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CERAMIC POWDER COMPACTION

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ABSTRACT

With the objective of developing a predictive model for ceramic powder compaction we have investigated methods for characterizing density gradients in ceramic powder compacts, reviewed and compared existing compaction models, conducted compaction experiments on a spray dried alumina powder, and conducted mechanical tests and compaction experiments on model granular materials. Die filling and particle packing, and the behavior of individual granules play an important role in determining compaction behavior and should be incorporated into realistic compaction models. These results support the use of discrete element modeling techniques and statistical mechanics principals to develop a comprehensive model for compaction, something that should be achievable with computers with parallel processing capabilities.

INTRODUCTION

In the manufacture of advanced ceramics, optimum properties are achieved by defining and developing processes to produce a target microstructure, and by controlling processing to minimize the concentration and scale of the defects in the finished product. To manufacture ceramics with reliable and reproducible properties, it is imperative to understand and control process-microstructure-property relations during the various stages of processing.

Consolidation is an important part of ceramic processing that comprises processes ranging from green body forming to thermal consolidation (e.g., sintering). The most common methods of forming for high volume components are uniaxial pressing and isostatic pressing.[1-4] Both processes use granulated powders because of their improved flowability and ease of handling

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compared to ungranulated powders. Granules are generally produced using a spray drying process.[5]

Particle or granule packing may be the single most important physical characteristic of a powder compact. In closely-packed powder compacts, densification during sintering occurs faster and more uniformly, to higher end-point densities, and with less overall volume shrinkage. Uniform particle packing in a green compact ensures uniform densification and shrinkage during sintering. Packing heterogeneities due to the presence of agglomerates (i.e., densely packed particle clusters) or pores due to poor packing are undesirable as they produce defects in the final sintered microstructure that reduce the overall reliability of the component. Density gradients created during forming (e.g., due to die wall friction) are undesirable as they promote differential or heterogeneous densification within the ceramic body, which often results in warping and cracking during sintering.[6,7]

Homogeneous and heterogeneous densification are illustrated and compared in Fig. 1.[6] Homogeneous densification, which is achieved with uniformly packed powders, results in a volume decrease without a change in shape. A lower green density compact experiences a greater overall volume shrinkage on sintering to the theoretical density. Heterogeneous densification, which occurs when density variations exist within the green compact, also results in a volume decrease; however, lower density regions can be physically constrained from sintering to theoretical density, resulting in shape distortion and incomplete densification.[1]

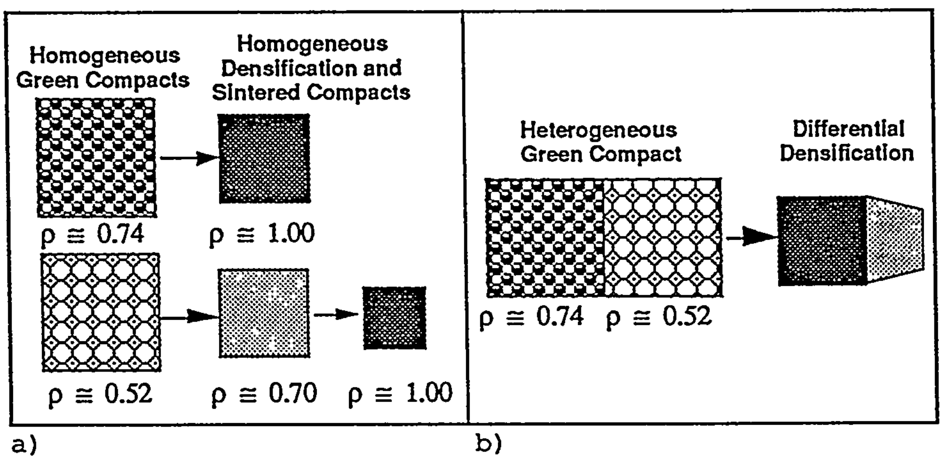

Fig. 1 Homogeneous densification during sintering of uniform density powder compacts with two different starting densities. b) Differential densification during sintering as a consequence of density heterogeneities in the green powder compact.

