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Improvement of recycle rate in laser sintering by low temperature process

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Abstract

Laser sintering process selectively solidifies its powder bed to obtain designated parts and leaves the rest unsolidified. When the remained powder is recycled, a certain amount of fresh powder is added to moderate the effect of deterioration by preheating in the previous batch. In terms of economy, improvement of the recycle rate of used powder is one of the big challenges. The authors are developing a novel laser sintering process that prevents part being processed from warping not by preheating the powder bed but by anchoring the parts to a rigid base plate. Since the new process, namely low temperature process, can lower the bed temperature than normal high temperature process, it is expected to reduce deterioration of the used powder. In present research, processablity of recycled powder is evaluated by its MFR, and it is shown that operation at a high recycle rate more than 90% is possible.

Introduction

In laser sintering process, a part of powder bed is selectively solidified by laser irradiation to obtain designated parts. Rest of the bed, the powder that remains unsolidified when a process for one batch is finished, is reused in the following batch. Exposed to high temperature, the unsolidified powder is deteriorated [1], and using the powder directly in the next batch leads to defects such as rough surface known as “orange peel.” To moderate the effect of material deterioration, used powder is mixed with a certain amount of fresh powder. Since material cost for one batch is equivalent to the cost for added fresh powder, the amount of the additional powder directly impacts material cost of part production. Thus, reduction of additional material is a big challenge in practical use of laser sintering technology. Generally, rate of additional fresh powder, refresh rate, in feed stock is 30% to 50%. [2, 3] On the other hand, in most cases, the volume occupied by parts in a bed is only 10% or less of the whole powder bed due to difficulty in part arrangement. Thus, amount of the powder to be purchased becomes three to five times of part volume. If repeated use of material at refresh rate of 10% can be, it becomes the amount of newly added material is almost equivalent to the amount of material consumed as a product.

In deterioration of powder during process, powder bed preheating plays a dominating role. In laser sintering, powder bed temperature is maintained in a range between melting and recrystallization temperature, which is called “process window.” Within the process window, the melted material stays in supercooled liquid state so that thermal stress that can be a cause of part warpage is suppressed. On the other hand, this powder bed preheating causes deterioration of the material.

The authors are introducing a novel laser sintering process that prevents parts from warping by anchoring them to a rigid base plate. Since the process, namely “low temperature process,” does not utilize supercooling phenomenon, it is not necessary to maintain the powder bed temperature within the process window during process. This unnecessity of tight temperature control [4-6] allows process temperature to be less than crystalizing temperature, and low bed temperature prevent the material deterioration leading to improvement of recycle rate.

The aim of this research is to access effectiveness of low temperature process in improvement of powder recyclability. Recycle powder with a refresh rate of 10% was tested. Several times of build tests were performed repetitively as the powder is regenerated after each build. Performance variation of powder and build parts are evaluated. MFR of powder material was also measured after each build to evaluate degradation of powder. For evaluation of part quality, relative density and surface roughness were used.

Material and Method

Material

PA12 powder (Vestosint®, daicel-evonik) was employed. This powder is developed for use in laser sintering. Serial tests started with brand-new fresh powder. After each test is finished, used powder from the bed which was collected from only part-cake was sieved and mixed with brand-new powder at a mixture rate of 9:1 to obtain powder stock for the next batch. DSC measurement result of the powder material is shown in Figure1. The melting point is 184 °C, the peak temperature of recrystallization is 144 °C. True density of the material is 1.03g/cm^3

