Constitutive Model for Dry Cohesive Powders
with Application to Powder Compaction

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Abstract

Continuum theory-based constitutive models suitable for simulating the load response of dry cohesive powders have been critically reviewed. Based on a set of criteria, three candidate models for further investigation have been identified: Cam clay, Adachi and Oka model, and Krizek et al.'s endochronic theory based model. Since the modified Cam clay model is the foundation of many advanced constitutive models, it was chosen and applied to compaction load response of a dry cohesive powder (wheat flour). The constitutive equation parameters were determined using four types of conventional triaxial tests: drained, undrained, mean effective stress and isotropic tests. Based on these tests, the three Cam clay parameters for wheat flour were: 2.1 (slope of critical state line), 0.130 (loading index) and 0.022 (unloading index). Low pressure uniaxial compaction tests were performed with the powder in a thin-walled aluminum column. A finite element model (FEM), with the modified Cam clay constitutive model for dry cohesive powder's response, was used to predict the compression behavior. The FEM calculated values compared favorably with measured wall strains. The FEM predicted stress distribution in the powder mass identified troublesome regions with large shear stresses and tensile stresses.

1. Introduction

The handling and processing of powders (e.g. agricultural, ceramic, pharmaceutical, metal and mineral) constitutes a large volume, high value-added industry. Pressed products are often fabricated from a mixture of powders and additives. After mixing and granulation, powders are transported to a processing stage where they may be tabletted as medicines, pressed into shapes for use in manufacturing industry, or pelletized as feed. All phases of the powder industry, namely, storage, handling, and processing are operations that require precise knowledge of the behavior of powder systems to achieve the desired control. To model the behavior, powder mechanics must be understood and defined, yet the mechanics are most complex. Particular difficulties arise when attempts are made to predict the mechanical behavior of cohesive bulk solids that are under externally applied loads. For example, a cohesive powder in storage will form aggregates and clusters, resulting in irregular flow. For highly precise manufacturing operations, as the making of precision components, expensive medicines, or when exact percentages of ingredients are needed to ensure product quality, flow deviations may result in significant economic loss. Therefore, it is desirable to design equipment that processes the bulk solid in a known, predictable manner. For this capability, knowledge of the load-deformation behavior of powder systems is essential.

For near net shaping of advanced materials, compacting powders in a die is a preferred method because no liquids are involved during forming, organic binders and lubricants comprise only a small fraction of the total solids, i.e. ~10 vol%. The binders and lubricants are easily removed prior to densification (i.e. sintering), and high production rates of net shaped components are achieved. For example, ceramic ferrites, capacitors, metal gears, carbide cutting tools, and small, complex-shaped electronic components are manufactured by die pressing. For these applications dimensional precision, uniformity of pressed density, freedom from defects and pressed surface finish are critical measures of quality.
Because ceramic particles are \(< 1 \, \mu\text{m} \) diameter and elastic, the powder is combined with organic polymers (i.e., binders) to form free-flowing \(> 100 \, \mu\text{m} \) granules. Pressing pressures typically range up to 100 MPa but are often limited to lower pressures to reduce die wall wear by the abrasive ceramic powders. Deformation of the binder-ceramic granules is regulated by adjusting the polymer properties and content. In this manner, the elastoplastic ceramic powder is changed to a viscoelastic or viscoplastic material during pressing [1, 2].

Despite the extensive utilization of die pressing, there are few detailed studies about the powder mechanics involved in the manufacture of high quality components by pressing. The literature that is available is mostly focused on empirical relations between pressed density and pressure. These empirical relations are system specific and provide little fundamental insight about factors controlling the above measures of component quality. Recently, constitutive and finite element modeling have been successfully applied to metal powder modeling. However, only a very limited number of the reported studies have measured the elastoplastic or viscoelastic or elasto-viscoplastic constitutive model parameters and validated the finite element models for the same powder under compression. Nevertheless, it is this approach that will enable a complete understanding of the mechanics of powder pressing.

2. Literature Review

An objective of this paper is to first review potential models for their suitability for describing ceramic powder compaction.

Because a vast number of models exist, we outline the essential categories of constitutive models and then focus specifically on models for load-deformation behavior of cohesive powders. In the second part of the paper, we discuss the utilization of the Cam clay model for low pressure consolidation of wheat flour. By this example, we demonstrate the testing approach required for characterizing the compaction of a constrained powder. Finally the Cam clay model and the measured parameters are combined into a fourth-generation FEM model. The results of this FEM simulation are reviewed and the relevance of this approach to ceramic powder compaction discussed.

An engineering approach to predicting the mechanical behavior of powders is to develop a constitutive model, and then predict how the powder will deform or flow under load. Constitutive models are important because they allow a designer to quantitatively predict material behavior, then produce an effective design based on calculated numbers. Models can be divided into two groups: empirical and rational. Empirical models are developed by taking experimental data of a material under specific loading conditions and then statistically determining the equations that will closely match the data. Rational models apply physical laws to describe the stress-strain behavior of the material. These models are based on either microscopic or macroscopic scale parameters. Microscopic models consider each particle in a bulk solid as a distinct entity and then predict stress-strain behavior based on a distribution of interparticle forces. Macroscopic (continuum) models treat the bulk solid as a continuum or interacting continua and describe load-deformation characteristics of the material as a whole.

