A Method for Predicting Powder Flowability for Selective Laser Sintering

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This work investigates a method for pre-screening material systems for selective laser sintering using a combination of revolution powder analysis (RPA) and machine learning. To develop this method, nylon was mixed with alumina or carbon fibers in different wt.% to form material systems with varying flowability. The materials were measured in a custom RPA device and the results compared with as-spread layer density and surface roughness. Machine learning was used to attempt classification of all powders for each method. Ultimately, it was found that the RPA method is able to reliably classify powders based on their flowability, but as-spread layer density and surface roughness were not able to be classified.

INTRODUCTION

As additive manufacturing (AM) becomes more widespread, emphasis has been placed on speeding up development of new materials that offer functionality beyond what is currently available. This work will focus on one AM process, selective laser sintering (SLS), which utilizes a powder feedstock to produce polymeric components. One method of producing complex materials with SLS is to employ an indirect approach where the feedstock is a mixture of melting and non-melting components. The melting component, typically a polymer, acts as a binder for the non-melting components, which add functionality to the composite. Typically, these composite materials undergo post-processing steps to achieve their desired properties after their geometric properties have been imparted by the SLS system. Developing new materials for indirect SLS is especially challenging due to interactions between the constituent materials that affect their performance in SLS.

Currently, a trial-and-error approach is taken when developing new indirect SLS materials. Fully testing a new material in a commercial SLS system requires multiple kilograms of material, a requirement that can be costly and time-intensive. Therefore, a pre-screening process that can quickly determine a material’s suitability for SLS using small volumes of material can improve the development process. In this paper, relative density, powder flow, and compaction characteristics are considered as screening criteria.

This work began with the hypothesis that there exists a simple metric to determine a powder’s flowability and compaction characteristics prior to the SLS process. In other words, there is a link between this a priori metric and some physical characteristics of the as-spread powder in the SLS system. It turns out that this link is difficult to determine and that the current methods of pre-screening SLS material do not explain the differences in manufacturability of different materials.

BACKGROUND

A pre-qualifying (or screening) step is often employed in powder-based manufacturing processes. The purpose of this measurement is to reduce amount of trial and error, and to expedite
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Amado et al. 3 proposed that powders for SLS have intrinsic and non-intrinsic properties. The intrinsic properties, such as melt temperature, are related to the chemical and physicochemical properties of the powder. Non-intrinsic properties, such as particle size, are more closely related to the pre-processing and production steps. In the case of SLS, non-intrinsic properties are those typically associated with the "dispersion, packing, and homogeneity" of each powder layer. The intrinsic properties of powders for SLS play a larger role during laser irradiation. Powder properties can be further categorized into static and dynamic. Static properties are those not altered by physical movement and/or do not change during the SLS process. Dynamic properties are either altered during the SLS process, or experience physical movement (Fig. 1).

Powder layer homogeneity and compaction are directly related to the surface quality and density of the final parts produced with SLS, and research suggests that powder flowability and compressibility are the key metrics for predicting this. 1 Unfortunately, a universal method of measuring these does not exist. On top of that, there is no clear correlation between methods, making comparison difficult.

A common measurement of powder compressibility is the Hausner ratio (Eq. 1), which relates the bulk to the tapped density of the powder, and is explained further in ASTM D7181. 8 However, the Hausner ratio does not closely resemble powder spreading in SLS, so some suggest that it may not be the best measurement. 6 Additionally, the tapped density can be difficult to perform with repeatability (it is strongly affected by the frequency and amplitude of tapping) and often has a high standard deviation. 9,10

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HR = \frac{\rho_{tapped}}{\rho_{bulk}}
\]

Van den Eynde et al. developed a powder flowability testing device which aims to more closely resemble the stress states during powder spreading in SLS. They used a modified Hausner ratio, called the packing factor (Eq. 2), which takes into account powder bed density. 6,11 However, the packing factor still uses tap density, an unreliable measurement. Verbelen et al. further utilized this device to analyze a number of commercial nylon powders for SLS. 5

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PF = \frac{\rho_{layer}}{\rho_{tap}}
\]

Amado et al. proposed the use of a revolution powder analyzer (RPA) to complement existing measurement techniques, as it closely resembles the dynamic powder-spreading process during SLS. Schmid and Siegelmeyer and Wollecke reviewed the various methods of measuring powder flowability, cohesion, and packing efficiency, 4,9 and found the RPA to have the lowest standard deviation, and, perhaps, be the best representation of spreading in SLS.

METHODS

This work combines existing pre-qualifying metrics with machine learning to investigate if a simple material-agnostic method exists to predict powder flow and compaction. Additionally, other pre-qualifying metrics from the literature are evaluated and compared to the RPA approach.

