PRELIMINARY INVESTIGATIONS ON THE DEPOSITION OF FINE POWDERS THROUGH MINIATURE HOPPER-NOZZLES APPLIED TO MULTI-MATERIAL SOLID FREEFORM FABRICATION

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Abstract

A concept for multi-material solid freeform fabrication is proposed to enable the fabrication of heterogeneous components. This concept features nozzles designed for depositing thin layers of multiple patterned materials followed by selective laser sintering for consolidation to desired densities. Although prior work on the design of small-scale nozzles for powder delivery is lacking, our design is guided by background theory for particle flow through hoppers. Experimental guidelines for the delivery of powders with particle sizes in the 10-125μm range through hopper-nozzle orifices with diameters in the 0.5-2mm range are presented. This is a preliminary investigation of particle flow behavior necessary for continuous mass flow rates under gravity, low gas pressure-assisted flow, and vibration-assisted flow conditions. As proof of concept, several patterned beds of single and multiple materials were deposited on an X-Y table. A simple model to predict the linewidth of lines deposited by gravity flow is presented.

1. 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 [11]. 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 to replace this roller device by an array of hopper-nozzles that can directly write lines, dots and patterned regions of multiple powdered materials [12].

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.

2. BACKGROUND

Granular bulk solids 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 irrespective of the column height.

Many researchers have investigated the flow of a powder through orifices under gravity and have established empirical correlations to predict the mass flow rate [1,7,8,9]. Among all these

correlations, Beverloo's correlation [1] is the most widely used to predict the discharge rate. It is given by

$$G_s = C\rho_B\sqrt{g}\left(D_0 - kd\right)^{2.5} \quad \text{g/s} \qquad (1)$$

where G_s is mass flow rate of particles(g/s), C is empirical constant, ρ_B is bulk density of powder(g/mm^3), g is acceleration due to gravity(mm/s^2), D_0 is hopper orifice diameter(mm), k is empirical constant for particle shape, and d is mean particle diameter. C and k are dimensionless constants and they take the values 0.583 and 1.4 respectively as reported by Beverloo et al. [1].

Spink and Nedderman [2] found that Beverloo's correlation is valid only for powders of particle size greater than 500 μm. Eq. (1) indicates that particle flow rate increases slowly as particle size decreases (curve-1 in Fig. 1). Significant deviations from this equation occur at small particle sizes, where it is found that the discharge rate passes through a maximum and then progressively and rapidly decreases as particle size decreases below 500μm (curve-2 in Fig.1). The reduction in flow rate of fine powders has been attributed to retarding influence of interstitial fluid by various researchers [2, 3, 4]. Spink et al. [2] developed an iterative numerical method based on stress distribution, voidage distribution and air pressure gradient distribution in the powder column to predict the discharge rate. However, their theoretical model did not match their experimental results. Moreover, their theoretical model is cumbersome to implement.

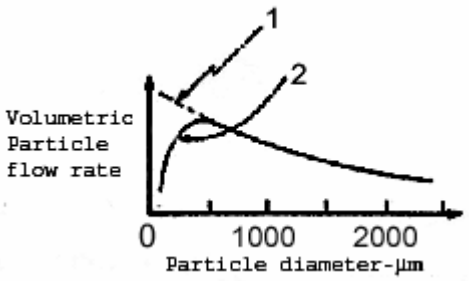


Fig. 1 Volumetric discharge rate vs. particle size [2]

The orifice size used in experiments by various past researchers was very large and on the order of 2-100mm [1,2,7,8,9], because their research was mostly concerned with industrial processing systems, such as feeding of catalyst pallets to a cracking plant. The smallest orifice size found in previous studies was 2mm used in the work of Beverloo et al. [1].

For development of a SLS hopper-nozzle powder delivery system we intend to use orifice sizes in the range 10μm-2mm. The powders used for SLS are typically 0.1-150μm in diameter. Flow behavior for this range of particle size and nozzle size combinations has not been investigated previously. The intent of this study is to determine whether a simple correlation for SLS powder sizes and intended orifice sizes can be extended from Beverloo's correlation. 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 [5]. Progress in this area must depend upon uncovering new correlations between the observed behavior and measured particle characteristics [6]. 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 results of preliminary mass flow rate experiments conducted on a number of particle sizes and hopper-nozzles.