Each type of model has positive attributes. Empirical models accurately reflect the data on which they are based and are more easily obtained. Microscopic models consider force reactions on each particle and thus are very sound with respect to obeying laws of momentum and energy. Rational macroscopic models are usually easier to understand than microscopic models. They also satisfy, on the average, the mass, momentum, and energy equations, and can be quite accurate if the model assumptions are reasonable.

There are several empirical models in existence, a popular one being Kondner’s model [3], which represents axial stress as a hyperbolic function of axial strain. Kondner’s model, being consistent with other empirical models, produces good results for clay under axial loading. But for all of these models, as examples, Kuno and Okada [4] and Saleeb and Chen [5], only a specific loading condition can be predicted, and load-deformation behavior is not physically explained by actual material properties or by the laws of mechanics.

A large research initiative has been underway since the early 1980s to model the stress-strain behavior of granular materials at the microscopic level, focusing on particle-particle interactions and using the resultant of these interactions to predict the behavior of each particle and then the whole assembly of particles. Behavior at that level is a function of, among other factors, particle geometry, surface characteristics, friction forces, and material hardness. Most models treat particles as smooth spheres or cylinders to allow for a feasible prediction of particle contacts. Due to the very large number of particles in a given volume and complexity of these behavioral equations,
Rational models for bulk solids need to initially include whether the material is cohesive or cohesionless. As mentioned, cohesion adds difficulty to a model. The majority of cohesionless macroscopic models have been developed for sand [6-8]. Two of these models have been verified for wheat en masse [9, 10]. Clay soil is the predominant cohesive bulk material that has been modeled. Early models date back many years [11] with subsequent models increasing in sophistication [12].

The application of continuum models developed for clay soils, which are typically in the saturated state, to powders that contain relatively low levels of free water may be questioned. Most soil models use effective stresses, which are the principal stresses minus the pore liquid pressure, to predict behavior. In this sense the particle matrix in the soil responds to the applied load, a condition similar to what would be expected in a dry powder. A second point is that soils are considered bulk solids, as are powders, grains, and granulated products, and hence many researchers agree that theories derived for soils are assumed to be valid for any bulk solid.

Early plasticity models made use of the Mohr-Coulomb criterion [12], which defined the yield locus of material. The yield locus is still very applicable to industrial needs, as illustrated by the widespread use of the Jenike flow function in hopper design [13]. But yield loci curves are restrictive in the sense that their construction is dependent solely upon maximum and minimum principal stresses that induce yielding or flow of the material; i.e., stress-path information and the influence of the intermediate principal stress ($\sigma_2$) are not considered in their formulation. These limitations are serious since load-deformation of bulk solids is known to be a function of path and $\sigma_2$. Models using the Mohr-Coulomb criterion are herein termed “limit-state”.

The Drucker-Prager criterion [14], similar to the Mohr-Coulomb criterion, is a widely used plasticity theory to describe failure. Drucker-Prager defined a plastic yield surface in terms of three principal stresses, with elastic strain states inside the yield surface and plastic strain states on the yield surface; thus, the material is considered elastic-perfectly plastic. A state of stress outside the yield surface is unstable. A major shortcoming of the Drucker-Prager model is that failure is predicted during a volume increase (dilatancy), whereas many clays fail during densification (contractancy).

The critical-state concept [15] was introduced to soil modeling and states that a bulk material reaches a volume after which additional loading produces flow with no further volumetric change. In other words, when a bulk solid is loaded, particles rearrange, deform, fuse, and fracture, causing the volume to either decrease or increase. Decreasing volume means strain-hardening and increasing volume means strain-softening. In either case, the bulk solid reaches a stress state at which additional load may change the shape of a given mass but not its volume. This state is the critical state characterized by a critical void ratio or critical density. Before reaching the critical state, the bulk solid undergoes continuous yield states; hardening (or softening) after each yield state. There are limitations to this theory, which include the assumptions of isotropic hardening and associative flow; i.e., yield function is the same as the potential function (the function that describes the plastic potential surface, the surface normal to the plastic flow vector).

Another type of continuum constitutive model, different from the plasticity models, is the endochronic theory [16]. The endochronic theory considers stress-strain behavior in terms of a thermodynamic process instead of a mechanical process, as considered by plasticity theory. Viscoplasticity now becomes a function of internal state variables of which intrinsic time is one [17]. The state of the material depends upon past strain history through an internal clock.

Constitutive models developed specifically for powders were generally aimed at defining a flow function. Flow functions became popular after the work of Jenike [13], and as a result many gravity-flow hopper designs rely on the Jenike data charts. It is much more difficult to design hoppers through which cohesive powders flow because the conventional shear tests, such as Jenike, direct shear, and Peschl, do not adequately predict the tendency of the powder to aggregate and arch. In constitutive modeling, cohesion is an extremely difficult phenomenon to describe mathematically. Consequently, much research effort has concentrated on quantifying cohesion. Matchett [18] and Molerus [19] each explain cohesion as the sum of interparticle friction forces. Although their methodologies were different (Molerus used particle contacts and Matchett used the concept of friction bonds across a sphere plane) they employed the same basic principles, that is, they explain cohesion as interparticle attractive forces, then use a distribution
of forces over the material mass, and finally sum the distribution to get a single cohesion parameter. The cohesion parameter could then be used in the constitutive equations to predict a yield locus.

