

Selective Separation Shaping of Polymeric Parts

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Abstract

Additive manufacturing (AM), or 3D printing has enjoyed a recent surge of attention over the past decade. AM is a process in which digital 3D design data is used as input to build physical objects by combining sequence layers of material. By increasing demand in use of additive manufacturing for fabrication of end-user parts, there is considerable interest in developing new techniques which can offer high quality customized parts at low cost. Selective Separation Shaping (SSS) is a new AM technology developed with the goal of fabricating low cost, high resolution 3D parts. The main advantage of SSS is that this process enables building fully functional pieces without the need of any intermediate binder or high cost laser operation. This process has been primarily applied to metallic, and ceramic materials and test cases were successfully built. There has been no study on fabrication of parts using polymeric material and the goal of this research is to examine successful fabrication of polymer parts. Nylon 6,6 has been used as starting base material and several test cases were fabricated to identify key factors in success of this process. Different classes of nylon are studied to achieve better understanding of material properties on success of fabrication and achieve an effective binding between layers. Finally, 3D printed parts built by SSS are presented.

Introduction

Selective Separation Shaping (SSS) was invented at University of Southern California by Professor Behrokh Khoshnevis [1]. The main advantages of SSS over other commercialized 3D printing approaches are its affordable cost, flexibility to work with multiple materials and its application for large scale fabrications. In this approach, part is selectively separated from its surroundings within a thin wall of barrier material called as separator (S-powder). Like other powder-based additive manufacturing processes, initially a uniform layer of base powder is delivered onto the build tank. This material is called as base material (B-powder). After one layer is paved, a nozzle is inserted into the base powder and printing process starts. In this step, the nozzle which is composed of a miniature needle moves along the contour of part. As this movement is initiated inside the B-powder, a vibrational stimulator is applied to deposit S-powder through the opening of the needle into the gaps. Subsequently, another layer of powder is spread on previous layer and process continues until all layers are processed shown in Figure 1.

In opposed to majority of other AM techniques, the nature of SSS enables fabrication of wide classes of material. Any material in powder form that can be solidified upon heating such as ceramics, metal, and polymers or upon applying curing agents such as cement (lime based), sorrel cement (magnesia based) and gypsum based composites can be applied. Moreover, unlike common additive manufacturing processes, a different tool path method is required for SSS since this process only considers part surfaces and disregards the part core. Nozzle rotation is also programmed into the toolpath which traces the contours of slices. This rotation helps obtain the most effective filling of gaps along the path.

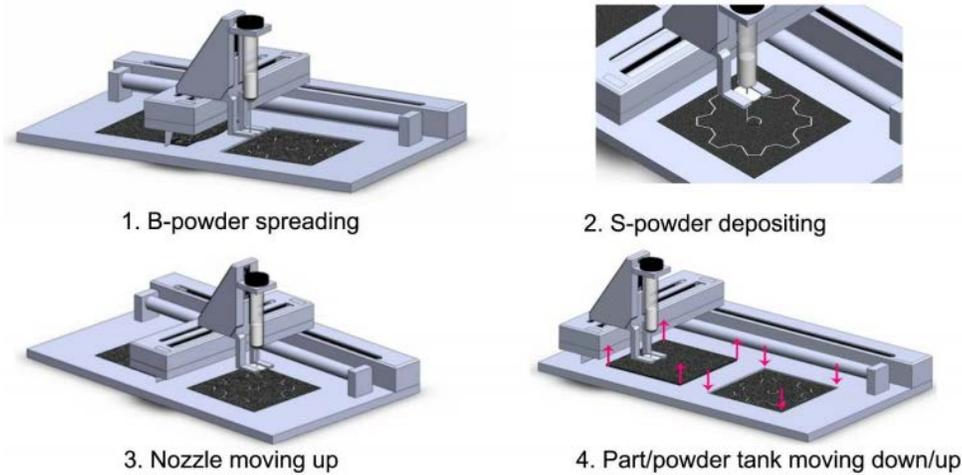


Figure 1. SSS process steps [1]