3. EXPERIMENTS

3.1. Hopper-Nozzle Design

Pipette tips, typically used for repetitive liquid dispensing, were an inexpensive and practical choice for producing multiple, nozzles with various opening diameters. The 1-200µL Uni-Tip© from BioPlas Inc., Fig. 2(a), is a siliconized polypropylene pipette tip with polished internal surfaces to help eliminate sample residue. The orifices of pipette tips are of nominal diameter 0.75mm with a half-angle of 6.575° (Fig. 3). 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.

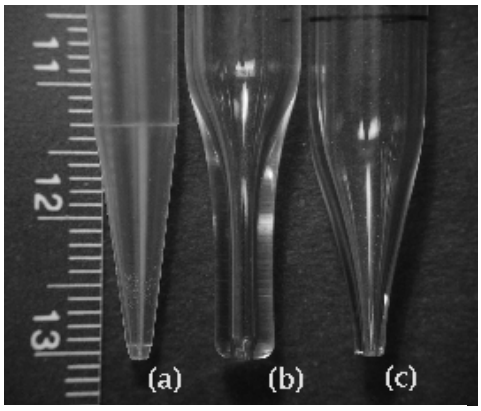


Fig. 2 Pipette tips, pipettes and drawn pipettes are used as test hopper-nozzles

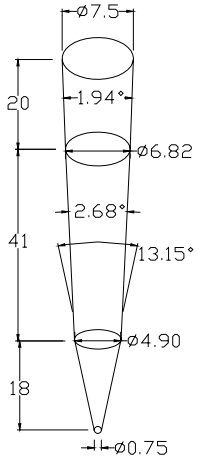


Fig. 3 Pipette tip dimensions

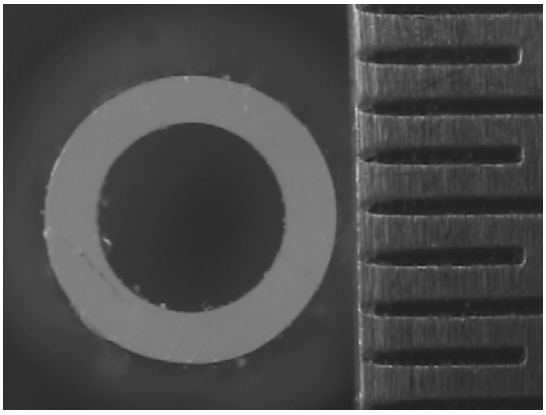


Fig. 4 A high resolution image of the cut orifice at approximately 40 X magnification

Because the tips are made of polypropylene, they were fairly easy to trim with a utility knife under a magnifying glass. Images taken with a high-resolution digital camera (Fig. 4) were used to make diameter adjustments and verify the quality of the cut surface [12]. Eleven 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 pipettes, Fig. 2(b), were diamond-ground to diameters 1.0-2.0mm in steps of 0.1mm. In addition, by heating and drawing the glass pipettes, smaller diameters in the 0.1 to 1.0mm range were produced, Fig. 2(c).

3.2. Powder Delivery Apparatus

An extended column for powder above the pipette tip is needed, and additional height was achieved with 30mm long, 8mm O.D., 5mm I.D Pyrex glass tubing. The tube was held vertically by a standard laboratory support stand and clamps (Fig. 6). 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 (Fig. 5).

Gravity and pressure-assisted flow conditions were achieved by modifying the conditions at the upper free surface of the powder column. When the top of the powder column is open to atmosphere, gravity flow condition is achieved. Pressure assisted experiments were designed to achieve continuous flow for powder/nozzle combinations for which there was no flow under gravity. A 250 psi compressed air supply, regulated by two Bellofram© type 70 precision air regulators, was fed through 1/4" O.D., 3/16" I.D. nylon tubing to the powder column (Fig. 6). 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. regulator, providing fine tuning in the low pressure range. Two Omega© digital pressure gauges with 0.01 psi resolution were joined to the regulators for pressure readings. A stopcock was used above the powder column to stop/start air-pressure assistance. Vibration assisted experiments were designed as an alternative to pressure assisted flow to get a continuous flow when there was no flow under gravity and to overcome some problems encountered with pressure assisted flow, as will be discussed later in this paper. For vibration assisted flow experiments, a piezoelectric (PZT) actuator strip was placed near the tip of nozzle (Fig. 7). The PZT actuator has a resonance frequency of 29 kHz, free deflection of 3.4 μm and could be actuated at any frequency in the range 0-25 kHz using a sinusoidal signal of different frequencies generated using a LabView program and National Instruments PCI-MIO-16E-1 input/output card.

