EXPERIMENTAL AND NUMERICAL STUDY ON THE FLOW OF FINE POWDERS FROM SMALL-SCALE HOPPERS APPLIED TO SLS MULTI-MATERIAL DEPOSITION – PART I

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Abstract

We present experimental guidelines for the delivery of powders under 100μm through hopper-nozzle orifice diameters on the order of 1mm. Small-scale hoppers will be incorporated into an SLS powder deposition system for creating thin layers of multiple powdered materials in a patterned bed. This is a preliminary investigation on the flow behavior for selected orifice diameters and particle sizes under gravity or low pressure-assisted flow conditions. A method for numerically modeling the gas-particle behavior in hopper-nozzles is presented and conditions for achieving continuous mass flow rates are demonstrated.

INTRODUCTION

The development of SFF techniques for producing a new class of artifacts with spatially-varying structure and multi-functional characteristics is in part dependent upon the ability to deposit and consolidate multiple materials. In particular, the selective laser sintering (SLS) process is well-suited to the incorporation of multiple powdered materials. The SLS process presently uses a roller device to sweep thin layers of a single powdered material across the build area. It has been proposed that this roller device be replaced by an array of hopper-nozzles that discretely deposit lines, dots and regions of multiple powdered material in a patterned bed.

The designated name “hopper-nozzle” refers to the design of experimental nozzles based on existing hopper theory. In the chemical and process industries, hoppers have been inexpensively designed to store, discharge bulk solids, and eliminate undesirable flow instabilities (i.e. arching, rat-holing, and oscillatory flow). Unfortunately, difficult hopper and powder sizes are avoided due to the lack of fundamental understanding of flow phenomena. This can be attributed to the complex interaction of granular solid and interstitial fluid that plays a large role in delivery through small orifice diameters.

BACKGROUND

Granular bulk solids, like powder, exhibit characteristics unique from any other state. The granular mass is generally an amorphous, random-packing of particles influenced by the interstitial fluid occupying its voids. Unlike a fluid, the pressure under a vertical column of granular material is independent of its height, which makes a constant flow rate of material possible. Most flow theory has targeted particles above 500μm that are cohesionless (fair to free-flowing in nature) and insensitive to drag. Generally, particles below 50μm are small enough to be subject to cohesive effects. Those used with SLS are typically 0.1 to 100μm in diameter, and are more susceptible to air resistance when passing through the hopper orifice.

Evidently, the geometry of a hopper design is essential in achieving desired flow properties of the discharged material. The delivery of particles may happen under core flow (first in-last out) or the more desirable mass flow (first in-first out). For the design of a conical hopper, the hopper half-angle, α, and the orifice diameter, D_0 are the most important dimensions to be considered in preventing arching (bridging of particles that blocks flow) and rat-holing (an empty tunnel over the orifice due to core-flow). Jing and Li [9] provide useful information on hoppers for fine powders, stating an effective hopper half-angle of 10.2° or less for maintaining mass flow.

One of the most frequently used theories on flow from hoppers is W. A. Beverloo's modified correlation [2] for mass flow rate in conical hoppers. While there are other similar theories for mass flow rate, e.g. the hourglass theory, these do not take into account the effects of particle diameter. The modified Beverloo correlation is

$$G_s = C\rho_b\sqrt{g+\left(\frac{dp}{dz}\right)_0\frac{1}{\rho_b}}\,(D_0 - kd_p)^{5/2} \tag{2}$$

where G_s= mass flow rate of particles (g/s)
C = empirical constant (usually .58) dependent on hopper angle and angle of internal friction
ρ_B= bulk density of the powder (g/m^3)
g = gravitational constant of acceleration (9.8m/s^2)
D_0= hopper orifice diameter (m)
k = empirical constant for particle shape (1.6 for spherical particles)
d_p = mean particle diameter (m).

The term $1/\rho_B\ (dp/dz)_0$ is a modification of the gravitational acceleration term to include the interstitial pressure gradient. Beverloo's correlation states that as particle size decreases, there is an increase in the mass flow rate. Empirical results agree for particle sizes down to 500μm, where actual mass flow rate takes on much lower values than predicted.

