Solid Freeform Fabrication 2022: Proceedings of the 33rd Annual International Solid Freeform Fabrication Symposium – An Additive Manufacturing Conference
Reviewed Paper

Investigation into effect of beam defocusing in low temperature laser sintering of PEEK

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Abstract

Laser sintering of PEEK with the same powder bed temperature as PA12, which is lower than recrystallization temperature of PEEK, has been achieved at pervious study. However, the process at low bed temperatures requires a greater energy supply to melt the material, and it increases the risk of thermal decomposition. Expansion of spot size by defocusing is one of the effective ways to reduce the peak intensity of beam and allow providing more energy to the same laser irradiation range as avoiding the decomposition. In this study, specimens were prepared with the same amount of energy and difference beam diameters, to observe difference in internal voids and thermal decomposition of specimens. As a result, it was indicated that large spot size is effective on high energy supply without thermal decomposition and suppressing internal voids.

Introduction

Polyetheretherketone (PEEK) is one of the highest performing plastics with excellent heat resistance, chemical resistance, and high strength. Due to these properties, PEEK is used in automobiles, aerospace, and biomedicine, and is expected as a metal alternative material [1]. In addition, this material can be used as a feedstock material in material extrusion [2][3] and laser sintering (LS) of additive manufacturing technology. [4][5]

Laser sintering process requires preheating the powder bed to near the melting temperature of the feedstock material. Since the melting temperature of PEEK is about 340 °C, the powder bed temperature of about 300 °C is required in laser sintering of PEEK. This temperature is above the melting temperature of most plastic parts. Therefore, laser sintering of PEEK can be processed only with a high temperature resistant machine. But this machine requires high cost, such as large energy resource for keeping high powder bed temperature and high price of machine and material. This is one of the factors that hinder the spread of laser sintering of PEEK. It is a challenge of achieving laser sintering of PEEK at low cost.

The authors propose modified laser sintering process, namely low temperature process. This process suppresses the warpage of parts during the process by anchoring the parts to a rigid base plate similarly to metal powder bed fusion.[6] One of the advantages of this process is no restrictions on the setting of the powder bed temperature. [7] In our previous report, laser sintering of PEEK and PPS by low temperature process in lower powder bed temperature had been achieved in laboratory machine [8][9]. In addition, laser sintering of PEEK at powder bed temperature of 170°C has been succeeded [10]. This temperature is the same with the process of PA12, which is the most common material in laser sintering process, and available to commercially available low powder bed temperature machine.

However, the low temperature process of PEEK requires greater energy supply than normal laser sintering process to melt materials. When supplied energy increase, the risk of surface thermal decomposition will increase due to excessive temperature elevation by high laser energy intensity. To prevent the risk of thermal decomposition, it is necessary to increase supply energy without increasing energy intensity.

In laser welding and selective laser melting of metal, laser defocusing is using for prevention of high intensity of laser without suppled energy decreasing.[11][12] The distribution of irradiation intensity of gaussian laser depends on square of laser radius shown as below.

$$I = \frac{2P}{\pi {\omega_0}^2} e^{\frac{-(x^2+y^2)}{2}} \tag{1}$$

From this relationship, it is possible that keeping supply energy with prevention of laser intensity by large spot diameter by defocusing. From previous study, efficacy of laser defocusing in suppress thermal decomposition is known, but detail investigation have not been yet.

In this study, the difference of parts that was built when the spot diameter was changed by defocusing in the same supply energy was investigated. The appearance, density, and internal structure of the build parts were used as indicators to evaluate the difference. This result clarifies the effectiveness of defocus in low temperature process and contributes to the improvement of the process.

Methodology

To investigate effect of spot diameter by beam defocusing, low temperature process was performed with same supplied energy and difference spot diameters. The appearance, density and internal structure by cross-section were observed. Simple rough estimation of surface temperature was performed by ANSYS to understand phenomenon in detail of relationship between surface temperature and spot diameter.