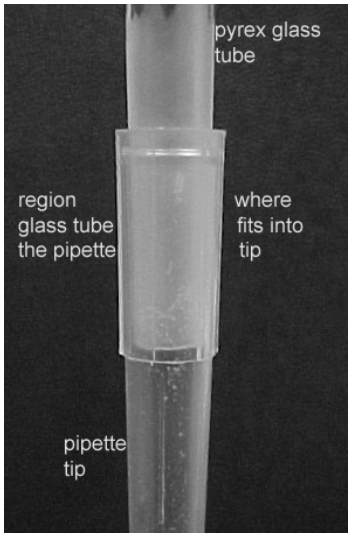


Fig. 5 Glass tube attached to pipette tip to get extended column of powder

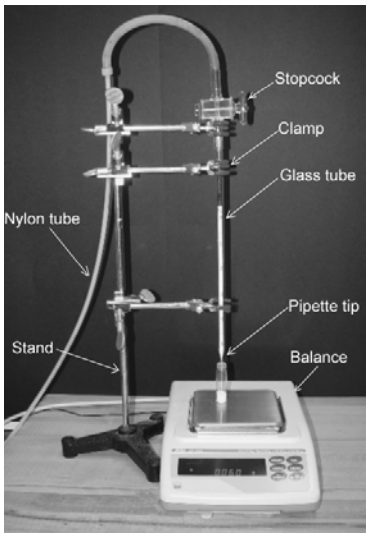


Fig. 6 Test setup; data acquisition with 1-mg resolution balance, 10Hz sampling with PC interface

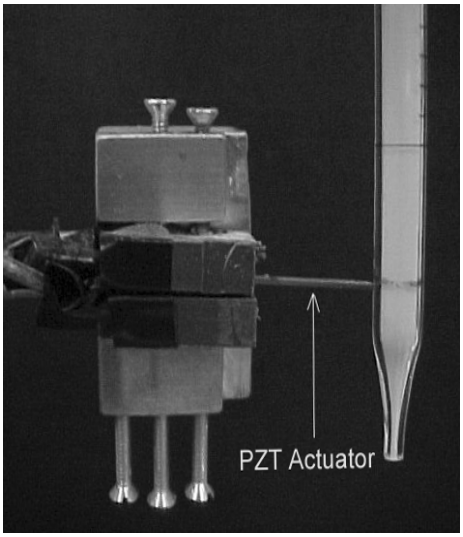


Fig. 7 Use of a PZT actuator strip for vibration assisted flow

3.3. 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. The particle sizes used were 325-400mesh (38-45μm), 270-325mesh (45-53μm), 230-270 mesh (53-63μm), 200-230mesh (63-75μm), 170-200mesh (75-90μm), 140-170mesh (90-105μm), and 120-140mesh (105-125μm). All the powders had bulk density of 1.3 g/cc.

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© software for easy data transfer. 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.

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 bottle. Four samples were taken under each test condition, and mass

data from the balance was transferred directly to a PC and stored as generic data files. The files were then processed using a program written in Matlab© for generating plots of the mass accumulation and mass flow rate versus time.

3.4. Angle of Repose Experiments

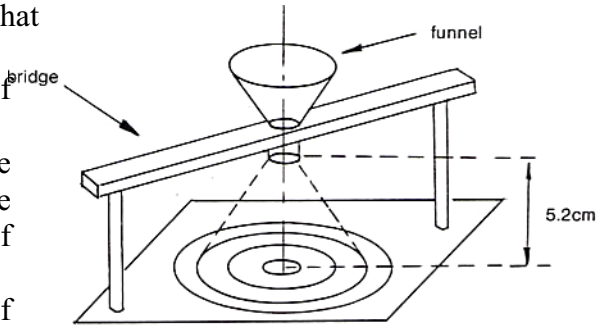
The angle of repose is defined as the angle of the free surface of a pile of powder to the horizontal plane. It is a measure of flowability and cohesiveness of a powder. A modified Angle of Repose of a Heap test (British Std. 4140) [10] was used to characterize the flow of each of the test particles. Some of the modifications include:

a) A substrate of identical powdered material as that of deposited powder was used.

b) A polyethylene funnel with inner diameter of 4mm was used.

c) The height of the polyethylene funnel above the substrate was lowered from 5.2cm to 5mm to save powder used in each test (only 0.5 grams of powder was needed).

d) Concentric circles used to measure the angle of repose were drawn for every 5º.