Both rational and empirical constitutive models have been presented and under rational models, microscopic and continuum models have been introduced. In a review of all model types, the rational continuum-type models are most attractive. A powerful continuum model has the potential to predict accurately powder behavior under a variety of loading conditions with a reasonable amount of testing and computation.

2.1 Discussion of models

A literature review of the key models for describing the load response of cohesive particulate materials identified two models that possess the most attributes crucial for the development of a generalized particulate mechanics model, namely: path-dependency, physically meaningful parameters, three-dimensional anisotropic capability, time-dependency, the capability to predict cyclic loading response, and the application of the model to an industrial setting. These attributes were first used as criteria for selecting models for preliminary review. Once a preliminary screening was done, the attributes mentioned were again used to rank the reviewed models in terms of their appropriateness for further testing of dry cohesive powders [20].

2.1.1 Modified Cam clay model

The review of continuum modeling reveals that a major distinction can be made between how models predict failure in powder systems. The limit-state models [21-23] were inadequate since they do not provide the required stress-path information.

Among the stress-path dependent models discussed, most [24-30, 31, 32, 33, 34] were based on the Cam clay model [15]. The Cam clay model was the first to use the critical state theory to describe yield criterion and hardening. The critical state concept [12] was used by Roscoe et al. [35] as an attempt to describe yielding of soils during triaxial tests. Roscoe et al. [35] relied upon earlier works of Hvorslev [36] and Gilbert [37].

The Cam clay model used three critical-state parameters: \( M \), \( \lambda \) (slope of critical-state line); \( \lambda \) (slope of loading path); and \( x \) (slope of unloading path). Parameters \( e_0 \) (initial void ratio), \( p_c \) (hardening parameter) and \( N \) (consolidation parameter) are also used in the elastoplastic constitutive equations. All parameters are derived from conventional triaxial tests. Based on numerous observations from implementation of the original model, a "modified" Cam clay model was formulated in which the original theory was intact except for the definition of dissipated work during plastic strain. Subsequently, the modified Cam clay model [12] redefined the flow function, which in effect altered the shape of the yield surface. Hereafter, only the modified Cam clay model will be considered. This model assumes the associative flow rule, i.e. the direction of the yield vector coincides with the direction of the plastic strain vector.

The modified Cam clay flow rule is given as:

\[
\frac{\delta W}{\delta \varepsilon} = (M^2 - \eta^2)/2\eta
\]

(1)

where \( \delta W \) represents differential strain, superscript \( p \) means plastic state, subscripts \( s \) and \( v \) refer to shear and volumetric states, respectively, and \( \eta \) is the ratio between mean effective stress \( p' \) and the deviatoric stress \( q \). For triaxial loading conditions, \( q = \sigma_1 - \sigma_3 \), where \( \sigma_1 \) and \( \sigma_3 \) represent principal stresses. The flow rule can be integrated to give the equation of the yield locus [12]:

\[
q^2 + M^2 p'^2 = M^2 p'p_c
\]

(2)

where \( p_c \) is, in effect, the size of the yield locus at critical state. A volume-pressure relationship has the following form [38]:

\[
V = N\lambda \ln (p)
\]

(3)

where \( V \) represents specific volume defined as \( 1 + e \), and \( e \) represents the voids ratio. The combination of Eqs. (2) and (3) gives a representation of the modified Cam clay yield surface in stress-space with axes of \( p' \), \( q \) and \( V \).

Given the definition of the yield surface in stress-space, the movement of material’s stress state can be predicted using constitutive equations. The derivation of these equations from the flow rule, the yield surface and the definition of plastic work is given in detail in several sources [12, 38, 39], and will not be repeated here. The strain components during flow are separated into elastic volumetric, plastic volumetric and plastic shear strains (elastic shear strains are assumed to be negligible). The constitutive equations are then defined by the following:

\[
\frac{\delta \varepsilon}{\delta \varepsilon} = \lambda / V [\delta p' / p' + (1 - x / \lambda)2 \eta \delta \eta / (M^2 - \eta^2)]
\]

(4)

\[
\frac{\delta \varepsilon}{\delta \varepsilon} = (\lambda - x) / V [\delta p' / p' + 2 \eta \delta \eta / (M^2 + \eta^2)]
\]

(5)
2.1.2 Adachi and Oka rheological model

The assumptions made in the development of Cam clay model were limiting: flow was considered associative; hardening was isotropic; plastic behavior was time-independent; intermediate principal stress was not relevant. Subsequent models removed one or more of the limitations to make the Cam clay model more general. Among the modified Cam clay models presented, only the one presented by Adachi and Oka [40] considered all of these limitations and, more importantly, defined the constitutive parameters in a lucid, physically analogous manner. This model will, therefore, be discussed in sufficient detail; that is, (1) the constitutive equation will be shown and the parameters explained; (2) any tests performed to determine parameters and validate the model will be discussed; and (3) a critique will be given of the model as used by the authors. For the sake of consistency, the original notation used by the authors will be used in this paper.