Material and Machine

Material employed this research is PEEK powder material, Vestakeep 2000FP (evonik). Material true density is 1300 kg/m^3. The melting point, crystallization temperature and glass transition temperature are 340 ℃, 280℃, andapproximately 150 ℃, respectively. Prior to experiment, powder was sieved to remove oversize particle. The average particle size after sieving measured with laser diffraction particle size analyzer MT3000-II (Microtrac) was 66 μm.

Employed machine was modified laser sintering machine RaFaEl300C (Aspect) which equip CO_2 laser. Laser scanning is controlled by Galvano mirror. The spot diameter on the powder bed surface can be changed by defocusing from 359 μm to 1685 μm.

Process condition

Table 1 shows laser irradiation parameters set. Powder bed temperature was set at 170 ℃ which is usable temperature forprocess of PA12 that is most popular in LS. This temperature is between crystallization temperature and glass transition temperature of PEEK so that thermal stress by solidification is released during process. Layer thickness was set at 0.1 mm, and energy per unit area, which indicated that supply energy was set to 0.20 J/mm^2, 0.25 J/mm^2, 0.30 J/mm^2. Only the spot diameter on the surface was changed in each energy per unit area without change laser speed, scan spacing, laser power. There are no offset of scanning pass for dimension adjustment and only filling laser scanning was done. Build shape of specimen was half scale tensile test specimen.

Table 1. Laser irradiation parameters set

Energy per unit area	Laser power	Scan spacing	Scan speed	Laser diameter on surface
0.20 J/mm^2	50.0W	0.05 mm	5 m/s	629 μm
				808 μm
				1281 μm
				1453 μm
				1685 μm
0.25 kJ/mm^2	62.5 W	0.05 mm		1042 μm
				1281 μm
				1453 μm
				1685 μm
0.30 J/mm^2	75.0 W	0.05 mm		1042 μm
				1281 μm
				1453 μm
				1685 μm

Density measurement

Parts density was measured by the Archimedes method used water as the medium. The measurement was performed at water temperature 23 °C and water repellent spray was sprayed on the surface to prevent water from entering the open cracks. The relative density, which is the measured density divided by the true density of the material, was used to evaluate.

Rough estimation of surface temperature

The qualitative trends in temperature of powder bed surface at various spot diameters were roughly estimated by transient heat transfer analysis using ANSYS. The simulation of 2.00 mm wide laser scan with scan spacing of 0.05 mm was calculated in a 2D cross section. Figure 1 shows schematic diagram of the analysis. The scanning direction of the laser in this figure is perpendicular to the surface of this paper.

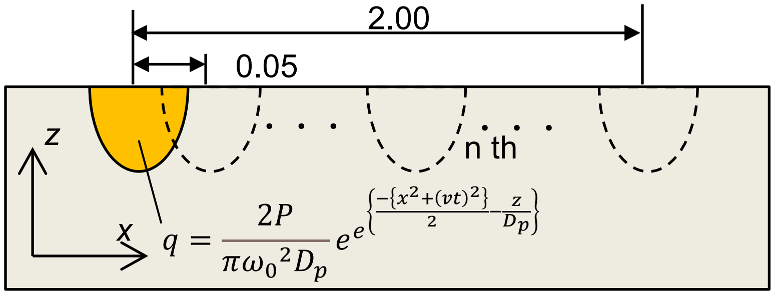


Figure 1. Schematic image of rough estimation of surface temperature.

Heat transfer and heat generation were set as boundary conditions. The heat transfer coefficient was set to 50 W/(m^2 K) assuming the effects of natural convection and radiant heat transfer on the flat plate. The heat generated by the laser irradiation in single scan pass is defined as follows based on Lambert-Beer's law and the intensity distribution of Gaussian laser, with the laser center on the powder bed surface as the origin.

$$q = \frac{2P}{\pi \omega_0{}^2 D_p} e^{\left\{\frac{-\{x^2+(vt)^2\}}{2} - \frac{z}{D_p}\right\}} \tag{2}$$

Here, v and t are a laser scan speed and a time from start of the laser scanning, respectively. D_p indicate a penetration depth defined as the distance that decrease the intensity from initial value to $1/e$. Table 2 and Table 3 show calculation parameters. For simplicity, the penetration depth is set at 100 μm and the latent heat of fusion and change in density from powder to solid due to melting were not considered.