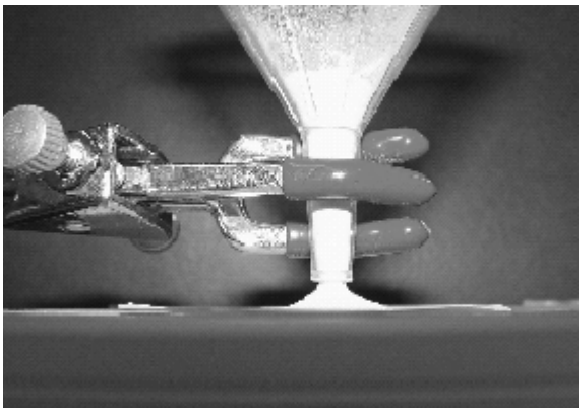


Fig.9. Modified setup to measure angle of repose

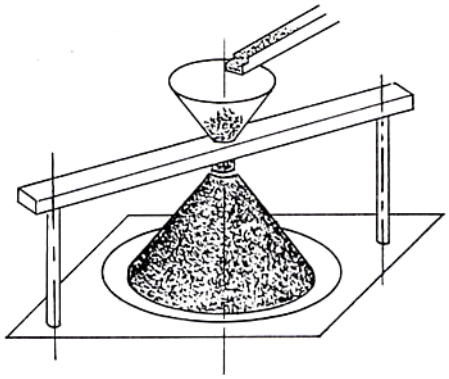


Fig.8. Setup for measuring angle of repose, British Std. 4140 [10]

3.5 Pattern Deposition Experiments

Deposition experiments were conducted on a servo motor-driven X-Y table, with optical encoder feedback. This table is controlled by an SB214PC multi-axis controller from ACS-Tech80 Inc. The maximum resolution of the table is 0.0005in. (0.0127mm). Patterned bed designs were programmed using ACS-programming language.

4. RESULTS AND DISCUSSION

4.1. Gravity Flow

Table 1 shows the average mass flow rate for the range of nozzle sizes and particle sizes tested under gravity flow. Under gravity, there was no flow for particle sizes below 63µm through the entire range of nozzle openings (orifice diameters) tested. Fig. 10 shows the plot of mass flow rate vs. nozzle opening for the four particle sizes that flowed under gravity. It is observed that there is very little variation in the mass flow rate with particle size in the range tested and flow

rate for all particle sizes, in the range(63-125μm) can be described by a single power law curve fit with $R^2 = 0.9823$

Orifice diameter D_0 (mm)	Average mass flow rate(g/s) for various powder particle sizes (mesh)						
	120-140 (105-125 μm)	140-170 (90-105 μm)	170-200 (75-90 μm)	200-230 (63-75 μm)	230-270 (53-63 μm)	270-325 (45-53 μm)	325-400 (38-45 μm)
0.75	0.0173	0.0173	0.0210	0.0168			
0.875	0.0259	0.0285	0.0245	0.0260			
1.00	0.0315	0.0275	0.0346	0.0423			
1.125	0.0484	0.0467	0.0474	0.0536			
1.25	0.0542	0.0650	0.0752	0.0692			
1.375	0.0947	0.1014	0.1048	0.1151			
1.5	0.1063	0.1064	0.1560	0.1493			
1.625	0.1745	0.1585	0.1976	0.1925			
1.75	0.2255	0.2237	0.2154	0.2072			
1.875	0.2758	0.2615	0.2700	0.2765			
2.00	0.3073	0.2991	0.2972	0.2901			

Table 1. Gravity mass flow results

From Beverloo's correlation, eq. (1), we get

$$G_s = C\rho_B\sqrt{g}\left(D_0 - kd\right)^{2.5} \Rightarrow D_0 = \left(\frac{1}{C\rho_B\sqrt{g}}\right)^{0.4} G_s^{\,0.4} + kd \qquad (2)$$

There is a linear relationship between D_0 and $G_s^{0.4}$. Therefore, if Beverloo's correlation, for some values of C and k, holds for this experiment, a linear relationship would be expected between D_0 and $G_s^{0.4}$ obtained from the experimental data. Fig. 11 shows a plot of D_0 vs. $G_s^{0.4}$ for the experiment. It is observed that experimental data can be described by a straight line with a fit of $R^2 = 0.9828$. Using a mean particle size of 91μm, from the slope and intercept on D_0-axis of this line and using eq. (2) we get

$$C = 0.604, \; k = 2.86 \qquad (3)$$

The values of C and k were also calculated for each of the four particle size distributions individually in a similar manner and are listed in tab. 2. The mean values of C and k obtained from these calculations are 0.0602 and 2.855 respectively, which are very close to those obtained above from a single line fit through all the experimental data. This shows that the value of C and k given by relation (3) can be used with eq. (1) with a mean particle size $\bar{d}$ =91μm to predict the mass flow rate for the particle sizes in the range 63-125μm. Thus, for the particle sizes in the range 63-125μm, flow rate under gravity can be predicted by a single expression

$$G_s = 0.604\rho_B\sqrt{g}\left(D_0 - 2.86\bar{d}\right)^{2.5} \text{ g/s}, \; 0.75mm \le D_0 \le 2.00mm, \quad \bar{d} = 91\mu m \qquad (4)$$