The intent of this study is to determine whether a correlation for SLS powder sizes may be extended from Beverloo's correlation with low-pressure assistance. Research on granular flow is still in the exploratory phase, and the time-dependent behavior of a bulk powder material may not yet be based on characteristics of the constituent particles [6]. Progress in this area must depend upon uncovering new correlations between the observed behavior and measured particle characteristics [16]. With this in mind, the study aims to provide experimental results on the use of hoppers with fine powders, the design of the test apparatus and numerical model, and results of preliminary mass flow rate experiments conducted on a number of particle sizes and hopper-nozzles.

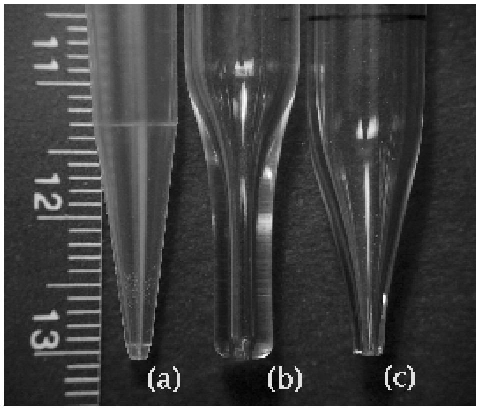


Figure 1. Pipette tips, pipets and drawn pipets are used as test hopper-nozzles.

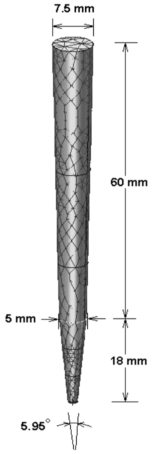


Figure 2. Pipette tip dimensions.

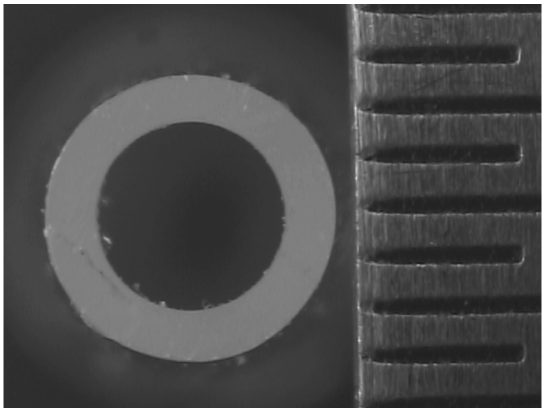


Figure 3. A high resolution image of the cut orifice at approximately 40 X magnification.

EXPERIMENTS

Hopper-Nozzle Design

Typically used for repetitive liquid dispensing, pipette tips were an inexpensive and practical choice for producing multiple, adjustable nozzles. The 1-200µL Uni-Tip© from BioPlas Inc., Figure 1 (a), is a siliconized polypropylene pipette tip with polished internal surfaces to help eliminate sample residue. The orifices of pipette tips are typically in the range of 0.75mm with a half-angle of 5.95°. They are designed for precise volumetric delivery; however there is no guarantee on the uniformity of the orifice diameters. A pack of 48 tips may have deviation up to 0.1mm.

Because the tips are polypropylene, they were fairly easy to trim with a utility knife under a magnifying glass. Images taken with a high-resolution digital camera (Figure 3) were used to make diameter adjustments and verify the quality of the cut surface. Tips with visible striations, residual material around the inner diameter, or irregular cross-sections were re-cut or eliminated. Diameters were calibrated with images taken from a 5cm long, 5mm nominal I.D. glass tube next to a measurement scale. A digital caliper is then used to measure ten different diameters along the glass tube from a printout of the cross-section. A measured diameter of 5.001+/-0.027mm indicated that using the high resolution digital camera is an accurate scheme for measurement. Images from the cut pipette tips were measured in the same manner, with magnification between 30X and 60X. The nozzles were re-cut until they were within 0.005mm of the nominal diameter. Fifteen orifice diameters from 0.75mm to 2.5mm in steps of 0.125mm were created for testing. As an alternative to the polypropylene tips, Pyrex 5mL glass pipets, Figure 1(b), were diamond-ground to diameters 1.0-2.0mm in steps of 0.1mm. The graduated markings on the pipets are used to observe the pressure changes with respect to the height of the powder column. Also by heating and drawing pipets, smaller diameters in the 0.5 to 1.0mm range were produced.