Table 2. Irradiation parameters for calculation

Initial temp.	Laser power	Scan spacing	Scan speed	Laser diameter on powder surface
170 ℃	62.5W	0.05 mm	5 m/s	359 μm, 629 μm, 808 μm, 1453 μm

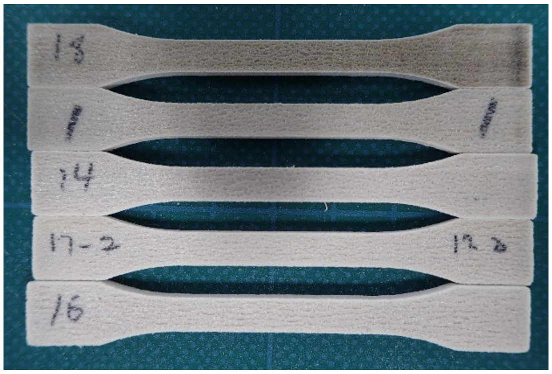
Table 3. Calculation parameters

Thermal conductivity [13]	Specific heat [14]	density	Heat transfer coefficient
0.26 W/(m K)	2.2 J/(g K)	1300 kg/m^3	50 W/(m^2 K)

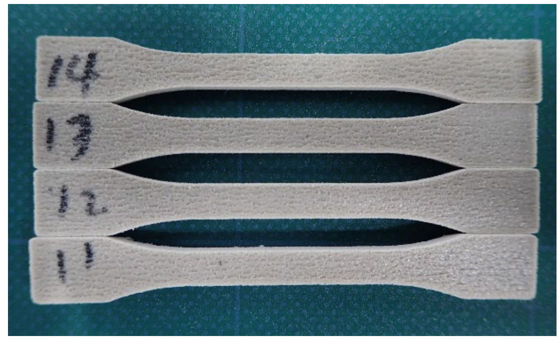
Experimental result

Figure 2 shows specimens of each energy per unit area. Surface discoloration was observed in the specimens irradiated with the small spot diameter with energy per unit area of 0.20 J/mm^2 and 0.30 J/mm^2. In addition, roughness was observed near the edges of the specimens irradiated with small laser diameter (1042 μm) with energy per unit area of 0.30 J/mm^2.

(A) 0.20 J/mm^2



(B) 0.25 J/mm^2



(C) 0.30 J/mm^2

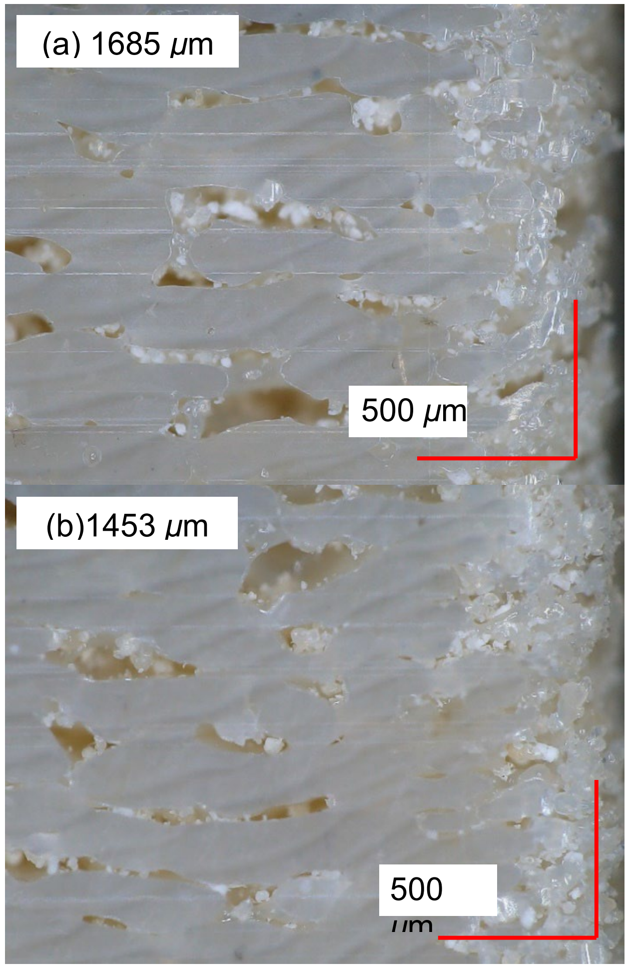
Figure 2. Appearance of the specimens in each energy per unit area with difference spot diameter. (A) shows specimens 0.20 J/mm^2 and Spot diameters in process are 629 μm, 808 μm, 1281 μm and 1453 μm in order from the top. (B) shows specimens in 0.25 J/mm^2 and spot diameters in process are 1042 μm, 1281 μm, 1453 μm and 1685 μm in order from the top. (C) shows specimen in 0.30 J/mm^2 and spot diameters are same with (B).