Adachi and Oka [40] defined the two yield functions in their model: static \( f_s \) and dynamic \( f_d \). The static yield function helps determine material parameters, and the dynamic function is used to define the stress-strain relation. Function \( f_s \) is the yield function described by the critical state theory and has the following equation:

\[
f_s = \sqrt{2J_2^{(s)}}/M^* \sigma_m^{(s)} + \ln \sigma_m^{(s)}
\]  

(7)

where \( J_2 \) is the second invariant of deviatoric stress tensor, \( M^* \) is a critical index parameter, and \( \sigma_m^{(s)} \) is mean effective stress. Superscript (s) denotes the static equilibrium state. Function \( f_s \) is the same as Eq (7) except that the dynamic state of stress is used. Yield functions were derived on the assumption of associative flow and the material being isotropic (anisotropy is mentioned as an appendix to the model [40]). The constitutive equation for the strain rate tensor during dynamic equilibrium of normally consolidated clay is expressed as:

\[
\dot{\varepsilon}_i = \frac{s_i}{2G} + \frac{[\varepsilon_0/(1+e)](\sigma_m^{(s)}/\sigma_m^{(s)})\delta_{ij} + ((1/M^*)\sigma_{m}^{(s)}\Phi(F)}{\sqrt{\sigma_m^{(s)}}}
\]

(8)

where

\[
\phi(F) = c_o \exp [m' \ln \sigma_{m}^{(s)}/(\sigma_{m}^{(s)})]
\]

Parameters \( c_o \) and \( m' \) are time-dependent material properties, \( G \) is shear modulus, \( s \) is swelling index, \( e \) is void ratio, \( s_y \) is deviatoric stress, \( \delta_{ij} \) is the Kronecker delta, \( \sigma^{(s)}_{m} \) is a dynamic hardening parameter, and \( \sigma^{(s)}_{m} \) is a static hardening parameter. Plastic volumetric strain is related to \( \sigma^{(s)}_{m} \) through the compression index \( \lambda \). On a practical level, Eq. (8) can be separated into parts and each term evaluated. The first term of Eq. (8) accounts for elastic deviatoric strain due to the presence of elastic shear modulus and the deviatoric stress tensor. The second term accounts for plastic volumetric strain since \( x \) is a volumetric parameter and the Kronecker delta \( (S_{ij})_s \) means only the volumetric stress components \( (\sigma_{ij})_s \) are non-zero. The rest of the terms account for the plastic deviatoric strain.

Determination of parameters follows a well-defined procedure, and all values are obtained using the triaxial test. Parameters \( \lambda \), \( x \), and \( e \) are obtained from consolidation and swelling tests. Shear modulus \( G \) and index \( M^* \) are obtained from triaxial compression tests, as was done in the Cam clay model. Parameter \( m' \) is found by taking the linear slope of the stress ratio \( q/\sigma_{m} \) versus \( \log \dot{\varepsilon}(t) \) for axisymmetric triaxial compression tests. For \( m' \), \( M^* \) must be known and \( \dot{\varepsilon}(t) \) (elastic strain rate) is neglected. Parameters \( c_o \) and \( \sigma^{(s)}_{m} \) can be combined into one parameter, \( C \), during plastic strain. Combining two parameters into one decreases the sensitivity of the equation, but is simpler to evaluate. Parameter \( C \) is determined using triaxial compression tests and plotting \( \varepsilon(t) \) versus \( q \), where \( \varepsilon(t) \) is neglected and \( m' \), \( M^* \), \( \lambda \), and \( e \) are as previously determined. An anisotropic consolidation parameter was discussed briefly by Adachi and Oka [40]. But more investigation is required to determine the potential of this parameter to represent the non-isotropic response of particulate materials.

Typical parameter values for Fukakusa clay, determined from triaxial compression and hydrostatic tests, are shown in Table 1. A plot of effective stress path for measured and predicted values at strain rate \( \varepsilon(t) = 0.0082\%/\text{min} \) is shown in Figure 1.
Table 1. Parameter values for Fukakosa clay [40]

| Parameter | Value |
|-----------|-------|
| $\lambda$ | 0.1   |
| $\chi$   | 0.02  |
| $M^*$    | 1.22  |
| $m'$     | 28.8  |
| $C_p$    | 0.72  |
| $G^*$    | 363 kPa |
| $C$      | $1.96 \times 10^{-9}$ s$^{-1}$ |

Fig. 1. Effective stress-path for Fukakosa clay at a strain rate of 0.0082%/min [40]. Symbols indicate actual data, the curve is predicted behavior, and the line is the critical state line. $q/q_{me}$ is normalized deviatoric stress and $\sigma_{me}/\sigma_{me}$ is normalized effective stress.

The constitutive model was applied in a two-dimensional consolidation analysis of a clay foundation during the construction of an embankment [33]. A finite element mesh was generated that replicated the clay embankment. Using the elastoplastic constitutive equations of Adachi and Oka [40] excess pore pressure versus total vertical stress and lateral displacement versus foundation settlement could be numerically calculated for the toe of the embankment via the finite element model. The FEM results were compared to an empirical model generated from actual field data. The FEM predicted a pore water pressure/vertical stress ratio of 0.75 for the first 33 days of consolidation and a ratio of 1.0 from 33 to 100 days, which almost exactly matched the empirical results. Similarly, the FEM predicted lateral displacements in the first 33 days of consolidation exactly matched the empirical displacements. The observed FEM displacements at 100 days consolidation were within 10%.