During press forming the die filling and compaction operations ultimately determine the size, shape, and properties of the sintered product.[8] The degree of density heterogeneity that exists after compaction depends on the characteristics of the powder, the initial filling of the die by the powder, the shape of the die, the forming pressure, and how the pressure is applied. During compaction, externally applied pressure is used to promote particle rearrangement, granule deformation, and consolidation of the particulate assembly. Particle coordination number, green density (i.e., the bulk density of the compact prior to sintering), and compact strength increase with increasing pressure during compaction,[9-12] while the volume and size of the porosity in the compact decrease.[13,14] Generally a finer and more uniform pore structure in a higher green density compact contributes to more uniform densification and higher end-point densities on sintering.[15,16]

Economically, a net-shape consolidation process is highly desirable, whereby compact density and densification are well controlled, uniform, and extremely reproducible. Net-shape processes not only minimize waste, but also offer the potential to eliminate expensive finish machining operations. Currently, the industry standard for as-sintered tolerance is 1.5%. Many ceramics are routinely sintered to within 1% tolerance, with some specialty cases down to 0.25%. An as-fired tolerance of 1% is adequate for many stand-alone ceramic components; however, more precise fitting components (e.g., engine components) typically require finish machining to size. Machining operations can easily double the cost of the finished part. An as-sintered tolerance of <0.5% can minimize and possibly even eliminate the finish machining operation, which would lower the total manufacturing cost, and could make ceramic components more cost competitive with metal and polymer components. The key to achieving a net-shape compaction process is to minimize or eliminate macroscopic density gradients in the particulate assembly during die filling and compaction.

To avoid the deleterious consequences of heterogeneous densification, ceramic fabrication processes must be designed and controlled with the intent of optimizing green density while minimizing both macroscopic density gradients (due to pressure gradients during compaction) and microscopic density variations (due to the presence of agglomerates and pores). Scientists from Sandia National Labs are currently working to develop and apply science-based computer and process models to better understand and control powder compaction. The objective of this paper is to provide a brief description of some of the components of this ongoing work. The first section highlights techniques that can be used to characterize macroscopic density variations in pressed powder compacts. The second section describes the evolution of efforts to model powder compaction, starting with Jannsen in 1895,[17] to what we hope to accomplish with our current and future computing capabilities. The third section describes experiments conducted on a spray dried alumina powder. The final section describes the results of tests on individual model granules and relates their mechanical behavior to their overall compaction behavior.

DENSITY GRADIENT CHARACTERIZATION

To test, refine, and validate models for powder compaction, model predictions of density distribution within a powder compact must be compared to measured macroscopic density gradients in a powder compact. The powder compact can be assessed on the basis of the overall fractional density of the compact, where fractional density, ρ/ρ_{th}, is the bulk density, ρ, divided by the true or theoretical density, ρ_{th}. This however provides only the average density of the compact. Mercury porosimetry provides a quantitative method to characterize the bulk density of a powder compact and its porosity, including pore volume and pore size distribution;[18,19] however, spatial density information is not easily obtained.

Some quantitative means of characterizing spatial density and mapping density gradients in a powder compact include scanning electron microscopy coupled with quantitative stereology,[18,20,21] ultrasound, x-ray radiography, computed tomography (CT), and magnetic resonance imaging (MRI).[22,23] Ultrasound, x-ray radiography, and image analysis have all proven useful for characterizing density gradients in ceramic powder compacts.[22]

Sandia research has determined that ultrasound has a respectable spatial resolution of 0.25 mm and density resolution of 0.5%, and produces the most quantitative density contour maps of pressed powder compacts.[22] Results from a rectangular slab cut from a cylindrical compact formed by pressing at 138 MPa (20 ksi) are shown in Fig. 2. This map shows the highest densities in the top corners (in contact with the single action pressing punch), the lowest in the bottom corners, and radial gradients from a high density at the edges to a lower density along the cylinder axis. Although the density gradients due to die wall friction might be expected to be symmetric about the cylinder axis, there are noticeable differences between one side and the other. This observation will be discussed further later in this paper.

X-ray radiography has slightly lower resolution than ultrasound, but may be preferable when cost is an issue, and/or when many samples need to be analyzed. Both ultrasound and x-ray radiography should be readily adaptable to a manufacturing environment (e.g., for product quality and assurance testing). Additional details about ultrasound density measurements and x-ray radiography, along with information on other techniques for characterizing ceramic compacts can be found in Ref. 22.