Figure 2. Test cases fabricated by SSS. Metal on top row; Bronze (left and middle), Steel (right). Ceramic parts on the bottom row; Portland cement (left) Lunar Regolith Simulant (middle) and Gypsum(right) [2,3]

Parts fabricated by SSS are supposed to hold similar mechanical properties as other additive manufacturing techniques. The complexity of this process, however, demands research on both process-ability of each material and their post-processing steps. In previous research efforts, metal and ceramic parts were built by this process [2,3] (Figure 2). The aim of this research is to expand application of Selective Separation Shaping for fabrication of polymeric parts. Table 1 summarizes the AM 3D printing fabrication methods for polymer fabrication. Prior to SSS, Selective Inhibition Sintering (SIS) process was developed and introduced early 2000 at USC [4]. In SIS process, the agent called as inhibitor is in liquid form and is deposited on each layer by means of commercial inkjet printer at the boundary profile. This will prevent base powder particles to fuse at treated sections. Several research studies were performed to propose a framework for selecting inhibitor for variety of base material [5,6]. Application of liquid agent in selective separation of parts which ideally makes the powder particles resistive to sintering under heat requires and in-depth understanding of underlying chemical reactions and is determined by base powder's chemical properties which is unfavorable. Instead of introducing a liquid separator, an alternative method is to employ an agent in solid form which can function as a physical barrier in this process. This method further expands application of this technique to other material such as metal, steel, and cement.

Table 1. *AM methods for fabrication of polymer parts*

| AM technology | 3D printing medium |
|----------------------|-------------------------------|
| SLA | UV light beam/Laser |
| SLS | Laser |
| FDM | Extrusion |
| 3DP | Binder |
| DLP | Digital projector screen |
| DMLS | Laser |
| SHS | Heat |
| SLM | Laser |
| SIS | Separator deposition (Liquid) |
| SSS | Separator deposition (Solid) |

Experimental setup and procedures

The SSS machine which was initially fabricated for metallic parts [1-3]. In fabrication of metallic parts, bulk sintering of the entire powder volume after layer-wise treatment with S-powder is performed. Processing of polymers; however, is obtained by layer by layer sintering of powder under heat. The motions of the machine consist of XYZ axes and these movements are controlled by adjusting speed and position of each axes. Sensors are employed to send feedback signals to the controller to adjust initial and end positions. There is rotational movement to adjust the positioning of the needle relative to the movement direction.

The other controlled system on SSS is the deposition system which is composed of a piezo disk stimulated by wave generator. The wave length, shape and frequency of vibrations are controlled using the wave generator and triggering and stopping deposition is controlled by the controller software. Figure 3 demonstrated the SSS beta machine used for fabricating polymer parts. The layer by layer sintering system has been combined in this process for polymers. There are temperature controllers used to monitor temperature during fabrication.

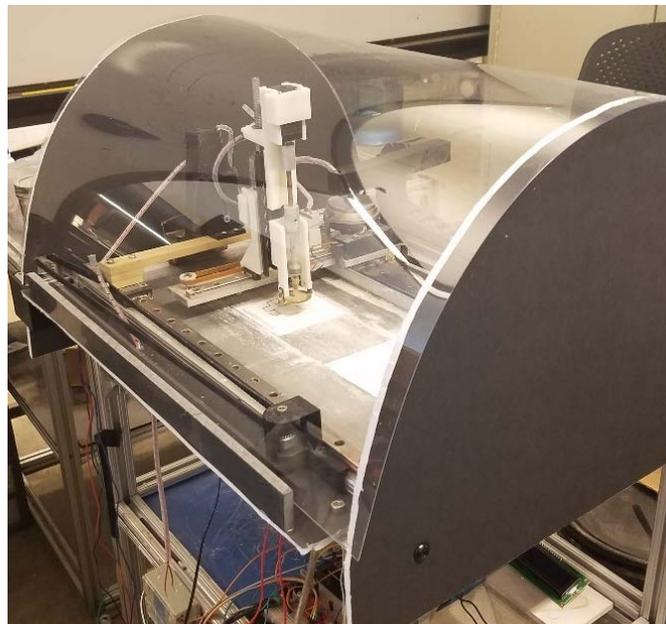


Figure 3. *SSS beta machine modified for fabrication of polymeric parts*

To avoid shrinkage due to sudden cooling down of layers, heat sheets are applied to the build tank and platform. Infrared lamps are as well used to maintain elevated temperature inside printing chamber. Before printing starts heaters are turned on and the tank temperature is raised to 70 C. The temperature inside the printing area should be also kept constant and results are satisfactory at 30 C inside the chamber.