Mass Flow Rate Experiments

The test particles were high-quality soda-lime glass beads, verified by a distribution histogram as 90% within the specified U.S. sieve mesh sizes and 90% spherical. Although uniform particles of an identical diameter are considered the most desirable, it is believed that SLS powders are better represented by small ranges of particle size within 10-100 micron. The particles have diameter ranges d=10-25, 38-45, 45-53, 63-75, and 90-106 µm. A majority of techniques for bulk materials testing encourage the use of a batch at least a few kilograms [16] however a few grams of each powder are acquired considering the particles are a sufficiently representative sample.

The A&D GF-200 Precision Balance, a 0-200g, 0.001g resolution digital balance with RS-232C serial interface was used for sampling. The balance has Windows Communications Tools© for easy data transfer to Windows© 95/98. In its most rapid response mode, the balance has a 10Hz sampling frequency. Data can be imported into Microsoft Excel© or stored as unformatted data for other applications. In addition to mass and time, the recorded data will display 'ST' or 'US' (stable and unstable) which indicates whether the sample has been stabilized on the pan. This is a useful indicator for the flow conditions of the experiment. US (unstable) generally indicates the powder is flowing and ST (stable) indicates flow has stopped and it is times to begin or end sampling.

Experiments began with the calibration of the balance with an ASTM standard 100g mass. The powder delivery apparatus was positioned over the scale, and powders were deposited into an 8mL narrow-mouth glass laboratory bottle. Twenty samples were taken under each test condition, and mass data from the balance was transferred directly to a PC and stored as a generic data files. The files was then processed in program written in Matlab© for generating plots of the mass accumulation and mass flow rate versus time. To identify time-dependent flow instabilities such as oscillation or arching and their degree of influence on the flow, a program was written for applying a fast Fourier transform (FFT) to the sample data. The Fourier transform, a mathematical tool frequently used for electrical signal processing, decomposes a complex signal into its basic parts, and display the strength of the revealed signals in a particular range of frequencies. By using an FFT, mass data sampled in discrete time intervals were converted into mass data as a function of frequency. An additional program was also created to filter noisy signals due to possible equipment error.

Powder Delivery Apparatus

An extended column for powder above the pipette tip is needed, and additional height is achieved with 30mm long, 8 O.D., 5 I.D Pyrex glass tubing. The tube is suspended vertically by a standard laboratory support stand and clamps. The end of the Pyrex glass tube was ground down, tapering to 7mm so that pipette tips could be conveniently attached and removed during experiments.

Modifications to the conditions at the upper free surface of the powder column allow for each of the mass flow and nozzle test conditions: gravity, closed- top, and pressure- assisted flow. Gravity flow experiments are conducted at standard atmospheric pressure (14.696 psi(101kPa)), which means the top of the powder column is open to air and may interact with the powder particles at the free surface as well as through the nozzle. Closed-top experiments are sealed at the top with a rubber stopper, permitting air to be exchanged for powder particles only through the nozzle.

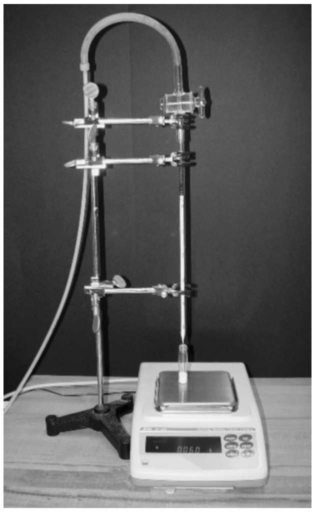


Figure 4. Test apparatus (pressure regulators not shown).

Pressure assisted experiments are designed to achieve continuous flow in pulsing powder/nozzle combinations and move cohesive powders by counteracting the viscous drag forces at the orifice. A 250 psi compressed air supply, regulated by two Bellofram© Type 70 precision air regulators, is fed through 1/4” O.D., 3/16” I.D. nylon tubing to the powder column. Both air regulators are capable of reducing the 250 psi line supply, however the 0-2 psi regulator has a much larger number of turns than the 0-30psi., providing fine tuning in the low pressure range. Two Omega© digital pressure gauges with 0.01 psi resolution are joined to the regulators for adjustment readings. Pressures of 0.25 (1.7kPa), 0.5 (3.4kPa), and 0.75psi (5.2kPa) are chosen for testing with each orifice diameters and particle size. The choice of pressures is based on observing that the minimal regulated pressure of 0.01 psi is raised to 0.25 psi. when added to a full powder column. A stopcock is used above the powder column to stop/start air-presssure assistance to prevent unwanted spraying of particles once the powder column has emptied.

