a few periods the amplitude becomes constant. In the present paper we compare experiments on carbonyl-iron powder and computer simulations. In contrast to limestone the particles are spherical and very hard. As we are going to show, the amplitude of the periodic stresses becomes constant already within one cycle in this case. Simultaneously all stress tensor differences due to different sample preparations vanish within this short transient, i.e., the powder forgets its history.

2. METHODS

2.1 Experimental Setup

The true biaxial shear tester allows the deformation of a brick shaped bulk solid specimen. The deformation mechanism is composed of a bottom and a top plate which are fixed at a distance of 36.5 mm from each other and four side plates which can be moved independently from a maximum distance of 130 mm to a minimum distance of 70 mm, always staying perpendicular to their adjacent ones. Thus the bulk solid specimen remains brick shaped and principal strains equal the normal strains (Fig. 1a):

\[
\epsilon_{ij} = 0, \text{ where } i \neq j; \quad \epsilon_{ii} = \epsilon_i.
\]  

(1)

The side plate’s velocities can be set in steps of 0.03 \(\mu\)m/s up to a maximum velocity of 65 \(\mu\)m/s corresponding to maximum shear rates of 0.1% per second. On the specimen’s border, the complete stress state is measured with three five component load cells [3], which are installed in the bottom and in two perpendicular load plates, LC 1, 2, and 3 in Fig. 1b. For the tests with the true biaxial shear tester, silicon grease is spread on bottom, lateral and top plates. Then they are covered with highly flexible rubber membranes in such a way that the membranes are
evenly deformed with the borders of the powder specimen. The friction between specimen and load plates thus is minimized, hence shear stresses are negligible. Therefore, the normal stresses on the specimen borders are principal stresses. The principal stress axes coincide with the deformation axes (Fig. 1a):

\[ \sigma_{ij} = 0, \text{ where } i \neq j; \quad \sigma_{ii} = \sigma_i. \] (2)

In this paper we use the engineering sign convention for the stress, i.e. a compressive stress is defined as positive. Deformations and stresses are continuously measured using a process computer, which also controls the deformation process \(^\text{[2]}\). As experiments with finite strains are carried out, we define the strains using the natural strain increment \(^\text{[4]}\) and obtain

\[ \epsilon_i = \ln \frac{L_i}{L_{i0}} \] (3)

for the strains in \(x\)-, \(y\)- and \(z\)-direction and for the volume strain respectively:

\[ \epsilon_V = \ln \frac{V}{V_0} = \ln \frac{L_x L_y L_z}{L_{x0} L_{y0} L_{z0}} = \epsilon_x + \epsilon_y + \epsilon_z \] (4)

In order to perform tests at constant volumes, one can easily control the condition \( \epsilon_x + \epsilon_y = \text{const.} \) (\( \epsilon_z = 0 \) during the complete measurement due to the fixed bottom and top plates). For test preparation, the powder is first sieved in \(z\)-direction into the tester. Then, it is precompacted in the same direction in order to homogenize the sample. After this, the sample is sheared with \( \dot{\epsilon}_x = -\dot{\epsilon}_y \), i.e., the sample’s volume remaining constant. The directions of shear are changed periodically. All experimental results presented in this paper have been obtained with carbonyl-iron powder with a median particle size of about 2 microns. This powder was chosen because its primary particles are perfectly round, in analogy to the particles used in the computer simulations.
2.2 Simulation Method

For the computer simulations we use a discrete element method, the Contact dynamics method [5]. In contrast to soft particle molecular dynamics, the volume exclusion of perfectly rigid particles and Coulomb’s friction law are implemented exactly [6]. The cohesion between the particles is included in a simplified model as an attractive force acting within a range leading to a force and an energy barrier to break a contact between two particles [7, 8, 9]. Furthermore, rolling friction between the round particles is implemented, also leading to the stabilization of large pores [7, 8]. We use two dimensional simulation because the influence of the torsional degree of freedom for a contact present in three dimensions is not well understood up to now.

All simulation results are given in the following units: As unit of length we take the particle radius $r$, as unit of mass $\rho r^3$, where $\rho$ is the mass density, and as unit of time the inverse strain rate $|\dot{\varepsilon}_x|^{-1} = |\dot{\varepsilon}_y|^{-1}$ (which was constant during the periodic kneading). In these units the simulation parameters were: The cohesion force between two particles, $F_c = 10^{22} \rho r^4 |\dot{\varepsilon}_x|^2$, the range of the cohesion force, $d = 10^{-4} r$, the friction coefficient, $\mu = 0.3$, and the rolling friction coefficient, $\mu_R = 0.2 r$ (which is the torque resisting the rolling motion divided by the normal force $F_n + F_c$ at the contact). We like to point out that for the typical values of the carbonyl-iron powder experiment [$r \approx 1 \mu m$, $\rho \approx 10^4 kg/m^3$ and $|\dot{\varepsilon}_x| \approx 10^{-4} s^{-1}$] our simulation parameter $F_c \approx 10^{-6} N$ is a reasonable value for the cohesion force between the particles.

The walls were cohesive, but frictionless, so that no tangential stresses could occur, as in the experiment. This guarantees, that the principal axes of the stress and the strain tensors were collinear.