Selection of base powder

Nylon (Polyamide) has been one of the most popular polymers applied in AM techniques due to its properties and relatively high mechanical strength [7,8,9,10]. We have focused on this class of material throughout this research work. Different types of Polyamide have been studied able to better characterize the impact of base material properties on quality of SSS parts. The goal is to determine the effect of properties of polymer on quality of final SSS parts. Figure 4 represents particle size distribution of Polyamides (PA) used in our experimental studies. SEM images for investigated polyamide powders are shown in same magnitude of x150.

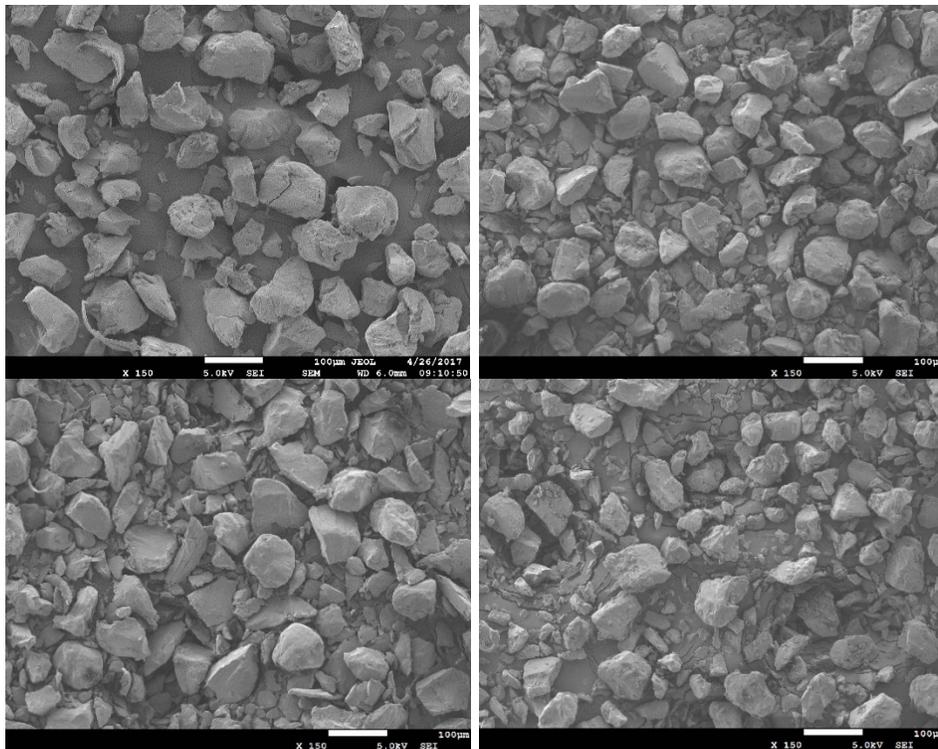


Figure 4. SEM images of Copolyamide(top left), Vestamelt 171(top right), Vestamelt 470(bottom left), and Vestosint(bottom right)

As it can be observed all types of polyamides have non-spherical shape with sharp edges (cryogenic milled particles). This is more significant in Vestamelt-470. Vestmalet 470 and Vestamelt 171 seems to have larger ratio of small size particles relative to other two types. In copolyamide, the distribution is more significantly skewed to the left as particles are mostly large except some very small particles.

Selection of separation powder

S-powder prevents polymer particles to coalescence under heat exposure on each layer. The inhibited boundary profile acts as a sacrificial mold that can be easily removed, and leaves a chemically pure part.