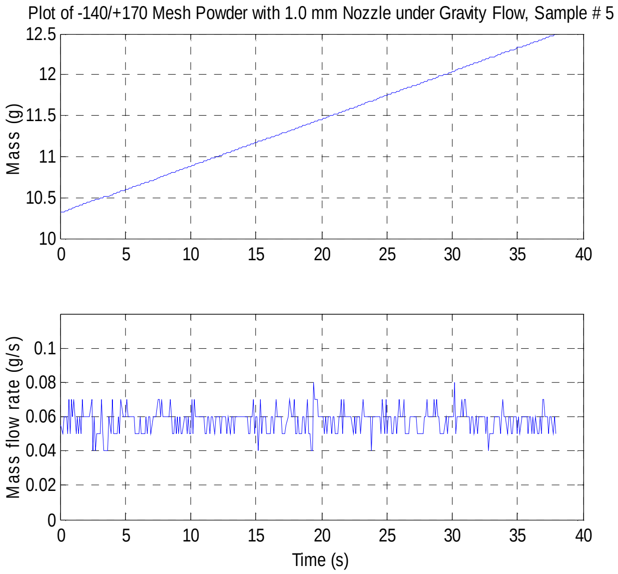
Numerical Model

Due to the highly complex multiphase flow behavior inside hoppers, numerical models are required for gaining detailed knowledge about gas-solid flow important for designing the proposed powder deposition system. In order to develop a scientific understanding of the flow of powders in our hopper-nozzle designs, numerical models based on kinetic theory of granular multiphase flow will be developed. This theory is based on the continuum equations of mass and momentum, and a transport equation for particle fluctuation energy based on the concept of a granular temperature to account for interactions between granular particles, as discussed by Gidaspow [3,4].

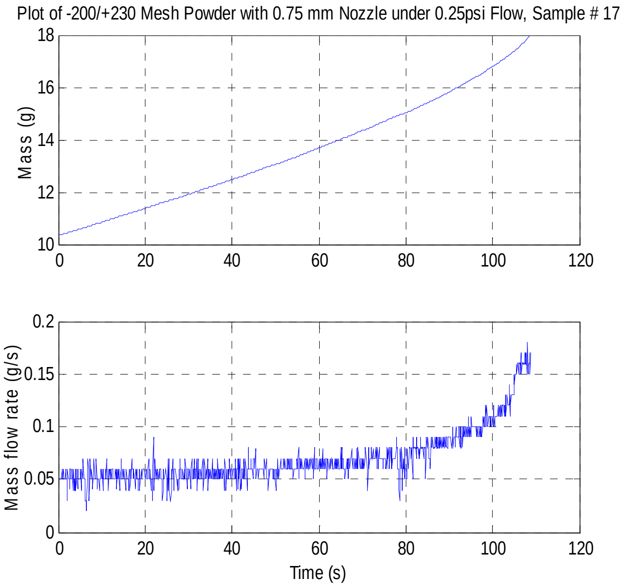
CFX, a computational fluid dynamics software that has previously been employed with considerable success for modeling granular multiphase flow [87-89] will be used to simulate 2-D and 3-D flow models of powders (10nm-100μm) inside hoppers (0.1-2.5mm openings). Conservation equations of mass and momentum will be developed for both the gas phase and the particulate phase using the Eulerian approach, and finite volume numerical methods will be employed to solve these equations. Detailed flow profiles of each phase in the hopper will be simulated, including the velocity and mass flow of particles, local solid fraction, and the pressure distribution of the interstitial gas. The 3-D numerical models will be verified by comparing the computed total particle mass flow with our experimental results. The effects of powder properties and hopper geometry on the bulk flow behavior of the particles will be studied. Critical conditions leading to undesirable arching, rat-holing and oscillatory flow behaviors will also be investigated.

RESULTS

Figure 5 shows the accumulated mass v. time, and Figure 6 the corresponding mass flow rate v. time for -140/+170 mesh (90-106µm diameter) powder under standard gravity flow from a pipette tip with 1.0mm orifice. This plot is a sample of a “continuous” flow condition, where a stable (within 0.01g/s) flow can be observed over the full length of the powder column. Similar flow behavior can be observed under gravity flow for powders in the 68 to106µm range for all pipette tip diameters down to 0.75mm. Arching in the orifice has been observed for particles smaller than 68µm, and tapping the pipette tip may temporarily induce a pulsing flow or one that stops completely.



