CHARACTERIZATION OF SELECTIVE LASER SINTERING™ MATERIALS TO DETERMINE PROCESS STABILITY

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Abstract

The Selective Laser Sintering™ (SLS) process has proved to be an excellent method for prototyping functional parts out of engineering thermoplastics such as polyamides. However, the material undergoes physical and chemical changes due to repeated heating cycles in the SLS equipment. This causes variations in powder characteristics and performance in the SLS process. With the increased utilization of SLS for direct manufacturing it is necessary to develop a characterization and testing system that can determine powder fitness to ensure process stability and part quality. Current powder recycling methodologies use an average virgin-to-used powder mixture. In a new approach, a testing mechanism to deliver a numerical, measurable material characterization will be discussed. Experimental results of repeated reuse of material and its resulting physical effects on mechanical properties, shrinkage, and chemical tests will be presented. A definitive testing and measurement process control will be shown to improve process stability and thus part quality and consistency.

Introduction

The 3D Systems Corporation, Valencia, CA, manufactures and sells Selective Laser Sintering™ (SLS) equipment and supplies powdered materials for use in this equipment. DuraForm™ is a polyamide polymer with excellent mechanical properties and is used to produce functional engineering models and prototypes. 3D Systems purchases this powder from a third party and supplies it to customers.

3D Systems and its customers have observed two different problems with DuraForm™ powder since its introduction: batch-to-batch variation in virgin material and progressive variation of powder performance after repeated use and reblending. As the SLS process transitions from functional prototyping and is incorporated into direct manufacturing processes, part quality and consistency is becoming a more important factor.

In an ideal process, all of the used powder could be recycled to achieve 100 per cent utilization. However, just as in injection molding, the repeated heat cycles change material characteristics resulting in part quality issues. Starting with virgin powder, the builds are very good with excellent surface finish and feature definition. As the powder is reused, the surface finish degrades and at some point becomes unacceptable. Therefore, virgin powder is blended into the mix in an attempt to keep the part quality at an acceptable level. SLS system users have generally blended their powders in the following manner:

1. Run a build.

2. Break out the parts, segregating the unsintered powder that can be easily broken up as “used” powder.
3. Sift the used powder in a vibratory sifter
4. Blend in a weight percentage of virgin powder and mix via mechanical methods.

The weight percentage varies from user to user and material to material. For DuraForm™ users generally blend 25 to 33 percent virgin powder. Powder management and blending is a critical part of the SLS process due to the high cost of SLS powders on a per pound basis. Blending too much virgin powder into the process raises costs and blending too little increases the chance of unacceptable surface finish and part detail.

Objective

The University of Louisville Rapid Prototyping Center proposed to investigate physical and chemical characteristics and mechanical property changes of DuraForm™ powder. The goals of this investigation were to identify those characteristics that correlate with poor performance and to develop an evaluation protocol that will ensure consistent, good performance. From those results it was desired to develop a cost effective, efficient measurement and testing procedure for blending that will provide a metric that can aid in the process control of SLS as a production process.

Methodology

Representative Parts

A build packet for a 3D Systems SLS2000 was created that utilizes the full build volume and the entirety of the powder supply pistons. A number of different parts were chosen to highlight surface finish in all three axes, fine feature detail, scale and offset parameters, and tensile specimens. A total of 48 parts were in the build. The entire build packet is depicted in Figure 1. The overall build height including warm-up was 11.9 inches.

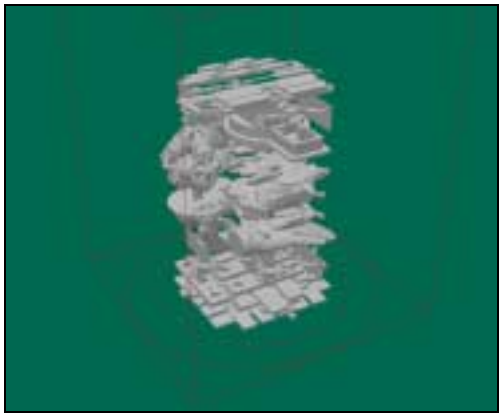

Figure 1. SLS2000 Build Packet

Processing and Powder Acquisition

The initial step in the process was to determine the best operating parameters for the virgin powder: laser power, feed temperatures, part bed temperature, and piston heater temperature. These critical parameters would remain constant for all of the future builds to

provide consistent heat input to the powder. These parameters can vary from machine to machine.