To choose an appropriate separator, several factors are considered. These include particle size and shape, density, melting temperature, decomposition temperature, flow ability (cohesion coefficient), and non-reactivity with polyamide material. Based on required properties, chromium oxide (Cr_2O_3) (Figure 5) was found to achieve followability, stability in high temperature, non-reactivity with polymer and availability in small particle size. The suitable range for S-powder in small scale fabrication is within the range of 20 to 90 micrometers diameter.

Table 2 presents material properties of the candidate separating material.

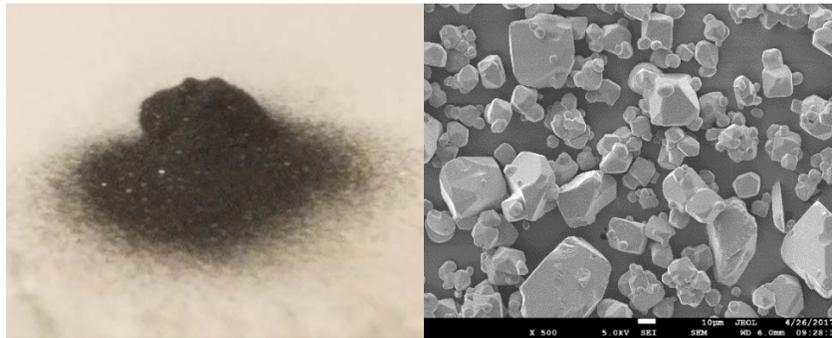


Figure 5. Chromium Oxide powder (left) and SEM image (right)

Table 2. Specification of separation powder

| | |
|--------------------------------|--|
| Chemical composition | Chromium Oxide (Cr_2O_3) |
| Sieve Analysis | 325 Mesh +20 microns |
| Melting Temperature | 2266 C |
| Density | 2.6g/cc |
| Hardness (Rockwell 15N) | 94 |
| Tensile Strength | 20.685 M Pa |

Effect of polymer properties on quality of layers

Differential scanning calorimetry is used to determine impact of melting and crystallization behavior of Vestosint, Vestamelt 470 and Vestamelt 171 on SSS processability. Samples 6 mg +/- 0.5 mgs are heated with 5 and 10 C/min up to 130 C followed by same cooling rate after heating is complete. The thermal properties of Copolyamide is provided by Arkema Co. Degree of crystallinity of all polyamide grades are calculated. The summary of the settings is shown in Table 3.

Table 3. *Thermal properties of polyamide grades*

| Polyamide | Glass transition temperature | Melting temperature | Enthalpy | Crystallinity (%) |
|------------------|-------------------------------------|----------------------------|-----------------|--------------------------|
| Vestosint | 46.76C | 120.29C | 40.6 | 0.15 |
| Vestamelt 470 | 50.43C | 102.90C | 19.9 | 0.07 |
| Vestamelt 171 | 53.73C | 112.70C | 19.5 | 0.07 |
| Copolyamide | 58 C | 182 C | 65.95 | 0.25 |

Among all grades of polyamide powders that were tested by SSS machine (Table 4), Vestamelt 171 and Copolyamide were successfully processed. Vestosint and Vestamlet 470 although showing advantageous material properties were not successfully processed due to high shrinkage during fabrication. Depending on base material properties, sintering and cooling rate, warpage of the layers can occur. In is also observed that as number of layers increases this phenomenon is more significant which can occur by penetration of heat into previously sintered layers and causing over-sintering. Moreover, depending on melting temperature(T_m) and crystallization (T_c) temperature this warpage can be delayed.

Table 4. *Polyamide processing by SSS*

| Polyamide | Disadvantage | Advantage |
|------------------|------------------------------------|---|
| Vestosint | High shrinkage | High strength, strong attachment between layers |
| Vestamelt 171 | Low strength | Low shrinkage, poor attachment between layers |
| Vestamelt 470 | High shrinkage | Medium strength, strong attachment between layers |
| Copolyamide | Powder spreading, High flowability | Low shrinkage, high strength, relatively strong attachment between layers |

It is observed that base material properties play a major role in success of fabrication in the SSS process. An ideal solution to this is to examine different polyamide grades and link their properties to expected performance in the SSS process. In this manner, through another set of experiments, effect of polymer properties on final density of parts is examined. All polyamide grades were exposed to radiation system within particular distance as applied in the SSS machine. ImageJ platform has been used for image processing and percentage porosity of layers for four polymer grades under same infrared heating treatment have been calculated.