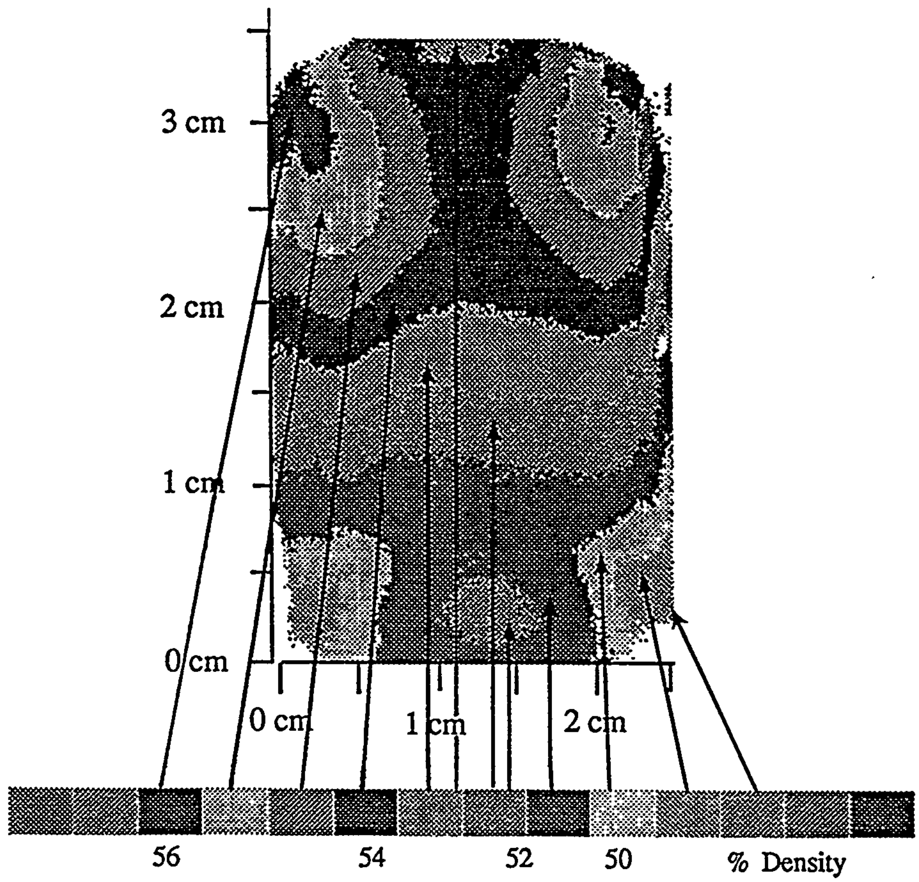

Fig. 2 Results of ultrasound velocity measurements showing the density profile in 3.5 cm high slab cut from a uniaxially pressed cylindrical compact of spray dried alumina. The central axis of the sample is at the position of approximately 1.25 cm on the horizontal scale.

COMPACTION MODELING

Models can provide a more comprehensive understanding of the compaction process, and information on the control necessary to achieve a uniformly high green density. One common modeling approach is to develop and use empirical expressions that relate compact density to forming pressure.[23,10,24] However, most empirical pressure-density relations lack a physical basis, and consequently, provide little or no information on the mechanisms of compaction that must be understood and controlled to achieve uniform densification.[10] These models also have little or no predictive capability to describe systems with different material characteristics. Furthermore, the expressions provide no information on the density gradients within the compact, only the average density.[23,24]

Despite their deficiencies, empirical pressure-density relations can be used in combination with mechanics-based numerical models that describe the state of stress in a particulate body during compaction, to map density and density gradients in pressed powder compacts.[25,26] Efforts devoted to predicting pressure gradients due to die wall friction date back as far as 1895, when Janssen predicted a stress gradient from the top to the bottom of a grain silo.[17] A recent analysis of powder compaction models by scientists at Sandia National Laboratories has determined that, while existing models can be used to predict general trends in stress state and density in a cylindrical compact, they do not accurately predict the stress distribution in a pressed body.[23,24]

Some major deficiencies of the existing models include: 1) they incorrectly assume/predict a constant stress state and density along the central axis; 2) an accurate analytical expression for the radial stress state is lacking (i.e., an empirical expression is generally used); and 3) the stress variation in the compact is generated solely from die wall friction (i.e., interparticle interactions are not addressed). In a recent study by Aydin et al. the third deficiency was postulated to be responsible for the discrepancies between their measured density gradients and those predicted by finite element analyses using a constitive model of compaction.[27] The model predictions showed the expected axial gradients along the die wall; however, the density profile along the die center did not agree with the observed trends.