Figures 5 & 6.



Figures 7 & 8.

In Figures 7& 8 a “continuous” mass flow can be achieved with 0.25psi gas assistance for 38-68µm particles and tip diameters down to 0.75mm. The initial mass flow rate achieved is very similar to that of the powder under gravity flow. However, this continuous flow behavior exists for a characteristic powder column length greater than 15-20cm. This phenomenon can be observed by the slight upward turn in mass accumulation (Figure 7), the more obvious increase in mass flow rate beginning after 50 seconds (Figure 8), and significant increase to 0.15g/s almost 3 times as the powder column empties. This is one demonstration of how fine particles that do not flow under standard gravity conditions may be assisted by a very low pressure to achieve comparable mass flow rates.

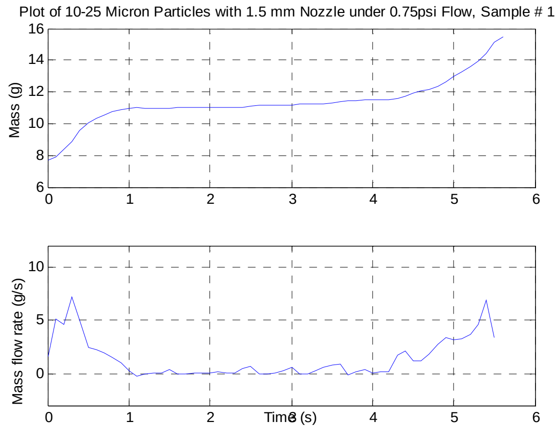


Figure 9 &10.

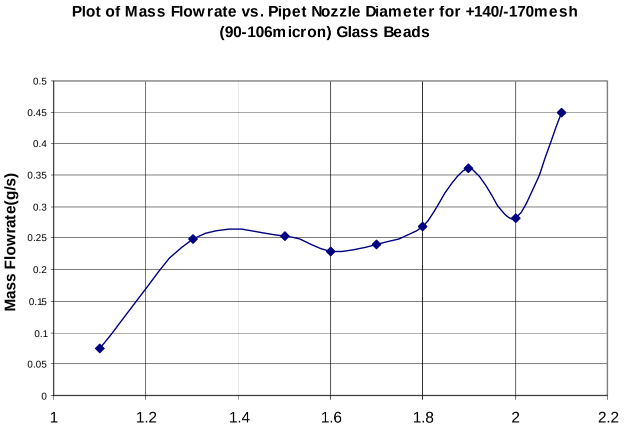


Figure 11.

However, Figures 9&10 indicate particles as small as 10-38µm are very difficult to deliver with a predictable mass flow rate. A higher level of pressure assistance (0.75psi is shown) pushes particles down the powder column at a high rate, yet these very cohesive powders are not being fluidized. By definition, the inter-particle forces of a cohesive powder resist fluidization, and interstitial gas has a harder time permeating the powder column. The flow may be subject to frequent stopping as shown. For now, highly cohesive powders flow out of the powder column at rates which are too high and unpredictable for incorporation with SLS.

Mass Flow Rates v. Diameter

Under gravity flow in pipets, for fixed particle diameter, continuous flow can be observed over a range of orifice diameters. However the flow rate exhibits a nonlinear relationship with respect to orifice diameter (Figure 11). There appears to be optimal combinations of hopper-nozzle diameter to particle diameter. This is helpful for recognizing that there is no simple assumption of a linear relationship between mass flow rates and reduced orifice diameters. Experiments are being developed to determine how this phenomenon relates to the taper angle of the hopper or electrostatic charging at the powder column walls.

CONCLUSIONS

A preliminary study of the flow of fine powders from small-scale hoppers demonstrates that highly spherical particle 38-100µm in diameter may be delivered at continuous mass flow rates with low gas-pressure assistance as small as 0.25psi. Alternate techniques must be developed for delivering very cohesive particles in the range of 0.1-38µm. This results of this study are useful for development of a hopper-nozzle array for incorporating multiple powders in the SLS process. Future work will include comparing the results of the numerical model with experimental data to enable further refinement of the model. Simple analytical models for characterizing deposited line width, line height and line deposition time as a function of mass flow rate, nozzle opening, nozzle to substrate distance and linear speed will further refine this process.

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