Research on relationship between depth of fusion and process parameters in lowtemperature laser sintering process

T. KIGURE*, Y.YAMAUCHI*, T. NIINO†

*Tokyo Metropolitan Industrial Technology Research Institute †Institute of Industrial Science, the University of Tokyo

Abstract

Model of low-temperature laser sintering, in which part warpage during process is prevented by anchoring of parts instead of high-temperature preheating, is discussed. Low-temperature laser sintering process allows powder bed temperature to be lower than those in normal laser sintering process which suppresses parts warpage by preheating powder bed above its recrystallization temperature.

When we introduce a new process or material, many experimental examinations are required to decide adequate building conditions. To reduce this process, theoretical process modeling and simulations are carried out. In stereolithography, relationship between laser irradiance and cure depth is known as "working curve," and used for fundamental equation for this technology. On the other hand, many theoretical models for laser sintering have been introduced, and most of them are thermal models dealing with heat transfer in powder bed. Contrarily, there are few reports concerning measurement and calculation of fusion depth though fusion depth can be obtained easily by experiment and working curve is a useful to determination of building parameters.

In this study, working curve which represents relationship between part thickness obtained by monolayer scan and incident energy was investigated. As a result of normalizing the power by the minimum power that can melt the surface of the powder bed, all the plots lay on the same single line. This line, namely master curve, is unique for each powder and useful to finding various parameters.

Introduction

Previously, the authors introduced a modified laser sintering process, namely lowtemperature process which suppresses part warpage during process by anchoring parts to a rigid plate. This process allows powder bed temperature to be lower than those in normal laser sintering process, which suppresses parts warpage by preheating powder bed above its recrystallization temperature. Up to now, it is reported that this technology can improve material recycle rate [1] and expand range of applicable materials [2] [3].

When we introduce a new process or material, many experimental examinations are required to decide adequate building conditions. To reduce this process, theoretical process modeling and simulations are carried out [4-7]. Pryre et.al [8] has achieved a simulation of fusion depth of PA12 and PEKK in single scan with a theoretical model incorporating experimentally measured penetration depth. Franco reported experimental research to obtain relationships between laser power and fusion depth, width in single scan [6].

In stereolithography, relationship between laser irradiance and cure depth is known as "working curve," and used for fundamental equation for this technology. The relationship is based on lambert-beer's law, according to which irradiance H at the depth z in powder bed can be expressed as

 $H(z) = H_0 e^{-z/D_p},$

where H_0 a D_p and irradiance at bed surface and "penetration depth," respectively. D_p is physical quantity corresponding to the depth at which initial irradiance is attenuated by 1/e. Using this law relationship between cure depth of photopolymer C_d and total incident energy per unit area of the resin surface E as

$$C_d = D_p \ln\left(\frac{E}{E_c}\right),\tag{2}$$

where E_c is critical energy per unit area, which is the minimum value allowing resin surface curing[9]. This plays an important role for determining building parameters as a fundamental equation for stereolithography [10].

Up to now, many theoretical models for laser sintering have been introduced, and most of them are thermal models dealing with heat transfer in powder bed. Contrarily, there are few reports concerning measurement and calculation of fusion depth though fusion depth can be obtained easily by experiment and working curve is a useful to determination of building parameters.

The goal of this study is providing a useful method to find the optimal parameter set for low temperature process. For the first step, in this research, the relationship between fusion depth and laser power is investigated. Thicknesses of parts processed by monolayer scan in various conditions were measured to find a common rule namely "master curve."

Material and Method

Material and Machine

Vestosint[®] X1556 (Dicel-evonic) was used in this study. This powder is based on Polyamide 12 and blended for laser sintering. Powder material properties are shown in Table 1. Experiments were performed with fresh powder without thermal degradation.

Melting point	Recrystallization	Particle	Bulk density	Specific heat		
	temperature	diameter(D50)	Durk density			
182°C	144°C	55µm	0.45g/cm ³	1.26J/g K		

Table1. Powder material properties

A commercially available laser sintering machine, RaFaEl300C[®] (Aspect Inc.) was employed. This machine is equipped with roller recoater and CO₂ laser. Laser spot diameter at powder bed surface is 350μ m.

Experimental method

It is known that part size and scan strategy affect fusion of powder very much [11]. To avoid the noise caused by variation of these parameters, all specimens were built in the same size and with the same scan strategy. Scan parts size is $20 \text{mm} \times 10 \text{mm}$ rectangular. Laser scan direction is +*X* and -*X* and lase sweep direction is only +*Y* direction as shown by Figure 1.