Recent experiments have demonstrated that density gradients are also present in isostatically pressed powder compacts (Fig. 3), where no die wall frictional forces are present, suggesting that interparticle frictional forces do indeed play an important role in the compaction process.[24]

It has been postulated that a comprehensive compaction model requires an analytical equation of state to relate the volume of a powder compact to forming pressure. The development of such an equation requires information on the mechanisms of powder compaction and information on how individual granules deform relative to the particulate assembly during compaction. Sandia scientists are currently exploring these issues. Because of the complexity of a model that must keep track of large numbers of granules, computers that have the capability to perform parallel processing are necessary. We have begun to utilize this capability to model compaction. Our first simulations have included dropping lead spheres into a square cross section container, allowing them to bounce and roll until reaching their equilibrium configuration, and then compacting them. Simulations were run with 16 and 125 lead particles; the results for the smaller number of particles are shown in Fig. 4. Admittedly, this small number of particles is not representative of a production compaction process; however, it does provide a starting point for simulation code modifications.

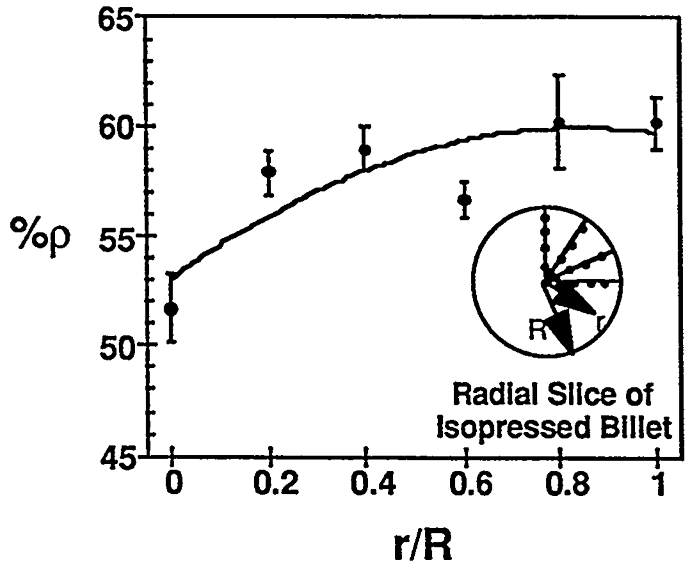

Fig. 3 Density as a function of radial distance for a disk cut from a uniaxially pressed cylindrical compact of spray dried alumina.

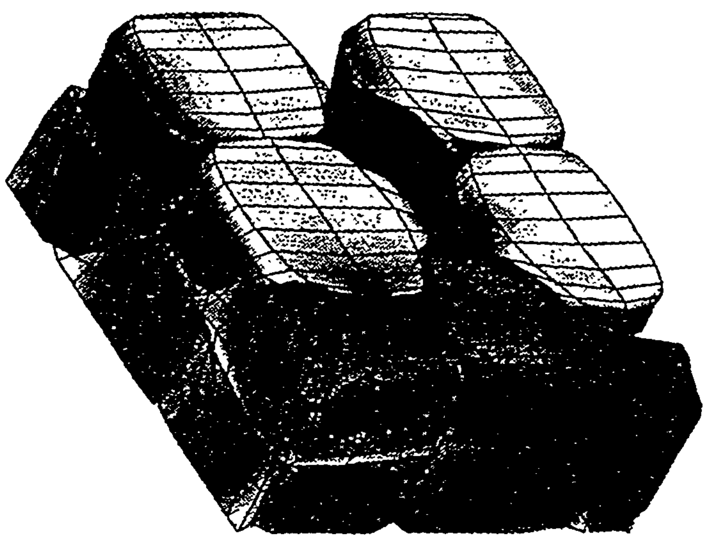

Fig. 4 A discrete element model simulation of the filling and compaction of lead spheres in a square cross section die.

The compaction process is further complicated by the statistical nature of the packing and the properties of the granules themselves (discussed in next section and Ref. 28). Our research indicates that granule deformation is a statistical process, suggesting that it may be possible to more accurately predict ceramic powder compaction using probabilistic models.[28] These results are intriguing as volume-based probabilistic processes have been used with some success previously to model powder compaction.[29] Further support for a probabilistic modeling approach has come from a recent collaborative effort between scientists from Sandia and the University of New Mexico. A volume-based statistical mechanics model for ceramic powder compaction has been developed whose predictions compare favorably with experimental results of density vs. compaction pressure.[30] Ultimately a realistic model for compaction will need to account for both discrete particle interactions and for the statistical variation in granule properties.