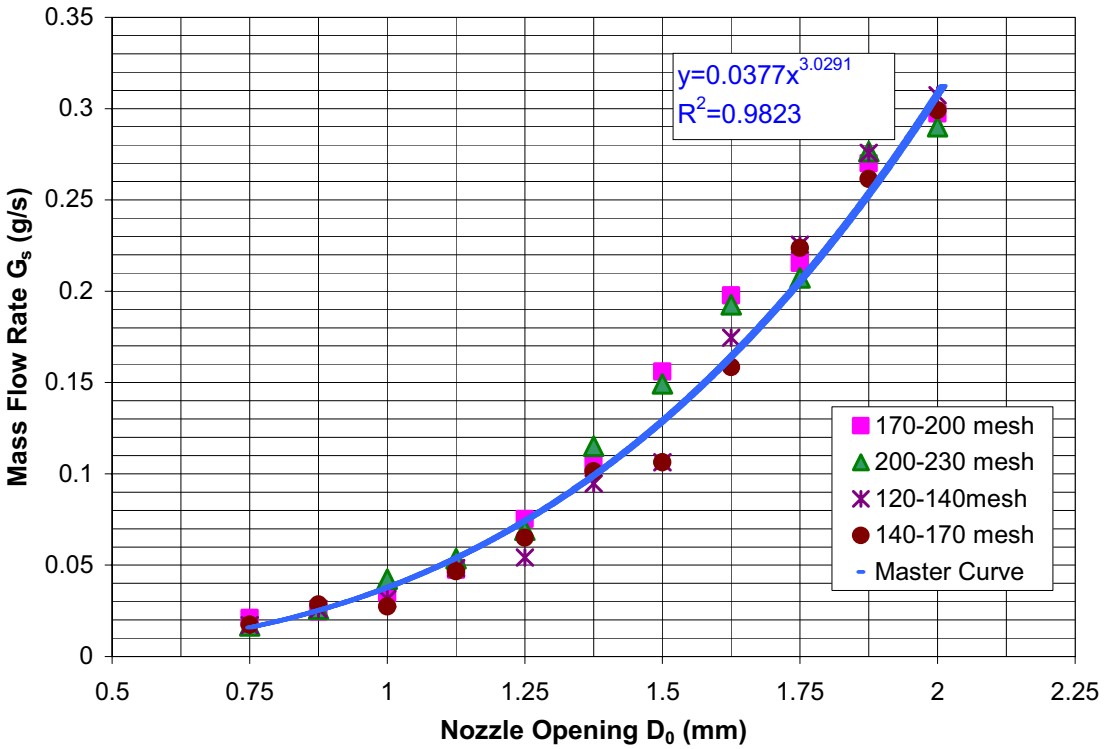


Fig. 10 Mass flow rate as a function of nozzle opening with particle size as a parameter