Figure 1. Scan strategy and measuring point

Powder bed temperature was set at 70°C, 100°C and 130°C, which are lower than recrystallization temperature of the material. Scan interval was set at 0.05mm, 0.10mm and 0.15mm. These parameters are summarized in Table2. Thickness of each part was measured at the middle of the solidified region with a micrometer to obtain solidification depth.

01						
Powder bed temperature	Layer thickness	Scan interval	Laser power	Scan speed		
130°C 100°C 70°C	0.1mm	0.05 mm 0.10 mm 0.15 mm	0.5 ~ 28.0W	2000 mm/s		

Table2. Building parameter set

Fundamental equation

Assuming that Lambert-Beer's law is applicable to attenuation of the ray in powder, the fundamental equation of stereolithography is applied to laser sintering as

$$D_F = D_p \log\left(\frac{E}{E_c}\right),\tag{3}$$

where D_F is fusion depth of powder. E_c for laser sintering is energy required to raise powder bed temperature to its melting point. When a powder with a thickness of D_F is melted and solidified, thickness of obtained plate, T_s , is thinner. In addition, bulk density of powder ρ_b is smaller than the density of the plate. Assuming that the density of the plate is true density, ρ_t , by material melting, we can describe solidification thickness, T_s as

$$T_{s} = \delta_{b} D_{F} = \delta_{b} D_{p} \log \frac{E}{E_{c}},$$

$$\delta_{b} \triangleq \frac{\rho_{b}}{\rho_{t}},$$
(4)

where δ_b is relative bulk density of powder. Introducing relative penetration thickness T_p , T_s is written as

$$T_{s} = T_{p} \log \frac{E}{E_{c}},$$

$$T_{n} \triangleq \delta_{p} D_{n}.$$
(5)

Total amount of supplied energy to unit area of powder bed surface E_T is written using scan interval S_p and laser scan speed V_s , and laser power P as

$$E_T = \frac{P}{V_s \times S_p}.$$
(6)

Taking thermal dissipation during process we introduce coefficient of dissipation α which depends on scan speed and scan interval. α is ratio of total energy which was consumed in powder melting to total amount of supplied energy. Then total energy to powder bed per unit area *E* can be written as

$$E = \alpha E_T. \tag{7}$$

Substituting equation (6) and equation (7) to equation (5), we obtain following equation.

$$T_{s} = T_{p} \log \frac{P}{P_{c}}$$

$$P_{c} \triangleq E_{c} V_{s} P_{s}$$
(8)

Here we define P_c as critical laser power, which gives critical energy, E_c . Here we defined working curve as a diagram for relationship between solidification thickness and laser power, which is described by equation (8).

Experimental result

Figure 2 shows specimens obtained by single layer scan. Powder bed of temperature and scan interval were set at 130°C and 0.1mm, respectively. Three laser powers, 2W, 10W and 20W were tested. Figure 3 and 4 shows micrographs of cross sections and surfaces of the specimens out of corresponding laser powers, respectively. Thickness of the specimen increased as laser power increased. When laser power was greater than 10W, grains were melted so much as to be fused completely, though sticking grains were still existing when power is low at 2W. Spherical voids were found in upper region of specimen when laser power was 20W.



Figure 2. Specimen shapes obtained by single layer scan. Powder bed temperature and scan interval was 130°C and 0.1mm, respectively.



Figure 3. Cross sections of specimens. Powder bed temperature and scan interval was130°C and 0.1mm, respectively.



Figure 4. Surface of specimens. Powder bed temperature and scan interval was130°C and 0.1mm, respectively.

Figure 5 and 6 show working curves for different powder bed temperatures and scan intervals. In Figure 5, working curves with the same powder bed temperature are plotted in the same chart. Contrarily, working curves with the same scan interval are plotted in the same chart in Figure 6.

All the curves were similar in shape and parallel to each other. They differed in position

depending on temperature and scan interval. Solidification thickness increased as temperature increased or as scan interval decreased.



Figure 5. Working curves for different powder bed temperatures of 70°C (a), 100°C (b) and 130°C (c).





Laser power [W]

Figure 6. Working curves for different scan interval of 0.05mm (A), 0.10mm (B) and 0.15mm (C).

Discussion

Working curves

Each working curve can be divided into three sections. The first one is in the range where laser power is relatively low. In this range, grains are not melted completely. The second one is in the range where laser power is in middle range. In this range, grains are melted completely and high part density is obtained. The third one is in the range where laser power is the highest. In this range, decomposition of the plastic occurred, and generated gas expands the parts to increase solidification thickness. In the following discussion, we deal with the data from the second section only.