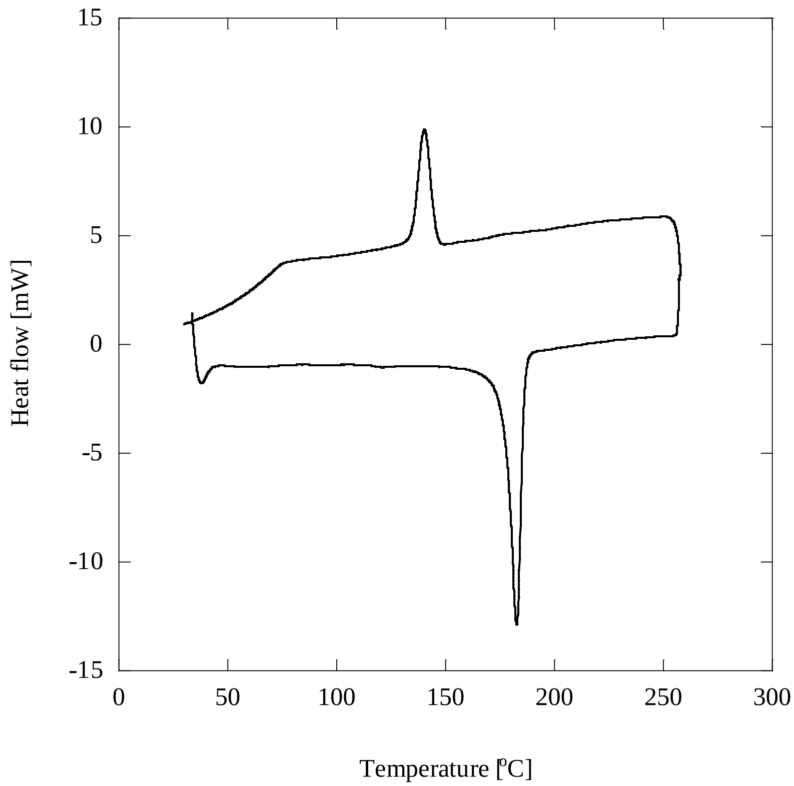

Figure 1. DSC curve of vestosint® fresh powder
(Heating rate: 10°C /min. Holding at 260°C for 5 min. Cooling rate: 5°C /min .)

Process Parameters

A commercially available machine (RaFaEl300C®, ASPECT) was used. Process parameter set is shown in Table 1. The parameters of low temperature process are obtained by the preliminary test as those that provide the highest density as a primary index of mechanical strength. Powder bed temperature of 130°C for low temperature process is lower than the recrystallization temperature of the powder.

Table 2 shows building time and data height of each process in this research. The building time was set to be roughly the same between low temperature process and high temperature process. Each process was repeated 5times. Figure 2 shows build layout of each process. In all builds, parts were arranged so as to minimize mutual thermal effect.

Table 1. Processing parameter set

Building process	Powder bed temperature	Layer thickness	Scan interval	Laser power	Scan speed	Energy per unit area
Low temperature	130°C	0.1mm	0.10 mm	12.0W	2.0 m/s	60.0 mJ/mm^2
High temperature	170°C	0.1mm	0.14 mm	24.5W	10.0 m/s	17.5 mJ/mm^2

Table 2. Build time and data height of each process

Number of cycles	Low temperature process		High temperature process	
	Time	Height	Time	Height
1	3h06min	9mm	3h13min	40mm
2	3h05min	9mm	2h28min	28mm
3	3h22min	9mm	3h11min	40mm
4	3h00min	9mm	2h39min	28mm
5	3h00min	9mm	2h02min	18mm

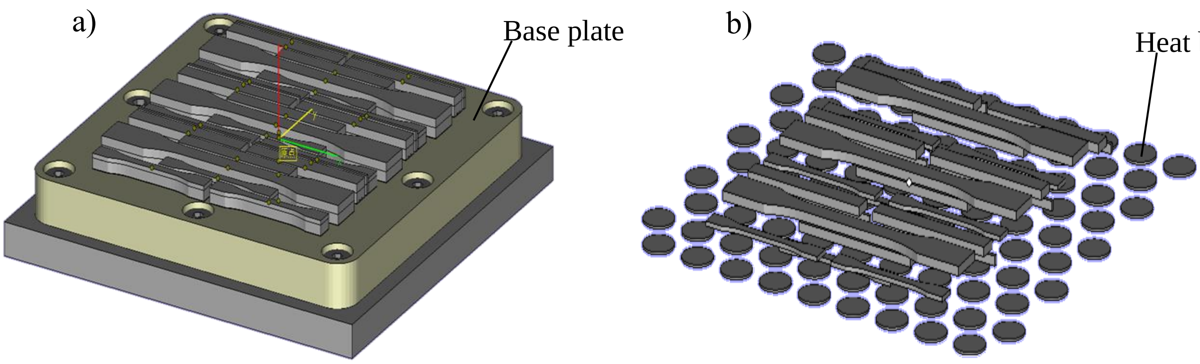

Figure 2. Build layout a) low temperature process b) high temperature process

Base plate

A base plate to anchor the parts on was employed to prevent them from warping. It was a flat plate of the same material manufactured by high temperature process. To ensure the flatness of the anchoring surface, it was back-upped with rigid aluminum palate which was tightly fixed to the plate by screws as shown in Figure 3.