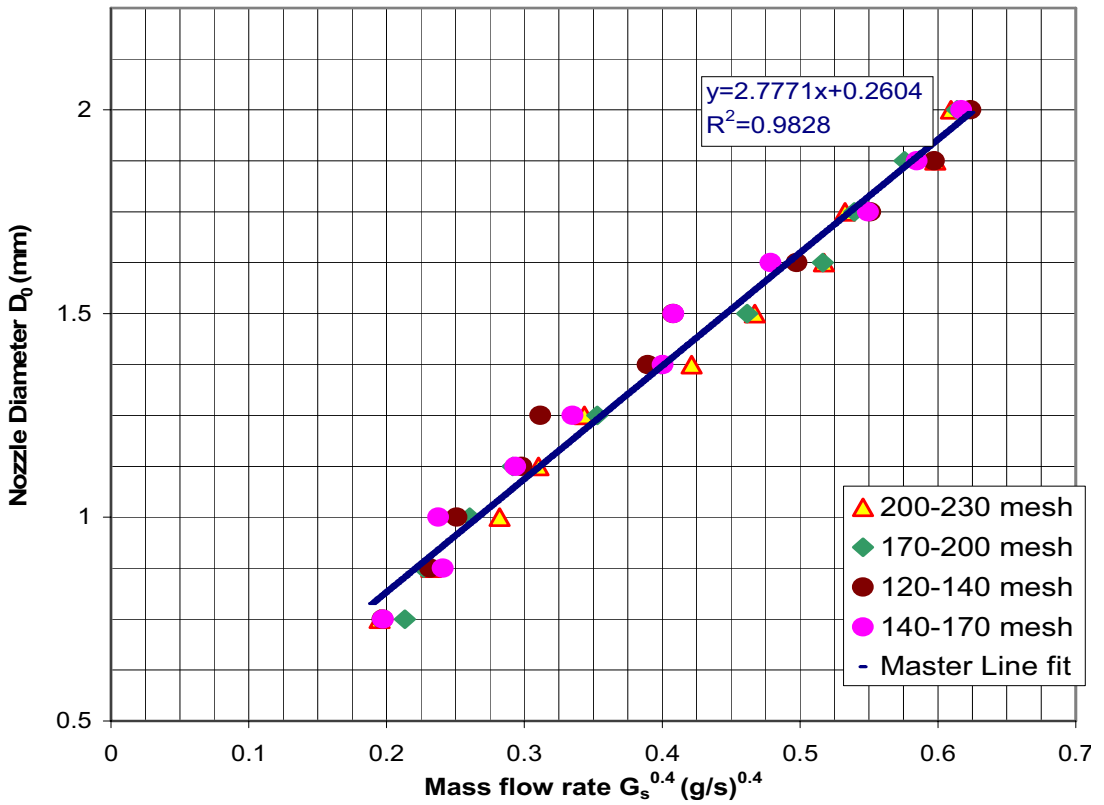


Fig. 11 Master linear plot of D_0 vs. $G_s^{0.4}$ for the experimental data of all the particle sizes tested

It should be noted that the value of C =0.604 obtained from our experiment is nearly the same as that (0.583) obtained by Beverloo et al. [1], while the value of k is almost double (2.86 vs. 1.4). The differenece in the values of k might be due to the fact that the particle size used in our experiments (10-125µm) is one order of magnitude smaller than that(1.6-3.0mm) used by Beverloo et al. It is possible that the value of k depends on the particle size, d. However due to lack of experimental data for the particles in the range 125µm-1.6mm the exact dependence of k on d could not be deduced.

Particle Size	C	k
120-140 mesh	0.650	2.665
140-170 mesh	0.601	2.828
170-200 mesh	0.576	2.774
200-230 mesh	0.580	3.154
Average	0.602	2.855

Table 2. Values of C and k obtained from individual plots of D_0 vs. $G_s^{0.4}$ for each particle size

4.2. Pressure-assisted flow

As mentioned earlier, particles below 63µm do not flow under gravity through the nozzle sizes tested. To fluidize these powders gas pressure assistance was used. However, some problems were encountered. When the column height was low, the powder spurted out with very high velocity, an undesirable effect. Minimum possible kinetic energy of powder particles is desired so that powder spreading on a substrate is minimized when a pattern is deposited. For the fine powders (e.g., 10-25µm), constant flow rate was not achieved; the flow was sporadic and unpredictable as shown in fig.12. Hence, an alternate approach to depositing fine powders consistently was sought.

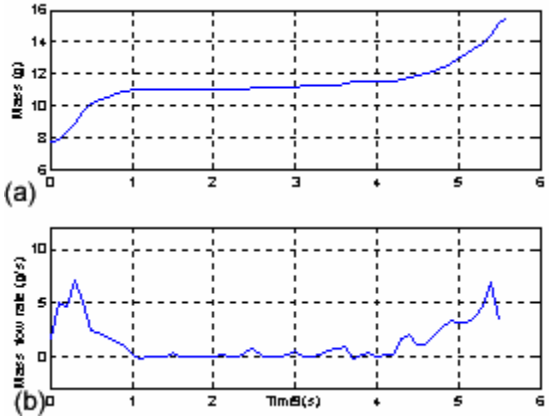


Fig. 12 (a) mass vs. time plot, (b) mass flow rate vs. time plot for 10-25µm powder from 1.5mm nozzle under 0.75 psi pressure

4.3. Vibration-assisted flow

To overcome the problems presented by pressure-assisted flow, vibration assistance was used. A PZT actuator strip, as shown in fig. 7 and discussed earlier, was placed near the tip of nozzle to vibrate the powder column with low amplitude and high frequency. The plot of flow rate vs. vibration frequency for two powders that did not flow under gravity is shown in fig.13. It is observed that there is a peak at 13 kHz for the particular position of PZT with respect to the nozzle opening that was used in the experiment.