Table 5. *Polyamide processing by SSS*

| Polyamide grade | Method | Distance from exposure | Avg. particle size | Percentage porosity |
|------------------------|---------------|-------------------------------|---------------------------|----------------------------|
| Vestosint | Infrared | 4.5mm | 139.81 | 2.72 |
| Vestamlet 171 | Infrared | 4.5mm | 150.52 | 17.03 |
| Vestamelt 470 | Infrared | 4.5mm | 180.52 | 26.45 |
| Copolyamide | Infrared | 4.5mm | 155.61 | 16.22 |

It is observed that Vestosint offers lowest percentage of density compared to two other grades that were successfully processed. However, application of this powder in SSS process was limited due to high percentage of shrinkage. Vestamlet 171 and Copolyamide both offer %16 density on single layer.

Test cases were fabricated and it was observed that Copolyamide parts obtain higher density and stronger attachments between the layers. Figure 5 shows top, middle, and bottom layers of 2.5 gears fabricated by Vestamlet 171 and Copolyamide. This is a representation of gradual change for these polymer parts as we look at layers sintered successively.

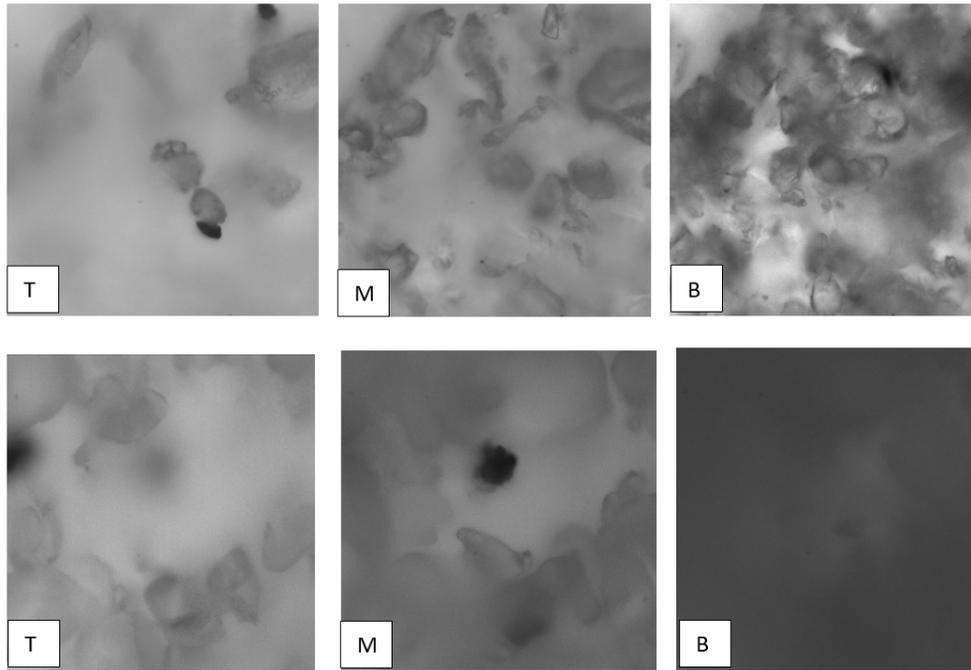


Figure 5. Top, middle, and bottom layers of samples at 40 times magnitude for Vestamelt 171 (top row) and Copolyamide(bottom row)

As we approach bottom layer for each sample, we achieve different levels of densities. Vestamlet 171, shows higher level of porosity compared to Copolyamide. Although all types of polymers show similar particle shapes and relatively close size distribution, it is observed that crystallization behavior plays major role in characterizing their performance in the SSS process. As the window between melting point and crystallization for type of polymer increases, less shrinkage is observed and found most suitable for SSS processing application.