COMPACTION EXPERIMENTS

Although pressure-density relationships are only valid for the average density of the compact, they can still be useful for evaluating macroscopic compaction parameters such as the density after pressing to a specific pressure. To evaluate powder compaction, spray dried alumina powder was poured into a cylindrical die and uniaxially pressed to 138 MPa.[8] Measured displacement and the mass of powder were converted to percent theoretical density and plotted as a function of the compaction pressure. Corrections due to load-frame and die compliance were made.[31] The initial aspect ratio, H_i/D (where H=height and D=die diameter), was varied between 0.15 and 5.0. Die diameter was varied between 3.2-25.4 mm (1/8 and 1"). Tests were conducted with a displacement rate of 0.5 mm/min. No die lubrication was used. The general shape of the compaction curve is shown in Fig. 5. The results for various die diameters are shown as green density vs. aspect ratio in Fig. 6.

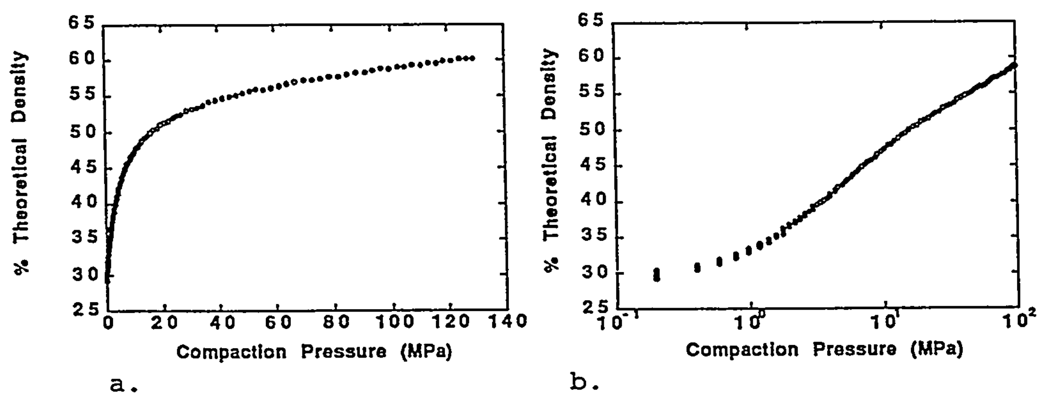

Fig. 5 a. Relative density vs. compaction pressure for spray dried alumina granules.
b. Relative density vs. log compaction pressure for spray dried alumina granules.

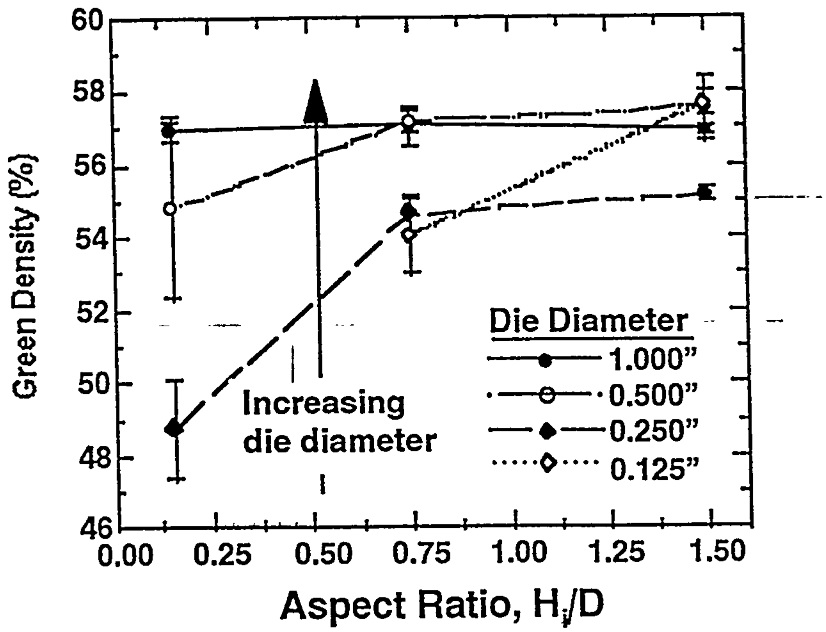

Fig. 6 Green compact density vs. aspect ratio for different die diameters.