Three containers of DuraForm™ from the same lot were selected for the trial. A sample of the virgin powder was taken and labeled. The SLS2000 feed cartridges were loaded with powder. The feed rate was such that the build packet would use the entire supply of powder, thereby exposing it all to a single heat history. The complete build was run and all parts were broken out. All of the unsintered powder was broken up and sifted through a vibratory sifter. The overflow cartridge powder was added to the part cake powder and mechanically mixed thoroughly. A sample of this mix was taken and labeled.

The resultant remaining powder was put back through the SLS process using the same build packet. The volume of powder used by the parts decreased the amount of powder available for the second build. Therefore, the build would not complete the full height. The build decrease in height was approximately 12.5 percent. The build was run until the build terminated due to lack of powder. Again, the parts were removed from the part cake and labeled. All powder was sifted, mixed, and a sample was taken and labeled.

This process was repeated until the build conditions deteriorated or there was too little powder remaining. This was a total of seven consecutive builds for DuraForm™.

Physical and Chemical Testing

Several tests were planned for the part samples and powder specimens. The interest was in examining the possible correlation between part quality and several test criteria to indicate material stability and degradation in the process.

Melt-Index determination: Melt-index was measured using an extrusion plastometer according to ASTM D1238. This index is a measure of the flow characteristic of the molten polymer and is sensitive to differences in the basic polymer structure due to changes in molecular weight. Changes in melt-index should correlate with changes in the build characteristics of DuraForm™ during the laser sintering process.

Differential scanning calorimetry: Differential Scanning Calorimetry (DSC) was used to compare the melting and solidification behavior of the powders. Detailed run parameters were established during preliminary testing and included a 100% nitrogen environment, heating at constant rate to a set temperature above the melting point of the material and cooling at a fixed rate to room temperature. The resulting thermal scans are compared in order to identify differences in features such as melting or solidification onset temperature, melting or solidification range and heat of fusion.

Mechanical properties: Mechanical properties of test bars of the materials were measured using an Instron test machine. Measurements included tensile yield strength, rupture strength and elongation. Five standard ASTM tensile specimens were built with the long axis oriented along the X direction and five along the Y direction.

Surface appearance and detail: A subjective evaluation was used to compare similar parts on a run-by-run basis. The evaluation was used to determine the lowest acceptable part quality for a user. This result gave the baseline for end-use testing.

Results

Melt Index

The melt index testing was conducted using ASTM standard procedures for nylon materials. A temperature of 235°C and a weight of 2.16 kg were used for the DuraForm™ material. The results are shown in Figure 2. It can be seen from the graph that the viscosity of the material decreased with repeated heat exposures from the SLS process. The melt index test is a good indicator of changes in molecular weight. In this test, the results indicate an increase in the molecular weight of the material with multiple heating cycles. In injection molding of thermoplastics, the melt index will generally increase with multiple thermal exposures indicating a decrease in molecular weight.