Adachi and Oka’s [40] elastoplastic model has several advantages. First, the plastic strain increment is a function of time; therefore, creep phenomenon can be predicted, and the model, most likely, can predict cyclic loading with some adjustments. Second, the model has the potential to include material anisotropy. Furthermore, the parameters used in the model ($\lambda$, $\chi$, $e$, $G$, $M^*$, $m'$, and $C$) have an easily understandable physical representation. An apparent shortcoming, however, is that of all the model parameters, $\chi$ alone seems to be able to account for cohesion. To explain, the unloading index $\chi$ is determined from the void ratio $e$ versus log $p$ (mean pressure) plots during the unloading cycle of consolidation. Since elastic strains after compression are plotted versus applied load, cohesive forces will have a tendency to keep the particles together and to resist any observed recovery to the original volume. No other parameter directly includes cohesive forces.
The final consideration is that all testing by Adachi and Oka [40] was mainly on saturated clays. The issue of water content has been mentioned as a concern, since the powders in question will generally not have high water contents. In a drained compression test, the capillary force from the pore water will tend to pull the particles together, thus functioning as a cohesive force. This would be equivalent to the interparticle cohesive forces, such as van der Waals and chemical forces, generated in a dry powder. For comparable undrained tests, void ratio change will have to be incorporated in the constitutive equations. The fact that Trassoras et al. [41] effectively used a clay model for tungsten-carbide powder is encouraging.

2.1.3 Krizek et al. endochronic model

Another model, namely the endochronic model developed by Krizek et al. [42] and used by Ansal et al. [43] is also a promising candidate. The model of Krizek et al. [42] focused on five important points: (1) recoverable and irrecoverable strains as soon as loading begins, (2) deformation is a function of stress history and strain rate, (3) volumetric stress components cannot be neglected, (4) normal strain has components due to volumetric and hydrostatic stresses and shear strain only due to shear stress, and (5) shear and bulk moduli vary with time (thus, they are functions of plastic strain, strain rate, and change in volumetric strain due to friction). The model assumes that the material’s resistance to shear stress is a first-order function of effective normal stress and interparticle distance. For consistency, notation and symbols used by the authors will be followed.

The constitutive equations for the model are based on two state variables, intrinsic time (Z) and a densification/dilatancy measure (λ). The intrinsic time increment, dZ, is a function of f (rearrangement measure), t (time), Z1 and τ1 (both material constants). Rearrangement measure f is determined from a coupled hardening/softening function (F). Determination of strain is a two-part process. The first part, which is a function of deviatoric stress, is to determine λ, which is a function of another densification/dilatancy function (L). Since λ neglects hydrostatic stress, strain includes bulk modulus (K) in the formulation for volumetric strain. The constitutive equations can be written as:

\[ d\varepsilon^d_{\gamma} = \frac{d\varepsilon^v_{\gamma}}{2G} + s_{\gamma} d\varepsilon^t_{\gamma}/2G \]

\[ d\varepsilon^v_{\gamma} = d\sigma^t_{\gamma}/3K + d\lambda \]

where

\[ dZ = f[f(F), t, Z_1, \tau_1] \]

\[ d\lambda = f(L) \]

and \( d\varepsilon^d_{\gamma} \) is incremental shear stress tensor, \( d\varepsilon^v_{\gamma} \) is incremental strain tensor, \( d\sigma^t_{\gamma} \) is incremental effective volumetric stress, \( G \) is shear modulus, \( d\lambda \) is the increment of \( \lambda \), and superscripts d and v mean deviatoric and volumetric, respectively.

To determine the internal variables \( Z \) and \( \lambda \) in Eq. (9), \( F \) and \( L \) have to be calculated from 14 constants: \( \alpha_1, \alpha_2, \alpha_3, \alpha_4, \beta_1, \beta_2, C_1, C_2, C_3, C_4, C_5, \tau_1, \tau_2, \) and \( Z_1 \). These are all determined using data of cycling, constant strain rate tests on clay isotropically consolidated under different effective stresses. A trial and error procedure was used to vary the parameters to reproduce these data curves. Parameter values were computed by Krizek, et al. [42] for New Field clay and are given in Table 2. These values were then used to obtain the predicted curve of axial strain versus number of cycles shown in Figure 3, and also for the predicted curve of pore pressure versus number of cycles shown in Figure 4. The close agreement of predicted and measured values show the ability of the model to handle cyclic loading of clay.

The endochronic model of Krizek et al. [42] has many attributes. Model predictions correlate well with observed stress-strain behavior. The constitutive equations are time-dependent since time is internal to the equation. Increases of pore pressure and axial strain owing to cyclic loading were predicted with fair accuracy. A major drawback of the model is that none of the parameters can be directly linked to cohesion since the parameters do not have any simple physical representation. Also, the method the authors used to determine parameters is not clear at this point, except that a statistical approach was used.