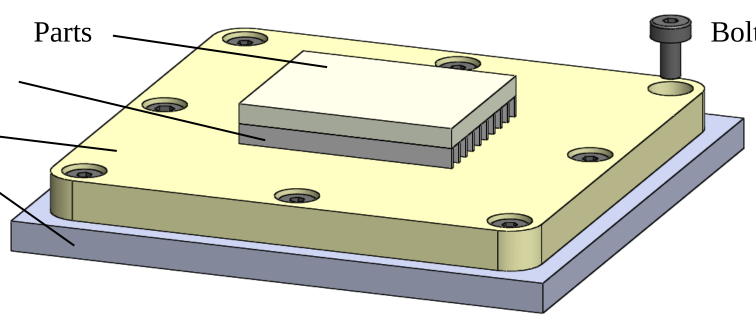

Figure 3. Base plate

Parts density

Since relative density of a parts has a strong correlation with its mechanical strength, part quality provided by each powder condition was evaluated by the density. Densities of cubic specimens with nominal dimensions of 80mm×10mm×4mm were measured by Archimedes method using following equation.

$$\rho_{parts} = \frac{m_{air}}{m_{air} - m_{water}} \rho_{water}$$
,

where m_{air} is weight of a part in air and m_{water} is weight in water, and ρ_{water} is density of the water.

Surface roughness

Since rough surface of parts is a major defect often found when we use degraded material, surface roughness of upper surface of parts was measured. Using 3D laser microscope (OLS4000, Olympus), surface profile in the range of 1.8 mm × 1.8 mm was measured at a magnification of 432 times. Arithmetic mean height S_a was used as evaluation index of surface roughness.

Melt Flow Rate (MFR)

It is well known that MFR of powder material decreases as quality of the material is degrading [1, 2, 7]. Unsolidified powder was collected from the part-cake after process, and MFR was measured by melt flow tester (MELT INDEXER P-101, Toyo seiki) at a load of 2.16kg and temperature at 235°C.

Experimental result

Density

Figure 4 shows the relative densities for each process cycles. Density was higher in low temperature process for all the process cycles. In the both of low temperature and high temperature processes, density decreased as process was repeated in general. The decrease rate of the relative density obtained by low temperature process was smaller.

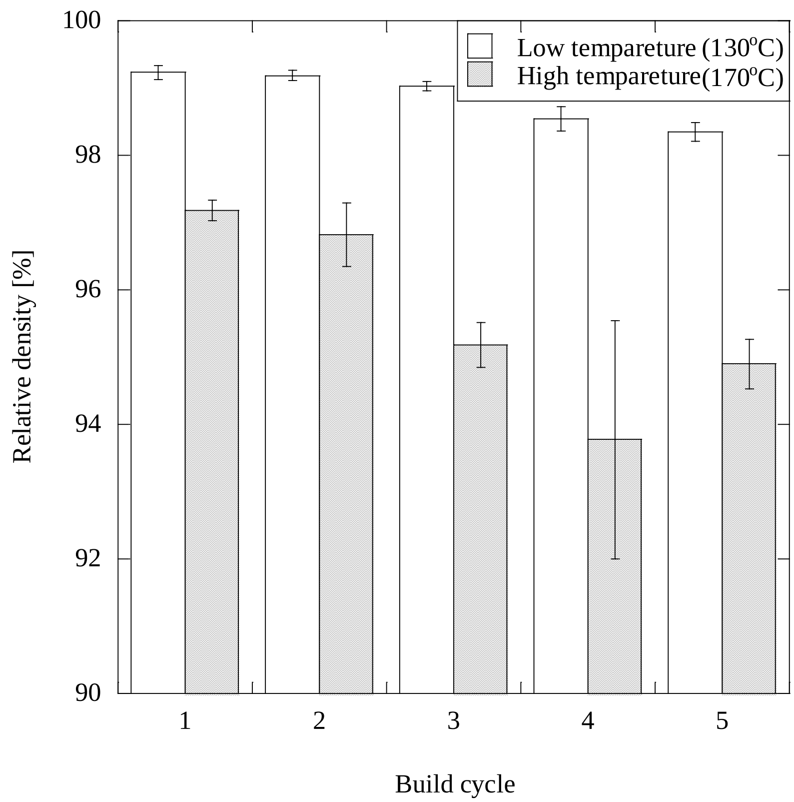

Figure 4. Relative density of each build cycles

Surface roughness

Figure 5 shows the measured arithmetic mean height S_a. Roughness of part from high temperature process showed larger irregularity and saturates after cycle 3. Roughness of low temperature processed part increased until cycle 4 and decreased at cycle 5. For the process roughness was doubled after several processes.