The largest die diameter shows no effect of aspect ratio on compaction. For the smaller dies, compact density increases as the aspect ratio increases, with the smallest die producing the lowest density at a given aspect ratio. There are two results to explain: why is the density significantly lower for the smaller diameter die and why does density increase with aspect ratio?

The fill density (prior to compaction) vs. the aspect ratio shows again, that there is no effect of aspect ratio for the largest die (Fig. 7); however, the fill density increases with aspect ratio for the smaller dies. Similar results were obtained in a study by Bocchini.[32] Clearly there is an effect of die filling that produces lower densities for smaller die diameters. This effect is likely related to the size of the granules relative to the die diameter, and the low packing density at the die wall relative to the body of the compact (Fig. 8). The effect of the die wall is more pronounced with smaller die diameters because the volume fraction of particles in the die wall region is larger for smaller dies.

Higher aspect ratios likely produce higher densities because the mass of the granules themselves increases packing density. Despite the fact that compaction forming pressures are significantly larger than the pressure due to the mass of the powder, the influence of filling density persists throughout the compaction process. That is high green densities are achieved with higher fill densities. This effect may be due to the fact that there is a distribution of particle (granule) sizes, and smaller granules have an opportunity to fill the interstices between larger granules during filling; however, once compaction begins granule rearrangement becomes severely limited. As such lower densities that are introduced during filling persist throughout compaction.

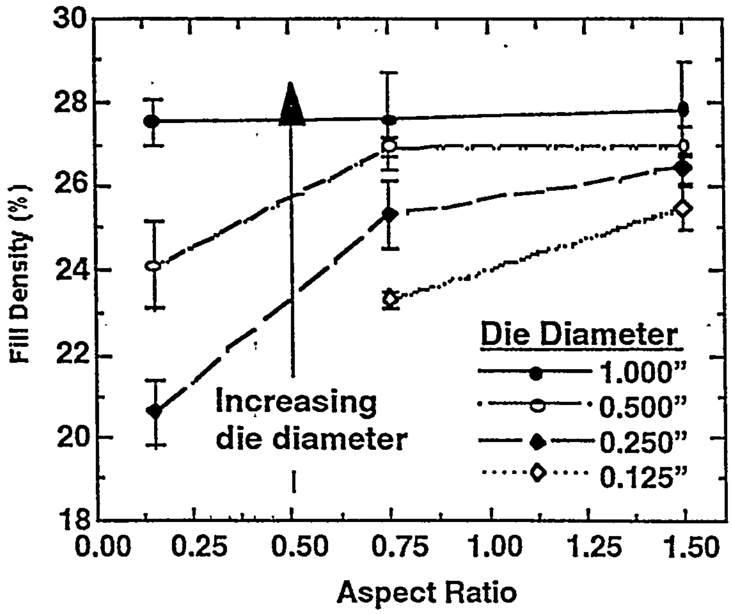

Fig. 7 Fill density vs. aspect ratio for different die diameters.

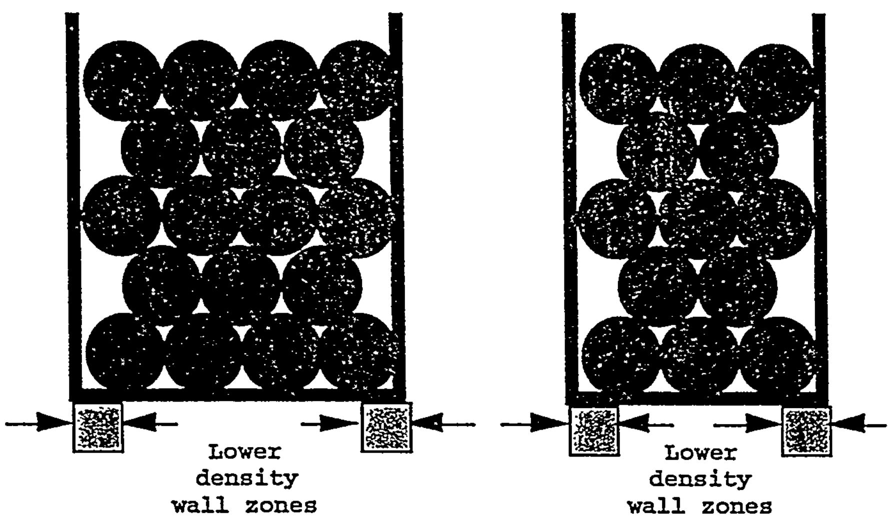

Fig. 8 Schematic of effect of granule size vs. die diameter. Lower densities measured for smaller diameter dies reflect the fact that a greater volume fraction of the compact is made up of the more loosely packed particles along the die walls.