3. RESULTS

For the two-dimensional simulations, we prepared two different kinds of initial configurations in the following way (cf. Fig. 2): One is a ballistic deposit [10], which was precompacted in the direction of the deposition [8] up to a volume fraction of $\nu = 0.42$. The other is an off-lattice DLA cluster [11] centered in a square box, and then precompacted biaxially to the same volume fraction, shrinking the square box symmetrically. Note that the first preparation procedure tends to lead to anisotropic, but homogeneous configurations, while the second one results in inhomogeneous, but isotropic configurations (the initial DLA cluster is essentially round and has a higher density at the center). This was done deliberately to demonstrate that even in this case both configurations reach the same (periodic) stress/strain relationship.

The precompacted systems are now used as initial configurations for the periodic shearing at constant volume. As in the experiment, the shearing was performed with constant logarithmic strain rate. The maximal strain was 36% (with
Figure 2: a) Initial configuration 1: Ballistic deposit. b) Initial configuration 2: DLA cluster. c) Configuration 1 precompacted uniaxially in the deposition direction. d) Configuration 2 precompacted biaxially (keeping the square symmetry). e) Configuration 1 after one cycle. f) Configuration 2 after one cycle.

Figure 3 shows the time dependence of the two principal stresses, averaged over 20 independent runs (with different, but similarly prepared initial configurations), including the precompaction. Moreover the data were smoothed by averaging them over a running time window of 5% of the period.

Clearly, the stresses are qualitatively different during the precompaction: For configuration type 1 (ballistic deposit) and uniaxial precompaction one gets anisotropic stresses, the larger stress belonging to the direction of compaction. For configuration type 2 (DLA-cluster) and biaxial precompaction both principal stresses increase in the same way, but when the square is deformed into a rectangular shape at constant volume (first 1/4 cycle), the principal stress in the expanding direction drops to a small positive value, while the other component stays large. This behavior agrees qualitatively with the experiment with the biaxial precompaction, Fig. 4.

The time dependent stresses within the first half cycle ($t/t_{\text{cycle}} = 0$ to $1/2$) in Fig. 3a are qualitatively different from the ones in all subsequent half cycles. Sim-
Figure 3: Simulation results: Principal stresses as functions of time during periodic deformation a) for configuration type 1 [volume constant for \( t/t_{\text{cycle}} \geq 0 \)], and b) for configuration type 2 [volume constant for \( t/t_{\text{cycle}} \geq -1/4 \)].

Figure 4: Experimental results: Principal stresses as functions of time during periodic deformation of biaxially precompacted carbonyl-iron powder.
ilarly the first 1/4 cycle \((t/t_{cycle} = -1/4\) to 0\)) in Fig. 3b is different. However, after these transients, it seems that the same stress pattern is repeated period after period, also in the experiment Fig. 4. In order to compare the stress patterns for the two cases, Fig. 3a and b, we averaged them over 9 repetitions. The results are shown in Fig. 5. Within the error bars the stress pattern for the samples obtained from uniaxially precompacted ballistic deposits is the same as for those obtained from biaxially precompacted DLA-clusters. Moreover \(\sigma_y\) simply coincides with \(\sigma_x\) shifted by half a period, as it should. This shows, that after the transients the stresses do not show any dependence on the preparation history. This is also reflected in Fig. 2e and f, which show that after one cycle the particle configurations

![Graph](image)

Figure 5: One period of \(\sigma_x\) and of \(\sigma_y\), averaged over all repetitions after the initial transient. a) For \(\nu = 0.42\) as in Fig. 3, b) for \(\nu = 0.63\). In both cases the curves for initial configurations of type 1 [full line] respectively type 2 [dashed line] coincide within the statistical errors.
are qualitatively indistinguishable.

Figure 5b shows the stress pattern for a higher solid fraction. Here the maximum strain compared to the square shape was 16%. Again one gets the same pattern in spite of the different types of original configurations and precompaction histories. However, the shape and amplitude of this pattern is completely different. This shows that the periodic stress pattern depends on the solid fraction.

4. CONCLUSIONS

We investigated the strain-dependent stress in a cohesive powder, which was periodically deformed at constant volume and strain rate. It was shown that contact dynamics simulations agree qualitatively with experiments on carbonyl-iron powder.

For the simulations two types of initial configurations were prepared. One had anisotropic stress and fabric tensors (uniaxially compacted ballistic deposits), the other was isotropic (biaxially compacted DLA-clusters), but both types of configurations had the same volume fraction. These initial configurations were then periodically deformed at constant volume. After a transient which lasts only less than a period, one obtains for both types of initial configurations the same periodic stress pattern. This leads us to the conclusion that periodic shear like steady state flow makes the powder forget its history quickly. It was also shown, that the periodic stress pattern depends on the solid fraction.

5. REFERENCES

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6. NOMENCLATURE

\( \epsilon \) – strain tensor, dimensionless
\( \sigma \) – stress tensor, Pa
\( L \) – size of the sample chamber, m
\( V \) – volume of the sample chamber, m\(^3\)
\( r \) – particle radius, m
\( \rho \) – mass density, kg m\(^{-3}\)
\( d \) – range of the cohesion force, m
\( F_c \) – cohesion force, N
\( F_n \) – normal force, N
\( \mu \) – coefficient of tangential friction, dimensionless
\( \mu_R \) – coefficient of rolling friction, m
\( \nu \) – volume fraction, dimensionless

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