The RPA method consisted of putting 20 ml of material into a glass jar 55 mm diameter by 27 mm height. The jar was placed on a rolling mill and spun at 37 rpm. Side illumination was used to enhance the contrast at the powder–air interface, and a video camera was used to capture the data at 30 frames per second for 60 s. The video data were processed in ImageJ to extract the powder–air interfaces. Figure 2a presents a still image from the video data and Fig. 2b shows the powder–air interface that is extracted.

Flowability metrics were extracted from the RPA video data by fitting two lines to each powder–air interface, the left line containing the avalanching powder and the right line containing the fluidized powder, as seen in Fig. 2c. The fit data were broken down into 30-frame segments and fit statistics were
extracted from each segment. A total of 60 segments (1800 frames) were taken for each material. The key statistics measured from each segment were the mean and standard deviation of the slopes as well as the average root-mean-square error (RMSE) values. These 6 metrics were used to define the flowability of each data segment.

The 6 key metrics extracted from each RPA data segment were compiled into feature vectors and used in a quadratic support vector machine (SVM) to determine if the powders could be differentiated, as depicted in Fig. 3. Classification was performed in Matlab using 5-fold cross-validation.

The other pre-qualifying metrics investigated were: the Hausner ratio (bulk density and tapped density), packing factor (spread density and tapped density), pressed density, absolute density, and spread-layer surface roughness. Bulk density was measured by pouring 70 ml of powder through a funnel (30°, 4.7 mm opening) into a 100-ml graduated cylinder and measuring the powder's mass. Tap density was determined by taking the powder-containing graduated cylinder from above and tapping on a hard surface from a height of ~15 mm for 2 min at roughly 2 Hz. Pellets of each powder system were produced in a 1-in (c.25-mm) die press using 1000 ± 10 kgf, and their outer dimensions...
measured using a dial caliper. All masses were obtained using a balance with resolution of ± 0.001 g. True (absolute) density was measured using helium pycnometry (Quantachrome Ultrapyc 1200e, 20 psi purge, 3-run av.). Powder bed density was determined by spreading a 100-μm layer of powder onto a known volume using the counter-rotating roller in a 3D-Systems HiQ. The baseplate of known volume was placed on top of the balance with a resolution of ± 0.1 g. A process schematic for these metrics is shown in Fig. 4. Results for bulk, tapped, true, and pellet density are provided in Table I.

The as-spread surface roughness was measured using a Keyence VHX7000 optical profilometer.* Each powder was spread into a 500-μm deep pocket manually using a doctor blade (round-edge, 45° blade angle); six replicates of each powder were performed. Five surface roughness parameters were calculated using the Keyence software for the entire scanned area (57 mm²); mean height deviation (Sa), maximum peak-trough height (Sz), RMSE of height deviation (Sq), mean skewness (Ssk), and mean kurtosis (Sku). The surface roughness parameters were compiled into feature vectors and used in a quadratic SVM to determine if the powders could be differentiated. Classification was performed in Matlab using 5-fold cross-validation, similar to Fig. 3.

Two powder systems were investigated in this study: aluminum oxide + nylon, and carbon fiber + nylon. The alumina/nylon mixture was adapted from Deckers et al.12 with slight modification**. The carbon fiber/nylon mixture was designed as a proxy for carbon fiber-reinforced structural polymers. An array of powder mixtures was produced, each mixture with different weight percentages of polymer binder and structural material. For the remainder of this document, the names of the powder mixtures will be encoded as “wt.% structural component—wt.% nylon”. For example, 10C-90N indicates 10 wt.% carbon fiber and 90 wt.% nylon, and 80A-20N indicates 80 wt.% alumina, 20 wt.% nylon.

RESULTS

The RPA data show that there is a clear difference in flowability among the materials tested. Heat maps of the powder–air interface for two materials, 100N and 10C-90N, are given in Fig. 5. The color scale represents the percent likelihood that the interface passes through that pixel throughout the 60 s of RPA data. In both datasets, the material is more stochastic near the jar edges and more consistent near the center of the interface; however, the 100N material displays more uniformity than the 10C-90N material.

The box and whisker plots, Fig. 6, show that the different materials have different distributions for the 6 features, indicating that the material flow properties, as measured with RPA, are unique for each material.