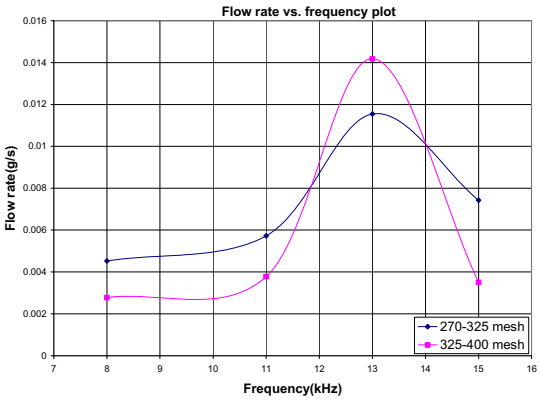


Fig. 13 Plot of mass flow rate vs. frequency for flow through 0.5mm glass nozzle

Continuous flow was obtained even for very fine powder (10-25µm) through a 200 µm nozzle opening. An additional benefit of vibration assisted flow is that automatic valving to start and stop the flow was achieved without any additional mechanism. Further investigation on vibration assisted flow is under way.

4.4. Angle of Repose and Linespread

The angle of repose for various powders as measured by powder piling experiment is shown in tab. 3.

Particle size μm (mesh)	250-300	90-106 (140-170)	63-75 (200-230)	45-53 (270-325)	38-45 (325-400)	10-25
Angle of repose (degrees)	30	35	40	45	50	50

Table 3. Angle of repose

The angle of repose is a measure of cohesiveness of a powder; the larger the angle the more cohesive is the powder. When a line of powder is deposited, the width of line depends on the nozzle diameter and the cohesiveness of the powder. The spread of a deposited line is more for less cohesive (free flowing) powder than for more cohesive powder. Therefore, for the same nozzle diameter, the width of a deposited line is more when angle of repose is less and vice versa.

A simple model to predict the linewidth deposited under gravity flow was developed based on angle of repose and principle of mass conservation which translates to the fact that the volume of the powder exiting the nozzle per unit time should be same as volume deposited if we assume the bulk density of the powder to be the same in the nozzle and the deposited line. For the powders in the 63-125μm size range, the linewidth is given by the following relations:

Let θ be the angle of repose and $h = \dfrac{0.604\sqrt{g}\left(D_0 - k\bar{d}\right)^{1.5}}{v}$ (5)

Where v = velocity at which nozzle is moving (mm/s)

Case-1:For $\theta > \tan^{-1}\left(\dfrac{h}{D_0 - k\bar{d}}\right)$ Linewidth, $B = \left(D_0 - k\bar{d}\right)\left[1 + \dfrac{0.604\sqrt{g}\left(D_0 - k\bar{d}\right)^{0.5}}{v\tan\theta}\right]$ mm (6)

Case-2:For $\theta \le \tan^{-1}\left(\dfrac{h}{D_0 - k\bar{d}}\right)$ Linewidth, $B = \dfrac{1.55\sqrt[4]{g}\left(D_0 - k\bar{d}\right)^{1.25}}{\sqrt{v\tan\theta}}$ mm (7)

Fig. 14 shows pictures of lines of 140-170 mesh powder (θ = 35º) deposited at three different speeds from 0.75mm nozzle. Table 4 compares the calculated linewidths with actual linewidths obtained from experiments. It is observed that the predicted linewidths matches experimentally measured values closely (within approximately two particle diameters).

V(mm/s)	Calculated Linewidth	Experimental Linewidth
68	0.58 mm	0.8 mm
56	1.00 mm	1.2 mm
25	1.51 mm	1.5 mm

Table 4. Comparison of predicted linewidth with experimentally measured linewidth