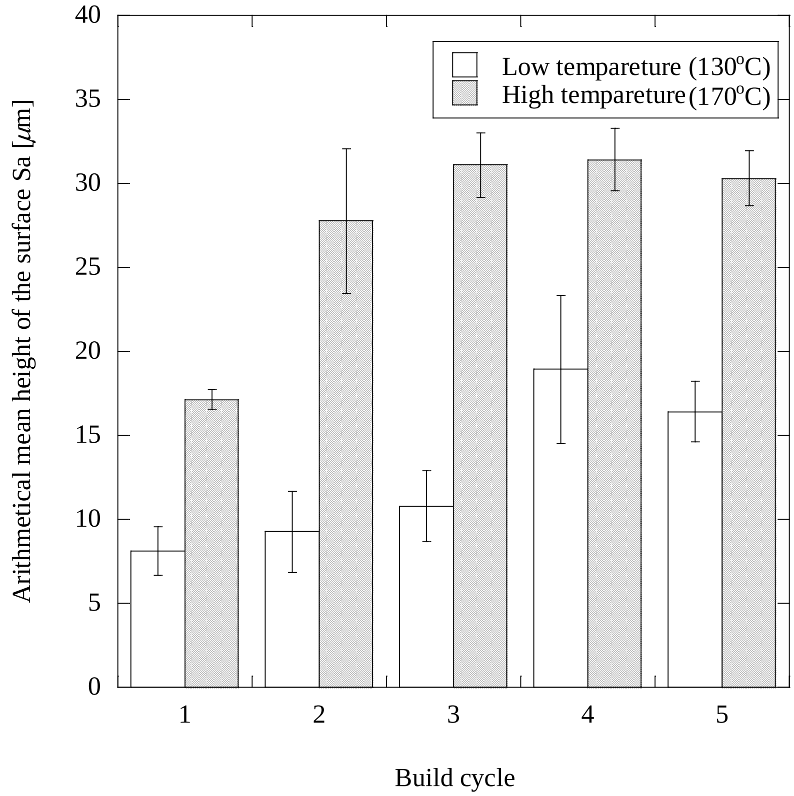

Figure 5. Arithmetic mean height S_a of each build cycles

Melt Flow Rate

Figure 6 shows variation of MFR. Low temperature reduced degradation of MFR in general. MFR degraded very much after 1st cycle, but decrease became negligible after cycle 3. MFR of powder material used in low temperature process decreased by a factor of 35% while MFR decrease of high temperature processed powder was 52%.