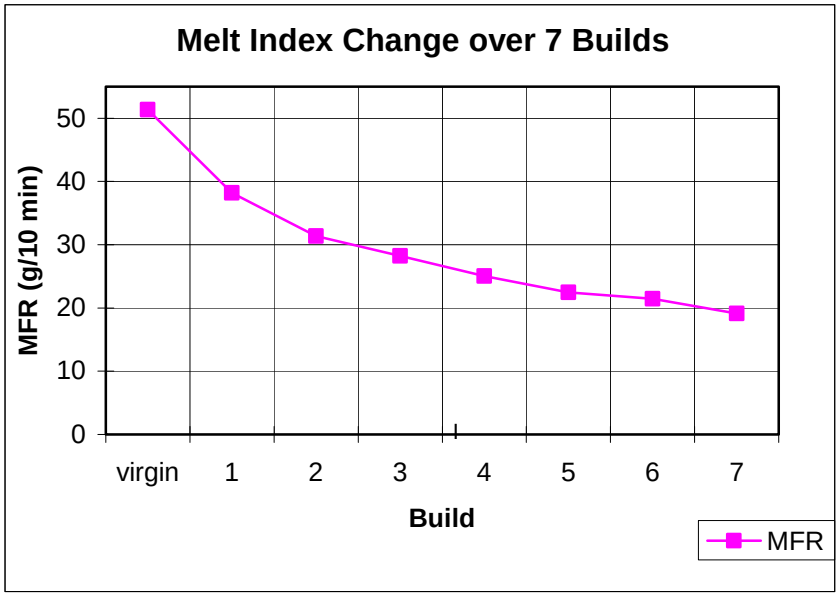

Figure 2. Melt Index Results

Differential Scanning Calorimetery (DSC)

Approximately 10mg of the sample powder is deposited into the chamber of the DSC equipment and the chamber is purged with nitrogen gas. The temperature is equalized at 75°C and then ramped at five degrees per minute to 225°C. Figure 3 shows a representative DSC output curve illustrating the melt point and the recrystallization points. The melt points for all of the runs are shown in Figure 4. The trend of the curve indicates that the melt point is increasing with multiple heat exposures.

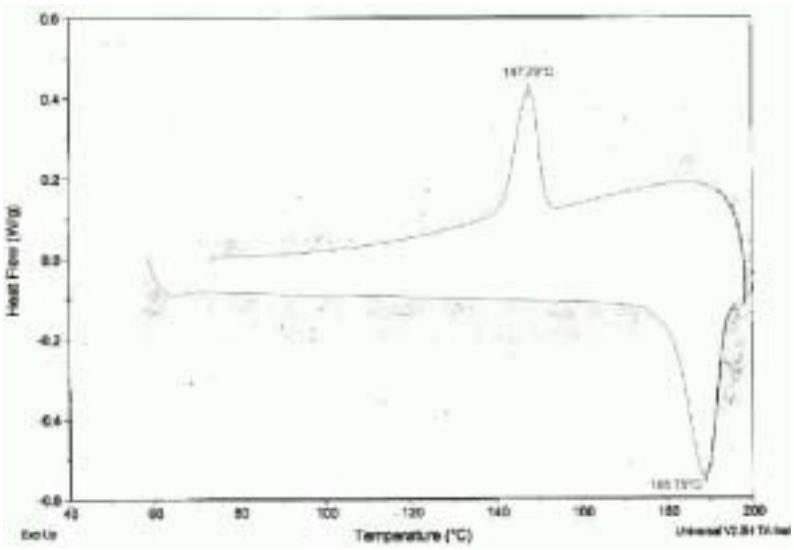

Figure 3. DuraForm™ DSC Curve

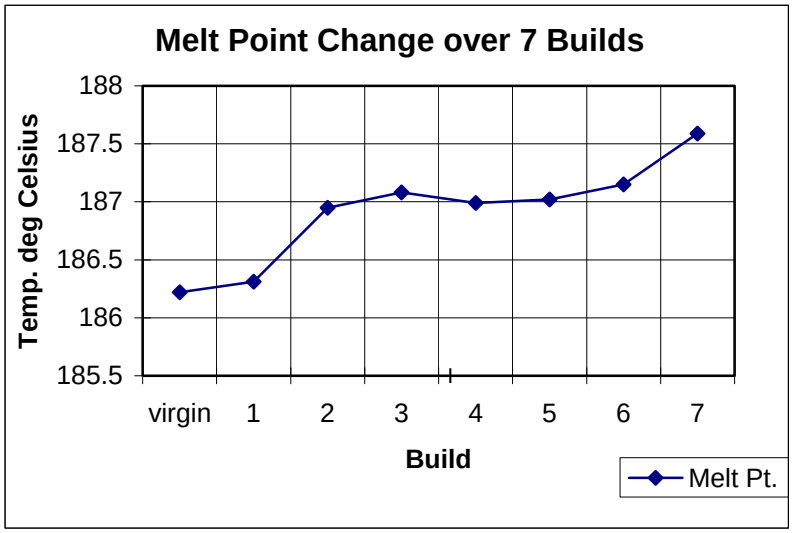

Figure 4. Melting Point

Mechanical Properties

Tensile strength and elongation results are shown in Figure 5. The tensile strength increases slightly over the first five builds. However, there is an approximately 25 per cent drop off in tensile strength at the sixth build. The tensile bars aligned along the Y-axis exhibited slightly greater tensile strength.