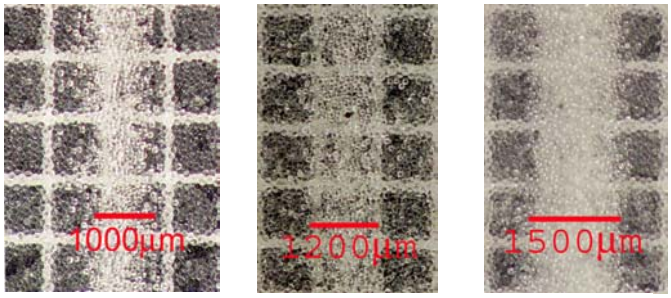
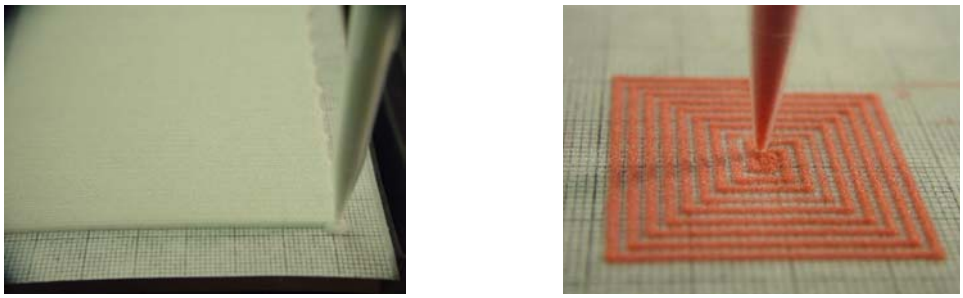


Fig. 14 Lines of 140-170 mesh powder deposited from 0.75 mm nozzle at nozzle speeds 68, 56, 25mm/s respectively (left to right)

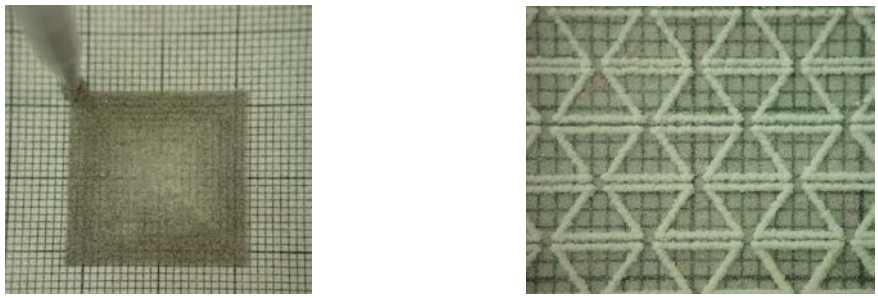
4.5. Pattern Deposition

The following illustrations are proof-of-concept for various types of single and multi-material patterned beds that are envisaged for the construction of various heterogeneous devices.



(a) (b)

Fig. 15 (a) Rastered bed of 90-106µm glass beads deposited under gravity flow; Area deposited: 60mm X 60mm, Total deposition time: ~120 seconds, Nozzle opening: 0.75mm **(b)** Single layer of a "Swiss Roll" microcombustor device deposited under gravity flow; Material: dyed red 90-106µm soda-lime, Nozzle openings: 0.75mm, Line Spacing: 2mm, Typical deposited linewidth: 0.8 mm, Deposition Speed: 25.4 mm/s, Deposition time: 30 seconds



(a) (b)

Fig. 16 (a) A single layer of a radial graded composition deposited under gravity flow; Material: soda-lime glass beads (90-106 µm) transitioning to Ti-6Al-4V (75-125µm), Nozzle opening: 0.75 mm, Line spacing: 0.75mm, Typical deposited linewidth: 0.8 mm **(b)** negative Poisson's ratio "bowtie structure" deposited with vibration assistance; Material: Nylon-6 (10-100 µm), Nozzle opening: 0.5 mm, Line spacing: 2.5mmX5mm(each half bowtie), Typical deposited linewidth: 0.2mm

CONCLUSIONS

A preliminary study of the flow of fine powders from small scale hoppers demonstrates that highly spherical particles in the size range 63-125µm may be delivered at continuous mass flow rate under gravity and the mass flow rates can be predicted by Beverloo's correlation with C=0.604 and k=2.86. The value of k might have a dependence on particle size, d, however this

dependence could not be deduced due to lack of necessary experimental data. For delivering powders below 63μm, alternate techniques need to be adopted. Gas pressure assistance can be used, but some problems are associated with it, e.g., powder spurting out with high velocity, and sporadic and unpredictable flow in the case of fine powders. Vibration assistance seems to have potential of delivering fine powders through small nozzle openings. This method also has an advantage of achieving automatic valving without any additional mechanism.

The linewidth model can be used to predict the widths of lines for powders with particle sizes in the 63-125μm range deposited under gravity through a nozzle opening in the range of 0.75mm-2mm.

The 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 further investigation of vibration assisted flow, developing a numerical model for granular flow processes, and developing more sophisticated model to predict linewidth.

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