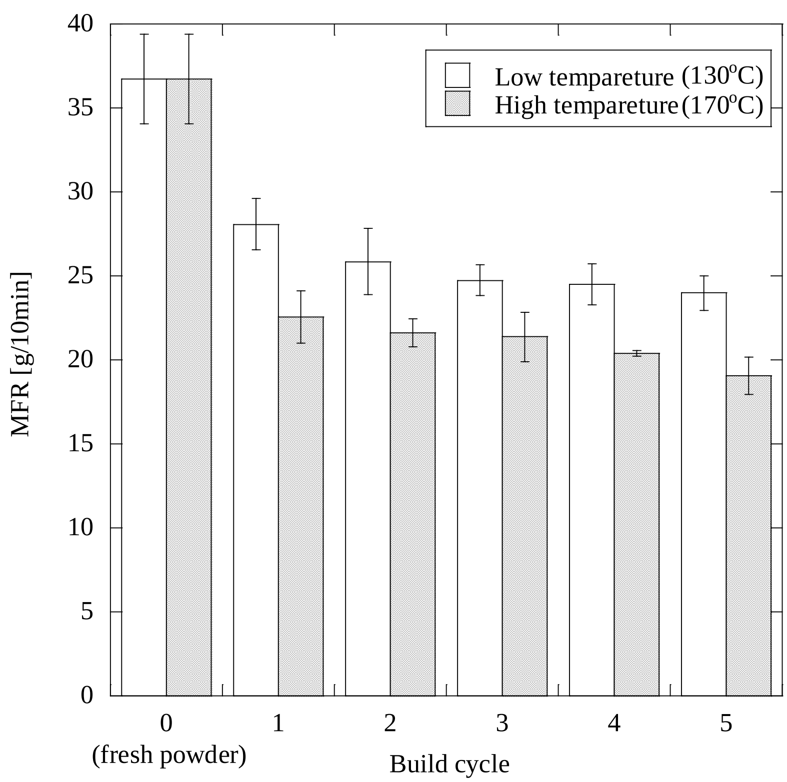

Figure 6. MFR of unmetled powder in each build cycles

Discussion

Reduction of part density as an index of mechanical property was less than 1% after processed 5 times. This result shows that recycle rate of 10% enough for low temperature process in terms of mechanical performance. Contrarily, density drop of high temperature process was greater than 2%.

Surface roughness was doubled after processed 5 times for the both of low and high temperature processes. Effectiveness of using low temperature process in variation of surface roughness is not clear. However, absolute value of surface roughness obtained by low temperature process was a much smaller than high temperature process for all process cycles. This reason is assumed as follows. Surface of a melted part of bed is relatively smooth owing to surface tension and gravity force. However, in high temperature process, the surface is roughen again when additional powder is poured for the next layer since it is adhered on the surface which is staying unsolidified in supercooled state. On the other hand, in the low temperature process, the surface once flattened by the tension and the gravity force does not adhere additional powder since the surface solidifies as soon as laser irradiation is finished. The increase in surface roughness due to process repeating may be due to the increased viscosity of the melt as Wudy *et al.* suggested [8].

The decrease in MFR became negligible after cycle 3. This is similar trend to 32% and 50% refresh rate in the high temperature process and indicate effect of mixing refresh material [3,7]. In the MFR of high temperature process in this research, there is no effect of mixing of fresh materials.

Degradation of MFR after process cycles is smaller for low temperature process than high temperature process. This indicated that powder bed temperature lower than recrystallization temperature suppressed effect of heat in material deterioration. According to the heating experiment by Dotchev *et al.*, they are shown that the value of MFR clearly differs depending on the temperature and higher MFR leads to better product quality [2]. This support that low temperature process can reduce material deterioration, and the better part quality derives this suppressed material damage.

Conclusion

In this research, improvement of material recycling rate of laser sintering using low temperature process was discussed. The process was repeated five times powder was regenerated at a refresh rate of 10%. Degradation in part density as an index of material property became less than 1% after the repeated processes and saturated. In terms of mechanical performance, we can conclude low temperature process is quite effective for improvement of powder recyclability. Surface roughness was increased by a factor of two in the both of low and high temperature process. Although we did not find advantage of using low temperature process in prevention of degradation in surface finish, low temperature process always provided better surface quality than high temperature process. Low temperature process could not suppress drop of MFR after the first process using brand-new powder, but the decrease became negligible after the third process. This shows that 10% of refresh rate is enough in terms of material deterioration for low temperature process.

We can conclude that low temperature process can eliminate cost multiplication issue due to low recyclability.

Acknowledgements

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Reference

[1] Gornet et al, "Characterization of selective laser sintering materials to determine process stability", Proc. Solid Freeform Fabrication Symposium 2002 (2002) 546-553

[2] Dotchev et al, "Recycling of polyamide 12 based powders in the laser sintering process," Rapid Prototyping Journal, **15** (2009) 192-203

[3] Josupeit et al. "Material properties of laser sintered polyamide 12 as function of build cycles using low refresh rates", Proc. Solid Freeform Fabrication Symposium 2015, (2015) 540-548
[4] Niino et al, "Low temperature selective laser melting of high temperature plastic powder," Proc. Solid Freeform Fabrication Symposium 2015, (2015) 866-877
[5] Ito et al, "Implementation of tophat profile laser into low temperature process of Poly Phenylene Sulfide," Proc. Solid Freeform Fabrication Symposium 2016, (2016) 2194-2203
[6] Yamauchi et al, "Low temperature laser sintering of PA powder using fiber laser," Proc. Solid Freeform Fabrication Symposium 2016, (2016) 2204-2216
[7] Wudy et al, "Influence of Degradation Behavior of Polyamide 12 Powders in Laser Sintering Process on Produced Parts", AIP Conference Proceedings, **1593** (2014) 691 - 695
[8] Wudy et al, "Aging behavior of polyamide 12: Interrelation between bulk characteristics and part properties", Proc. Solid Freeform Fabrication Symposium 2016, (2016), 770-780.