Table 2. Parameter values for New Field clay [42]

| Parameter | Value |
|-----------|-------|
| \( Z_1 \) | 0.0155 |
| \( \alpha_1 \) | 4 |
| \( \alpha_2 \) | 25 |
| \( \alpha_3 \) | 10 |
| \( \alpha_4 \) | 0.75 |
| \( \beta_1 \) | 15 |
| \( \beta_2 \) | 2 |
| \( C_1 \) | 2.2 |
| \( C_2 \) | 1500 |
| \( C_3 \) | 0.25 |
| \( C_4 \) | 4500 |
| \( C_5 \) | 35,000 |
Both of the endochronic theory based models discussed in detail [42, 43] have the potential to predict the load response of selected food and agricultural products over a limited load range. References are cited [9, 10] where sand models were used for wheat. Feda [44] has published yield loci data on wheat, sugar, and lentils, and these fall in a form consistent with sands and clays. Finally, triaxial testing of sugar and flour show experimental trends similar to clays [45]. All of these results encourage further exploration of the models reviewed as candidates to be applied to food and agricultural materials.

The Cam clay model is a basic soil model upon which many other constitutive models are based. It provides adequate results for predicting soil behavior in conventional triaxial tests as well as in consolidation analysis and for determining foundation strengths. In addition, the model is relatively easy to use and understand. For these reasons, the Cam clay model was a logical starting point for application of soil models to dry cohesive powders under compaction.

3. Experimental Procedure

The modified Cam clay model requires the following tests for parameter determination [12]:

(1) Three conventional triaxial compression (CTC) tests, with the pore line ported to atmosphere, each at different confining pressures. These tests are referred to as drained tests.

(2) Three constant volume, triaxial compression tests, each at different confining pressures. These tests are referred to as undrained tests.

(3) Three constant mean effective stress (MES), triaxial compression tests, each at different mean effective stresses. The tests are referred to as MES tests.

(4) One hydrostatic triaxial compression (HTC) test with three unloading-reloading cycles. This test is referred to as the isotropic or HTC test.

Since all tests were designed for soils, particularly in saturated states, confining pressures are meant to be applied using a liquid medium. Given that dry powders are typically in unsaturated states and have low moisture contents when compared to saturated soils, it was considered more appropriate to use air as the pressurizing medium, as performed by Zhang et al. [46] and Bock et al. [47]. Specimen pore pressure was recorded and converted into corresponding volumetric change in specimen pore space. A schematic of the conventional triaxial tester used in this study is shown in Figure 5.

Drained tests are fairly straightforward in analysis and procedure. The triaxial specimen is axially compressed along the longitudinal axis via the Instron crosshead, which moves at a constant displacement rate and houses the load cell. Movement of the load cell was accurate to within 10⁻² mm, which is within 10⁻⁴% of the specimen height and sufficiently accurate for this investigation. A constant confining pressure is applied in the chamber (σ₃ = constant). Confining pressure was measured by a transducer (Omega model PX612-100GV, accurate to 0.7 kPa). Pore air is ported to the atmosphere, thus draining the specimen. To measure the volumetric change for the
drained tests, the pore line was ported to atmosphere after certain increments of pore pressure (0.5 kPa) accumulated. These increments were then summed at the end of each test. Drained tests were run at three confining pressures, as shown in Table 3.

Undrained tests posed particular problems, since, by definition, they are constant-volume tests. For dry, compressible powders, constant volume is not possible, since there are no saturated states as with soils. For undrained tests, the best that could be done was to close the pore line to have constant-mass tests, and monitor the pore pressure change. Confining pressures used for the undrained tests are shown in Table 3.

The mean effective stress (MES), $p'$, is defined as:

$$p' = \frac{(a_1 + a_2 + a_3 - 3u)}{3}$$

which becomes for triaxial conditions:

$$p' = \frac{(a_1 + 2a_3 - 3u)}{3}$$

(10)

where $u$ is the fluid pressure developed in the pore spaces of the powder. For the MES tests, the effective stress was held constant by altering the confining pressure, $a_3$, in proportion to the increase in $a_1$ supplied by the crosshead and the change in pore pressure. The mean pressures tested are shown in Table 3.

4. Finite Element Model

The FEM used was the fourth generation model in a series of useful models [48-51]. There are three components of the discretized system: 1) the stored material; 2) the structure (wall) surrounding the material; and 3) the interface between wall and material.

It is important to note that the global stiffness matrix, $[K]$, depended upon the strain and stress states of the elements. Strain and stress were likewise dependent upon the displacements of the nodes of the elements. In effect, $[K]$ was a function of global displacement vector $\{U\}$, more adequately expressed as $[K(\{U\})]$, and was non-linear. For the compaction cylinder, surcharge pressure, applied to the top of the powder mass, was divided into incremental pressures. Element force vectors $\{f\}$, were determined for an increment of pressure for each powder element, then inserted into the global vector $\{F\}$. Finally, an iterative routine was used to solve for $\{U\}$, (Figure 6) to converge the residual of the vector sum $[K]\{U\} - \{F\}$ below a tolerance value. If the residual was below tolerance, then a solution for $\{U\}$ was assumed and the program proceeded to the next pressure increment. If, however, the residual was not below tolerance, $\{U\}$ was changed (which also changed $[K]$) with the aim of reducing the residual below the tolerance. The number of iterations needed for convergence, in general, was less than seven, and so the maximum number of iterations was set to 50. If 50 iterations were exceeded, the program quit, meaning a force balance could not be reached. The solution routine was repeated for each pressure increment. A detailed discussion of element stiffness matrices, element property matrices, i.e., wall, powder and interfaces, is reported by Tripodi et al. [52].