Preliminary results

In this set of experiments more complex geometries are built by SSS machine. A series of test cases are designed to investigate the bonding strength, the different bonding mechanism. Figures below demonstrate samples made by Copolyamide and Vestamelt 171.



Figure 1. Test case built by Vestamelt 171 (left) and 2.5 gear fabricated by Copolyamide

Conclusion and future research

Selective Separating Shaping is a 3D printing process that has a great potential to offer fast speed, fine quality 3D parts. There is still high demand for 3D printing process that can work in small-medium size business and offer low cost parts while maintaining part quality. This platform enables fabricating multiple class of materials which further expands its application to other areas. This process is not only limited to small scale fabrications but also it has potential for fabricating on demand large scale structures. For polymeric part fabrication, due to the operation principle in SSS (printing on the boundaries instead of the part body), this process has a great potential to compete with other commercialized processes such as FDM and SLS in printing speed. Multiple nozzles can print at once on platform without interfering with one another to build multiple objects simultaneously. Compared to other powder-based polymer processes, the end-user product cost will be low as this process does not require laser scanning. The separating material can be purchased at low cost. In addition to that, variety of polymers can be applied to the machine and tested in powder form. In this research work, the impact of particle size, shape of base powder, as well as degree of crystallinity on final parts' density and overall shrinkage are studied. In preliminary experiments, samples are exposed to radiation and it is observed that crystallization is the key factor in success of fabrication in SSS process. Applicability of each polymer can be rapidly evaluated before testing in the SSS system using this approach. For future research directions, other than altering base material properties, sample of same polymers can be treated with radiation for varying lengths of time. The cohesiveness and mechanical strength of final part can serve as an indication of efficacy of sintering process. Finally, the best setting can be achieved by linking each polymer to its best suited sintering method.

References

- [1] Khoshnevis, Behrokh, and Jing Zhang. "Selective Separation Sintering (SSS)-An Additive Manufacturing Approach for Fabrication of Ceramic and Metallic Parts with Application in Planetary Construction." *AIAA SPACE 2015 Conference and Exposition*. 2015.
- [2] Zhang, Jing, and Behrokh Khoshnevis. "Selective Separation Sintering (SSS) A New Layer Based Additive Manufacturing Approach for Metals and Ceramics." *Solid Freeform Fabrication Symposium*. 2015.
- [3] Zhang, J., and B. Khoshnevis. "APPLICATION OF SELECTIVE SEPARATIONSINTERING IN CERAMICS 3D PRINTING." *Advanced Processing and Manufacturing Technologies for Nanostructured and Multifunctional Materials II: Ceramic Engineering and Science Proceedings, Volume 36 6* (2015): 151.
- [4] Khoshnevis, Behrokh, et al. "SIS—a new SFF method based on powder sintering." *Rapid Prototyping Journal* 9.1 (2003): 30-36.
- [5] Nouri, Hadis, and Behrokh Khoshnevis. "Study on Inhibition Mechanism of Polymer Parts in Selective Inhibition Sintering Process."
- [6] Khoshnevis, Behrokh, Mahdi Yoozbashizadeh, and Yong Chen. "Metallic part fabrication using selective inhibition sintering (SIS)." *Rapid Prototyping Journal* 18.2 (2012): 144-153.
- [7] Goodridge, R. D., C. J. Tuck, and R. J. M. Hague. "Laser sintering of polyamides and other polymers." *Progress in Materials Science* 57.2 (2012): 229-267.
- [8] Verbelen, Leander, et al. "Characterization of polyamide powders for determination of laser sintering processability." *European Polymer Journal* 75 (2016): 163-174.
- [9] Yusoff, W. A. Y., et al. "Influence of molecular weight average, degree of crystallinity, and viscosity of different polyamide PA12 powder grades on the microstructures of laser sintered part." *MATEC Web of Conferences*. Vol. 26. EDP Sciences, 2015.
- [10] Shi, Y., et al. "Effect of the properties of the polymer materials on the quality of selective laser sintering parts." *Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials Design and Applications* 218.3 (2004): 247-252.