The observation that density gradients introduced during filling persist throughout the compaction process may also explain why ultrasound results show noticeable differences in regions that should have been the same density based on die symmetry. These density gradients were likely introduced during die filling. This result highlights the importance of accounting for the discrete particle interactions, both in terms of their size relative to the die diameter, and their ability to rearrange prior to compaction.

CHARACTERIZATION OF INDIVIDUAL GRANULES

Producing a compaction model that accounts for the discreteness of the particles requires a knowledge of how the individual granules deform, and an understanding of the mechanisms of compaction (rearrangement only, deformation/fracture in conjunction with further rearrangement, removal of intragranular porosity, etc.). To obtain this information, experiments have been conducted on a model granule that is a solid glass sphere with a uniform 210 μm diameter.* Use of this model granule allows us to eliminate the effects of granule shape and size distribution inherent in compaction experiments on an assembly of spray dried granules. Although the glass spheres are harder and stronger than typical spray dried granules, they represent one of the extremes of ceramic granule behavior: that is the case where the binder is very hard and the amount of plastic deformation that occurs, especially at relatively low compaction pressures, is extremely limited.

Compaction of model granules produced the compaction response diagram shown in Fig. 9. Although there is some question as to whether the apparent linear regimes observed in Fig. 9 represent distinct mechanisms of compaction, our results indicate that there is very limited granule fracture in the first linear regime. We have also observed that the apparent "breakpoint" in the compaction curve does indeed scale with the fracture strength of individual granules (as measured in diametral compression tests), and that it appears to be more representative of the low end of the strength distribution.[28] Thus the breakpoint is at least a scaling factor, and stronger granules will shift the compaction curve to higher pressures, producing a higher apparent breakpoint. Higher breakpoints mean that granules can undergo rearrangement at higher compaction pressures to increase their packing uniformity prior to deformation and fracture, thereby minimizing filling and packing inhomogeneities. Onoda demonstrated that under conditions where die filling is non-uniform on a macroscopic scale (i.e., convex die filling), a granule that is strong enough to allow significant rearrangement prior to deformation/fracture may be

* Cataphote Inc.™ Microbeads, Jackson, Mississippi, 39226.

most desirable for controlled uniform compaction.[33] Similar logic may be applicable to the case where particle packing is non-uniform on a microscopic scale. Note that the macroscopic density increase that occurs during "rearrangement" is normally only a few percent; however, on a microscale there may be a very large increase in density if a large pore or packing defect is eliminated.

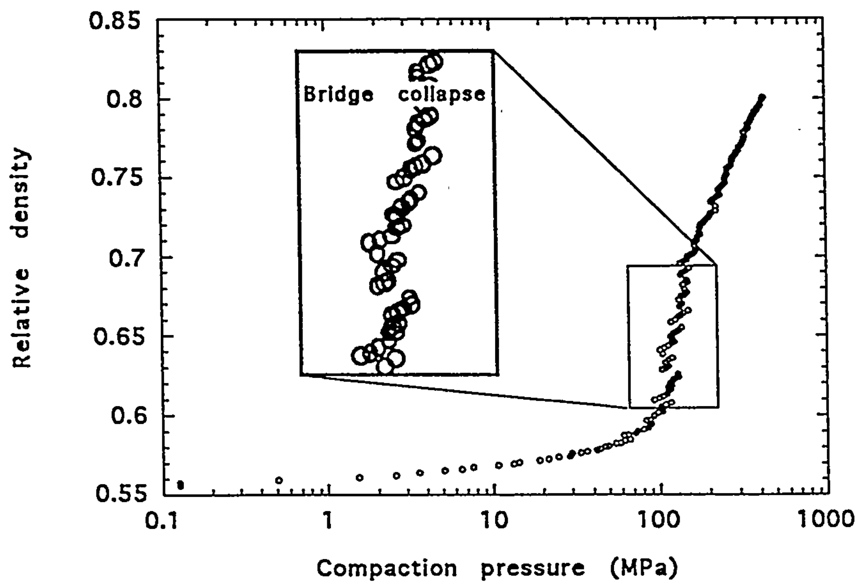

Fig. 9 Compact density vs. log compaction pressure for 210 μm diameter glass spheres.