Because a compressible powder was modeled (wheat flour), large deformations had to be accounted for during compaction. Deformations of 30% of the original height of the powder compact were expected. Such large deformations would result in nonsense terms generated for total strains (greater than 100%) if a domain change was not incorporated into the FEM. In order to simulate the compaction test in the FEM, four guiding rules were followed: i) incremental strain was calculated for all elements, and, if the total became too great ($>0.2\%$), the nodes were released from the fixed locations and the geometry allowed to adjust to the strain; ii) the entire mesh was reconfigured when the criterion in step (i) required it; iii) once a mesh (domain) change occurred, initial
stress and strain states, as well as voids ratios, were calculated for the new mesh; and, iv) stress and strain information was stored for all domains used. Based on these guiding rules, the FEM checked all powder element vertical strains \( (e_{zz}) \) to see if any exceeded a level of 0.2\% strain. Once a mesh change had been implemented, element stresses, strains and centroid coordinates were stored. Since the FEM was the type of model that applied incremental loads to the top boundary, vertical displacements for the top layer nodes were different. These vertical displacements for nodes at the top layer of the mesh were averaged to find a single value for vertical domain change. A radial domain change was subsequently calculated using the radial displacements of the wall elements. Once radial and vertical changes were calculated, the dimensions of the domain were adjusted and a new mesh configured to represent the powder mass. For the new mesh, any differences in nodal displacements at the top loading boundary were corrected by nullifying all initial displacements.

5. Compaction Cylinder Tests

It was important to test the constitutive models in an application that reflected common industrial applications. To achieve this aim, a compaction cylinder was constructed that used an INSTRON loading device to axially compress a flour sample prepared in an aluminum cylinder. A 60.6 mm diameter \( \times \) 121 mm height, 1 mm wall thickness, aluminum cylinder was built. This cylinder was instrumented with 45° rosette strain gages (Micromeasurements, Model \# CEA-13-125UT-350). These rosettes provided strain measurements in three orientations – hoop, 45°, and vertical – for the cylinder at each location. Gages were arranged vertically in three strips and attached to the cylinder at 120° spacing. Along each strip, rosettes were attached at three levels: TOP level at 89 mm from cylinder base, MIDDLE level at 57 mm from base, and BOTTOM level at 25 cm from the base. A schematic of the test cylinder with dimensions and gage locations is shown in...
6. Test Results and Comparison with Finite Element Modeling

6.1 Compaction test results

The Cam clay model was developed with the goal of predicting failure in soil masses due to foundation or embankment loads; typical applications of soils. For these conditions, water can seep through the soil mass, and soil particles rearrange and settle in reaction to the applied loads. Loads may become quite large, and the soil matrix experiences elastic stress states followed by elastic-plastic states. Finally, if loads become too great, the material fails in shear and yields. Yielding continues until the critical state is reached. Stress-strain predictions under these conditions can be readily handled.

Under the compaction loading, however, there were different constraints put on the particulate material. The particulate mass was confined in all directions by the cylinder wall and the cylinder floor. These constraints forced the material to stiffen during load- ing. The Cam clay model does not accommodate an increase in stiffness, nor does it allow any dependence of its parameters on stress. As shown in Table 4, all parameters are fixed values determined from specified triaxial tests [53] and do not vary with applied stress levels. Compaction loading, unlike triaxial loading, stiffened the wheat flour during strain, and the simple definition of some parameters, particularly the elastic parameters, were insufficient at high stress levels.

6.1.1 Dependence of Cam clay parameters on stress path and pressure

All of the Cam clay parameters were determined using the set of triaxial tests shown in Table 3. The confining pressures and mean effective stresses used for these tests were chosen based on test experience using wheat flour. By definition, Cam clay parameters do not vary with applied stress, and the pressure levels used to determine the parameters were considered adequate. It was shown in the data used to determine material parameters [53] that parameter values changed at different pressure levels, but mean values were chosen to predict load curves, with good results.

However, chosen pressure levels, and the consequent parameter values that were determined, had a significant impact on the FEM termination level. For example, parameter $M$ was changed from the value of 2.1, calculated using the triaxial data, to a lower value of 1.1, the lower value meaning an increase in strength in the material. The FEM simulated stress states to the desired level of 200 kPa. Originally, for $M = 2.1$, the FEM terminated at a pressure level of 46.2 kPa. Thus a stiffer material resulted in the FEM running to a higher pressure. Similar results occurred when altering $\lambda$, $\kappa$, and $\rho_c$, as well as for elastic parameters $E$ and $K$. If calculated parameter values were altered in ways that stiffened the material, the FEM simulated powder response greater than

![Figure 7](image-url)

**Fig. 7.** Schematic of compaction cylinder with strain gages

| Parameter | Value |
|-----------|-------|
| Critical state | |
| $\lambda$ | 0.13 |
| $\kappa$ | 0.022 |
| $M$ | 2.1 |
| Elastic | |
| $E$ | 4.9 MPa |
| $K$ | 4.2 MPa |
| Initial | |
| $\varepsilon_0$ | 1.06 |
| $\rho_c$ | 45 kPa |
the base value of 46.2 kPa. Likewise, softening the material caused a quicker program termination. Inspection of the stress states and property matrices during these numerous FEM simulations made it clear that one or more elements had reached a critical state. Elements at the critical state cannot accept more load. But the powder mass, being restrained by the aluminum cylinder, increased stress in the powder mass, which attempted to stiffen the material. A force balance was not reached, and the program terminated. Note that it would require only one element to reach the critical state and create a force imbalance, causing program termination.