Quadratic SVM results indicate that each video segment could be classified with an accuracy of 93.1%. The misclassification rate is given in the confusion matrix shown in Fig. 7, which shows that there is no misclassification based on material additive, i.e., no material with alumina additive was misclassified as one with carbon fibers, and vice versa. The high classification accuracy suggests that all the materials tested inherently have different flow characteristics; however, the classification

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*10 μm step size, ×200 magnification, 5 × 5 stitching (57 mm² total area)
**Dry-mixed in a high-shear blender (Chulux QF-TB159008) for 10 min; alumina: (Almatis A16 SG, d₅₀ = 0.5 μm), nylon: PA12 (ALM PA650 d₅₀ = 55 μm); mixture sieved to <250 μm
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Table I. Densities for powders in this study; the ± values correspond to one standard deviation

| Powder | Theoretical\textsuperscript{a} and literature values | Measured values |
|--------|-----------------------------------------------|----------------|
|        | True density [g/cc] | Bulk density [g/cc] | Tapped density [g/cc] | Hausner ratio | True density | Bulk density | Tapped density | Hausner ratio | Pellet density [g/cc] |
| 100N   | 1.02 | 0.46 | 0.50\textsuperscript{b} | (1.15-1.23)\textsuperscript{a,b} | 1.06 | 0.47 ± 0.02 | 0.52 ± 0.02 | 1.11 ± 0.04 | 0.65 |
| 60A-40N | 2.75 | 0.83 | 0.99 | 1.19 | 1.94 | 0.64±0.01 | 0.78±0.05 | 1.23±0.06 | 1.12±0.02 |
| 80A-20N | 3.32 | 0.96 | 1.16 | 1.21 | 2.47 | 0.73±0.01 | 0.91±0.05 | 1.25±0.06 | 1.45±0.01 |
| 90A-10N | 3.61 | 1.02 | 1.24 | 1.22 | 3.29 | 0.82±0.01 | 1.07±0.10 | 1.31±0.06 | 1.68±0.03 |
| 100A   | 3.90 | 1.08 | 1.32\textsuperscript{c} | (1.15-1.23)\textsuperscript{a,b} | 3.88 | 1.06±0.04 | 1.29±0.06 | 1.21±0.10 | 2.02 |
| 100N   | 1.02 | 0.46 | 0.50\textsuperscript{e} | 1.22 | 1.22 | 0.47±0.02 | 0.52±0.02 | 1.11±0.04 | 0.65 |
| 10C-90N | N/A | N/A | N/A | N/A | 1.18 | 0.39 | 0.47 | 1.21 | 0.63 |
| 25C-75N | N/A | N/A | N/A | N/A | 1.29 | N/A | N/A | N/A | N/A |
| 50C-50N | N/A | N/A | N/A | N/A | 1.57 | 0.48 | 0.61 | 1.28 | 0.68 |

\textsuperscript{a}Weighted sum, e.g. \( \rho_{\text{mixed}} = \rho_1 \times \omega_1 + \rho_2 \times \omega_2 \)

\textsuperscript{b}Van den Eynde et al.

\textsuperscript{c}German and Bose

\textsuperscript{d}De Oliveira et al.

\textsuperscript{e}Van den Eynde et al.

Fig. 5. Powder–air interface heat map for (a) 100% nylon and (b) 90% nylon with 10% carbon fiber. The color scale represents the percent likelihood that the interface passes through a given pixel (Color figure online).
does not suggest anything about quality of flow, only that different materials can reliably be differentiated.

Despite visual indications that each powder provided a different as-spread surface roughness (Fig. 8), attempts to classify the powders using a quadratic SVM were unsuccessful (< 62.5% accuracy). Figure 9 shows box and whisker plots for the surface roughness parameters, showing that many metrics are overlapping for the different materials. For example, a Sz measurement of 0.4 mm could be from any of the materials tested. The overlapping nature of these measurements helps to explain why classification was unsuccessful.

The most common flowability and compaction metrics in the literature were also investigated: the Hausner ratio (bulk density and tapped density), packing factor (spread density and tapped density), and pressed density. These results are shown in Fig. 10 and Table 1. Accurate powder classification was not possible with any of these metrics, indicating that, despite different flow and compaction characteristics for each powder, these methods do not have a fine enough resolution to be used for reliable pre-qualification for SLS.

**CONCLUSION**

The RPA method was shown to be sensitive enough for pre-qualification of powder for SLS, reliably classifying materials using a very low volume of material. However, a link from the RPA metrics to a physical quantity such as the as-spread layer density or surface roughness was not possible. Without linking the RPA data to a physical indicator of powder flowability, the results will not be able to predict a material’s suitability for SLS. The RPA method presented here does show promise as an SLS material screening process, and could be used
Fig. 7. Confusion matrix for classifying powders using a quadratic support vector machine.

Fig. 8. Surface roughness of various nylon/CF powders. (a) 100 wt.% nylon, (b) 10 wt.% CF/90 wt.% nylon, (c) 25 wt.% CF/75 wt.% nylon, (d) 50 wt.% CF/50 wt.% nylon.
Fig. 9. Surface roughness parameters for CF/nylon powders.
in the future with a subjective classification of suitable as-spread layers, or, possibly, an objective classification using alternative imaging techniques of the powder surface.

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CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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