Figure 7. Classifications of dominant phenomenon in the working curve

Penetration Depth

According to (5), we can obtain penetration depth of powder bed from the gradient of each working curve. Since all the curve are parallel, as mentioned in the previous section, they provide common D_p . Using parameters summarized in table 2, we can obtain D_p of 315.4 μ m.

Table 2. Parameter for calculation for penetration depth

Bulk density ρ_b	True density ρ_t	Relative penetration thickness T_p
0.45g/cm ³	1.03g/cm ³	137.8µm

Critical laser power

Critical power, P_c , is equivalent to x-intercept of each working curve. Since obtained working curves have different positions, their x-intercepts are different, and we obtain various critical powers, resultantly. Figure 8 shows the relationship between P_c and powder bed temperature. The relationship for the same scan interval was linear. Reason for this relationship can be explained as flowing. The amount of energy that is required to start powder bed melting is sum of heat of fusion and one for heating the bed to its melting point. Since the latter one is proportional to difference between temperature of bed and melting point, P_c and powder bed temperature are in linear relationship. Extrapolation of each line crosses the x-axis at the same bed temperature of 184°C. This temperature is equivalent to melting point. This result shows quite trivial fact that powder bed starts melting without laser irradiation when powder bed is hotter than melting point.



Figure 8. Relationships between powder bed temperature and critical power.

Master curve

In Figure 9, all the working curves are plotted in one chart with their laser power normalized by P/P_c . We can find that all the plots lie on the same line. The line always cross the x axis at "1," and gradient of the line represents the relative penetration thickness. Although, a single scan speed was used in this research the gradient is supposed to be the same even if we change the scan speed. Therefore, we can call this common line or curve as "Master Curve." The master curve provides property concerning absorption laser by powder, which helps us with finding parameters such as layer thickness. Unfortunately, solidification thickness varies very much with other conditions such as scan interval and scan speed.



Normalized laser power P/P_{c}

Figure 9. Relationships between powder bed temperature and normalized power

Conclusion

We discussed working curve for laser sintering which represents relationship between thickness of a part that is obtained by monolayer scan and supplied energy. For the parameter that represents energy supply, we did not choose total energy supply but laser power. Additionally, by normalizing the power with the minimum power that can melt the surface of the powder bed, all the plots were lay on the same single line. This common line can be called as master curve. This master curve is available for determination for optimal building parameter set in low temperature process.

Acknowledgements

This research is a part of "Strategic Innovation Promotion Program" supported by Cabinet Office, Government of Japan.

Reference

[1] T. Kigure, T. Niino, "Improvement of recycle rate in laser sintering by low temperature process", Proc. Solid Freeform Fabrication Symposium 2017 (2017) 550-556

[2] F. Ito, T. Niino, "Implementation of Top Hat Profile Laser into Low Temperature Process of Poly Phenylene Sulfide", Proc. Solid Freeform Fabrication Symposium 2016 (2016) 2194-2203
[3] T. Niino, U. Takashi, "Low temperature selective laser melting of high temperature plastic powder" Proc. Solid Freeform Fabrication Symposium 2015 (2015) 866-877

[4] G. Bugeda, M. Cervera, G. Lombera, "Numerical prediction of temperature and density distributions in selective laser sintering processes", Rapid Prototyping Journal, **5**,1 (1999) 21-26

[5] L. Dong et al, "Finite element analysis of temperature and density distributions in selective laser sintering process" Materials science forum, **553** (2007) 75-80

[6] A. Franco, L. Romoli, "Characterization of laser energy consumption in sintering of polymer based powders." Journal of Materials Processing Technology, **212** (2012) 917-926

[7] T.H.C. Childs et al, "Simulation and experimental verification of crystalline polymer and direct metal selective laser sintering." Proc. Solid Freeform Fabrication Symposium 2000,

(2000) 100-109

[8] P. Peyre et al, "Experimental and numerical analysis of the selective laser sintering (SLS) of PA12 and PEKK semi-crystalline polymers", Journal of Materials Processing Technology, **225** (2015) 326-336.

[9] P.F. Jacobs, "Fundamentals of stereolithography." Proc. Solid Freeform Fabrication Symposium 1992 (1992) 196-211

[10] J.G. Zhou, D. Herscovici, C.C. Chen. "Parametric process optimization to improve the accuracy of rapid prototyped stereolithography parts", International Journal of Machine Tools and Manufacture, **40** (2000) 363-379.

[11] Y. Yamauchi, T. Kigure, T. Niino, "Influence of process time and geometry on part quality of low temperature laser sintering", Proc. Solid Freeform Fabrication Symposium 2017 (2017) 1495-1505