Vertical strains in the compaction cylinder, predicted using the Cam clay model in the 4th generation FEM, agreed well with average measured values for surcharge pressures between 0 and 59.4 kPa surcharge [52]. However, predicted hoop strains in the compaction cylinder were somewhat satisfactory only for the BOTTOM level of strain gages [52].

### 6.2 Predicted stress distribution in the powder compact

The mesh used for all FEM simulations, showing respective element numbers, is illustrated in Figure 8. Mesh size (number of elements) was determined by running preliminary analyses on the FEM. The smallest mesh size (least number of elements) was chosen that provided the desired predictive capabilities.

**Figures 9 and 10** give the horizontal-plane and vertical-plane stress distributions, respectively, in the compaction cylinder using the FEM simulation, and for a surcharge pressure of 20 kPa. Horizontal-plane stress distributions are given with respect to sections cut in the compaction cylinder at planes corresponding to the TOP, MIDDLE and BOTTOM gage locations. In Figure 8, TOP and BOTTOM planes correspond, respectively, to the elements at the 87.2 mm and 21.5 mm heights displayed. The MIDDLE plane corresponds a level between the 47.6 mm and 60.8 mm heights in **Figure 8**. In effect, the horizontal-plane stress distributions show hoop and vertical stresses as they varied moving outward from powder mass center to the wall, along the TOP, MIDDLE and BOTTOM planes. For labeling purposes, hoop-direction stress for the TOP level will be termed $T_H$; vertical-direction stresses for the TOP level will be $T_V$; MIDDLE hoop-direction stresses will be $M_H$; MIDDLE vertical-direction stresses will be $M_V$; BOTTOM hoop-direction stresses will be $B_H$; and BOTTOM vertical-direction stresses will be $B_V$. With regard to the FEM output, all stresses were applied at element centers in the finite element mesh.
Therefore, locations of the element centers form the abscissa of the plot in Figure 9. Stress points at the element centers are linearly connected to approximate the distribution between the distinct elements.

Vertical-plane distributions are given with respect to section cuts along planes parallel to the cylinder centerline. These planes correspond to the radial values of 3.6 mm and 25.3 mm shown in Figure 8. These radii refer to the powder elements along the centerline of the compaction cylinder (3.6 mm) and the elements nearest the wall interface (25.3 mm). In effect, vertical-plane distributions show how powder stresses varied throughout the depth of the compaction cylinder. Again, stresses were applied at element centers; the locations form the ordinate of Figure 10. For labeling purposes, vertical-plane distributions for hoop-direction stresses near powder mass center will be termed C,H; vertical-direction stresses near center will be C,V; hoop-direction stresses near wall will be W,H; and vertical-direction stresses near wall will be W,V.

Vertical stress distributions at TOP and MIDDLE (Figure 10) were in agreement with the findings of Thompson [54]. Actually, Thompson proposed a parabolic stress distribution for a ram-on-compact. It is apparent that if stresses of TV (Figure 10) were curve-fit, a parabolic curve would be appropriate. At BOTTOM, vertical and hoop stresses were essentially zero since most of the load was taken out by the walls at BOTTOM. In Figure 10, hoop stresses at the wall tell an involved story. From 60 mm above cylinder base to the top loading boundary, hoop stresses at the wall are tensile. More comprehensive visual aids regarding the distribution of stresses in the powder mass are given in Figures 11 and 12. Two directions of stresses are illustrated, shear ($\tau z$) and hoop ($\Theta\Theta$), in Figures 11 and 12, respectively. Contour lines, i.e. constant stress lines, are given in intervals of 2 kPa. Figure 11 illustrates how predicted shear stresses in the powder mass accumulated near the intersection of the loading boundary and the retaining wall, which is also the intersection of the powder-wall interface and the top of the powder mass. The interface friction component pulls greatest at the area of load concentration (top of the powder mass) and causes the greatest distortion of powder elements (high shear stresses) near the wall. The result was a reversal in direction of hoop stresses, from compressive to tensile stresses near the wall of the cylinder (Figure 12).
7. Conclusions

This study has shown that the Cam clay model is adequate for predicting the powder’s stress state for low loading conditions. However, for the high loading conditions characteristic of ceramic compaction, pharmaceutical tabletting, or pelletizing modifications in the model will be required to adequately handle the characteristic large deformations, and high shear and tensile stresses of these processes. Another problem identified by these studies is that constitutive parameters are not constant over a wide range of loading conditions. Also, the prediction capability of the FEM is sensitive to definition of the powder-wall interface elements. However, by identifying these issues, it appears that the Cam-clay model can be applied to higher stress situations once the necessary modifications are incorporated into the testing program (i.e. constitutive parameter determination) and the model refined and expanded.

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Fig. 12. FEM generated Iso-hoop stress contours at 20 kPa pressure.
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