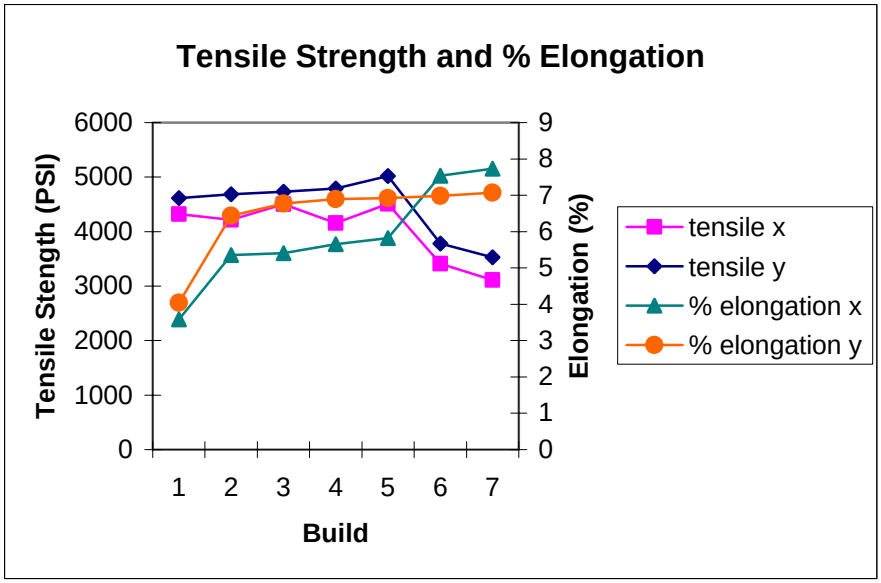

Figure 5. Tensile Strength and Elongation

Surface Finish

The evaluation of the surface finish was performed as a qualitative assessment. A surface finish commonly called “orange peel” is a known phenomenon that is associated with over used or degraded powder. The determination of an acceptable surface finish varies from user to user. Figure 6 shows parts with good and bad surface finish. The right view is a classical “orange peel” finish. Visual evaluation of parts from the seven builds indicate that the surface finish is excellent for the first three builds, acceptable for the next two builds, and unacceptable after that.

Figure 6 Part Surface Finish Comparison

Scale and Offset

Scale and offset test coupons were run in each build to track dimensional changes that can occur as the powder ages. This is an important test to understand if dimensional inaccuracies

are occurring due to variations in powder quality. The parts were measured and the results input into a scale and offset worksheet supplied by 3D Systems to track these values. Figure 7 is a graph showing the changes over the seven runs. The scale in the X direction varies from 2.8% to 3.4% with the Y scale varying from 3.0% to 3.5%. The offset ranges from 0.0057 to 0.0022 inches and from 0.0037 to 0.0005 inches in the X and Y directions, respectively.