Results from experiments on glass spheres also indicate that particle bridging, which has been postulated as a mechanism for producing density gradients in isostatic pressing, occurs during compaction. Bridges are networks of granules or granule that support pressures higher than the applied pressure (the implication being that there are also regions between the bridges that are supporting less than the applied pressure and are thus being shielded). Simulations using discrete element models have also indicated the formation of bridges during compaction.[34,35] In the compaction of glass spheres, a bridge supports high stresses until a sphere fractures or pops out of its position, allowing further rearrangement to occur. This stick-slip behavior can be seen in the blowup of the compaction response diagram in Fig. 9. When a bridge collapses there is a significant drop in pressure accompanied by a jump in density. This behavior also provides compelling support for using discrete element methods to model compaction.

Fig. 10 shows a Weibull plot obtained from strength distributions measured for the model granules. The very low Weibull modulus (obtained from the slope of the Weibull plot), indicates a very wide strength distribution. This data supports the use of a statistical mechanics approach in conjunction with discrete particle methods to develop a comprehensive model for compaction.

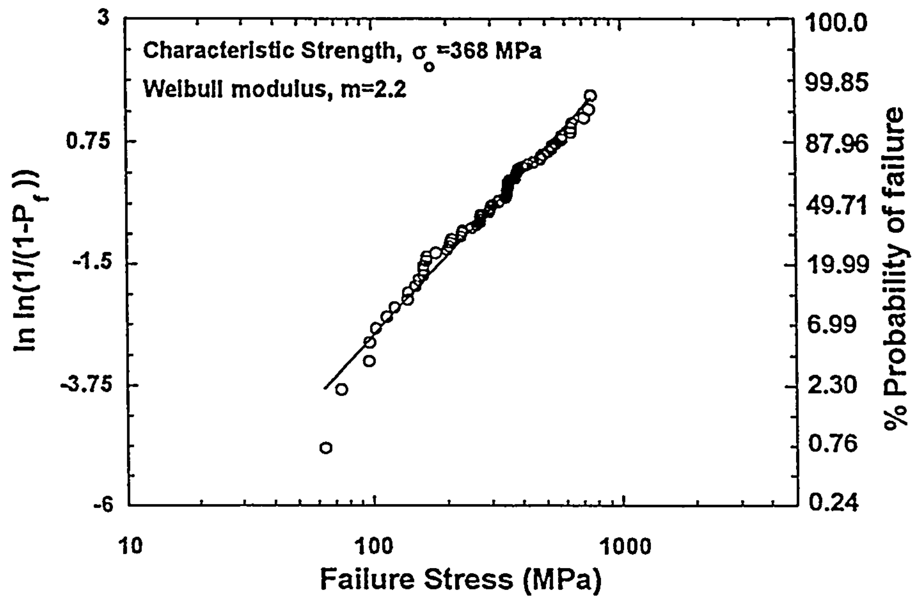

Fig. 10 Weibull plot for 210 µm diameter glass spheres broken in diametral compression.

CONCLUSIONS

There are two scales of density variation that are important in ceramic powder compaction: 1) macroscopic density gradients produced by non-uniform die filling and pressure gradients during compaction; and 2) microscopic variations due to poor granule packing in conjunction with limited rearrangement and incomplete deformation of granules during pressing. Our results support the philosophy that, under conditions where die filling or particle packing is non-uniform, a granule that is strong enough to allow significant rearrangement prior to deformation/fracture <u>may</u> be most desirable for controlled uniform compaction.

Discrete element modeling provides the potential to: 1) identify which material and physical parameters are likely to be important for compaction; and 2) obtain accurate predictions of compaction behavior. Experiments on individual granules support the use of a combined discrete element and statistical mechanics approach that accounts for the bridging behavior and the inherent variability in the mechanical response of the granules. Ultimately a useful predictive model of compaction will require a balance between computing resources and the inclusion of sufficient detail. The model will need to incorporate parameters that are measurable in a typical industrial lab setting, to provide a realistic representation of a very complex, dynamic process. The use of computers with parallel processing capabilities should allow us to identify the parameters and the level of detail that should be included in a desk-top, user-friendly computer model for powder compaction.

Application of a compaction process model in the manufacturing environment is expected to lead to cost savings through higher yields and the minimization of costly machining operations. Significant cost and time savings could also be realized through faster and more reliable component design (i.e., eliminating prototypes), shorter process development schedules, and the production of more reliable materials with improved performance.

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