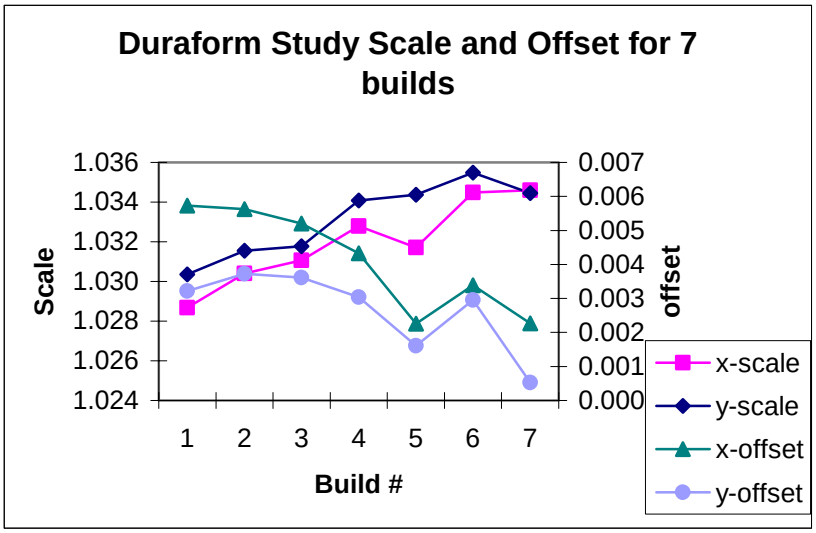

Figure 7. Scale and Offset Values

Conclusions

The most notable conclusion from these sets of results is that powder variation on a run-to-run basis can significantly impact the quality of the produced parts. While some variation may be acceptable for prototype parts, it is an unacceptable state for direct manufacturing. This fact alone illustrates the need for an effective powder management and testing system to control critical parameters.

The combined indications of the melt index test and the differential scanning calorimeter suggest a change in the polymer chemistry of the DuraForm™ powder exposed to multiple heat cycles. The decrease in the melt index value points to an increase in the molecular weight. Combine that with the increase in the melt point from the DSC equipment and it appears as if there is a limited "cross-linking" of the material similar to what might be seen in some thermoset material systems. While the DuraForm™ is a known nylon-based thermoplastic, it appears as if there is some lengthening of the polymer chains. This is the opposite of what normally occurs in many thermoplastics during the injection molding process. Repeated heat cycles or over exposure due to barrel residence times generally causes a decrease in molecular weight and an increase in the melt index. The material properties suffer accordingly.

The challenge was to find a cost effective method for tracking the fitness of the DuraForm™ powder. It is desired to have consistent powder properties in the direct manufacturing process to allow process temperature controls and scale and offset values to remain constant. The melt flow rate equipment has proved to be a good predictor and provide a metric by which to track the powder and check its condition.

The first requirement is to define a target melt index parameter at which to run the SLS process. This value can be extrapolated from the test builds by evaluating the surface finish of the parts produced during the runs. The physical strength of the part, tensile strength and elongation, does not fall off until after a point most users would consider the surface finish to be unacceptable. In addition, by using the melt index as the control, the user can control their surface finish at a specific level depending upon needs and part use.

Tracking the melt index after each individual build has shown a range of degradation during the build. Builds that have smaller cross sectional scans per layer show a smaller change in the melt index compared to "hotter" builds that have longer scan times and thus longer exposure of the powder to the heaters. Therefore, the amount of virgin DuraForm™ to be added to achieve target melt index has varied from 15 to 40 percent. Blending curves must be generated to create a method for determining the exact percentage of new material by weight to be mixed with the used material. Starting with used powder and adding varying amounts of virgin powder and then retesting for the melt index accomplished this. By populating a dataset with numerous blends it was possible to create a blending curve and resulting equations for a specific target melt index.

Since the inception of this testing procedure, the University of Louisville Rapid Prototyping Center (RPC) has seen very consistent results. Part quality has been reproducible build-to-build and the scale and offset numbers have remained constant. It has been a worthwhile tool to for incoming inspection of material and for determining the quality of unknown lot of used DuraForm™ powder. For example, if the melt index testing shows that more than 50% of virgin DuraForm™ is required to reach the target value, it can be discarded. Previously a visual, touch and feel method was used to find out which powder lots to discard. By tracking the mechanical properties and scale and offset numbers with each build, the SLS equipment may also be benchmarked. A small drift in the scale and offset was noticed, with no change in the melt index. Normally, the material would be blamed. Since the material quality was known, it was determined that it was a hardware issue with the equipment. This can assist in tracking the capability of the process and the machine.

As the SLS process continues to transition into the new realm of direct manufacturing of functional plastic parts, methods for controlling the process parameters are required. For the DuraForm™ material, the melt index has proved to be a good indicator of material fitness and a control parameter for the process. Additional work is currently in progress to determine similar testing for other SLS materials such as glass-filled DuraForm™.

Selected References

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