Figure 3 shows cross-section of the specimens built at energy per unit area 0.25 J/mm^2. Building direction is from bottom to up on paper. Brown spots which indicate occurrence of thermal decomposition was not observed in any specimens and the rate of voids were the least at spot diameter of 1281 μm. The void shape of specimens with large spot diameter was perpendicular to the building direction, while the void shape of the specimens with small spot diameter was oriented diagonally to the building direction.

Figure 4 shows the relationship between the spot diameter and the specimen width. The specimen width increases as the spot diameter and energy per unit area increase, and the relationship can be linearly approximated.

Figure 5 shows the relationship between the relative density and the spot diameter. As the energy per unit area increases, it shifts to the high-density side. The density also changed with the change of the spot diameter, and the density peaked at the spot diameter of 1281 μm at the energies of 0.25 J/mm^2 and 0.30 J/mm^2 per unit area.

Figure 6 shows the relationship between the peak intensity of the laser and the relative density. The specimen density showed peaks or bending point between intensity of laser 20 W/mm^2 and 30 W/mm^2 at all energies per unit area.


(a) 1685 μm
500 μm
(b)1453 μm
500
μm

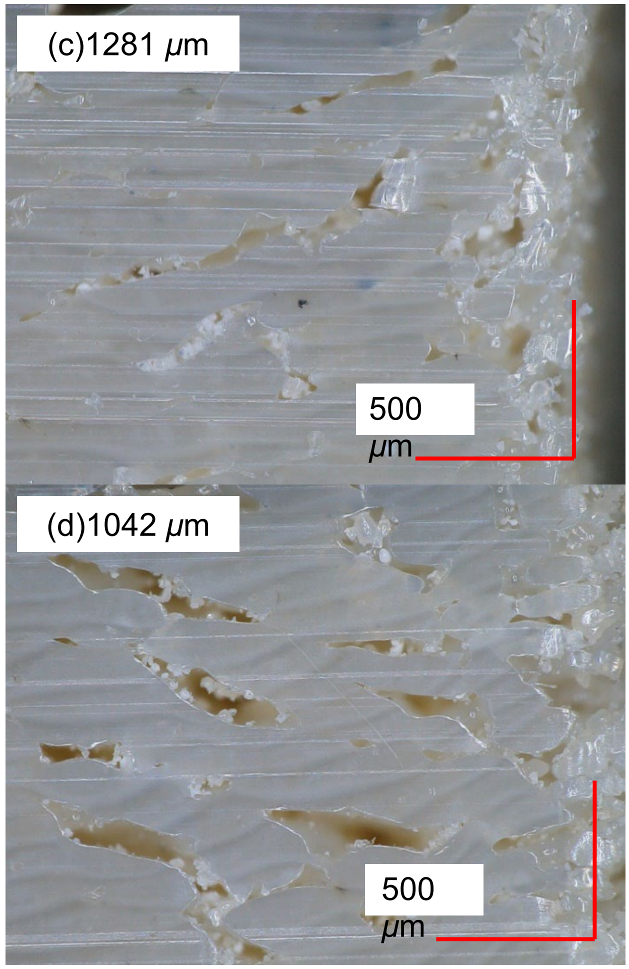


Figure 3. Cross section of the specimens in energy per unit area at 0.25 J/mm^2 in near of side. Laser diameters during process are 1685 μm (a), 1453 μm (b), 1281 μm (c), 1042 μm (d), respectively.

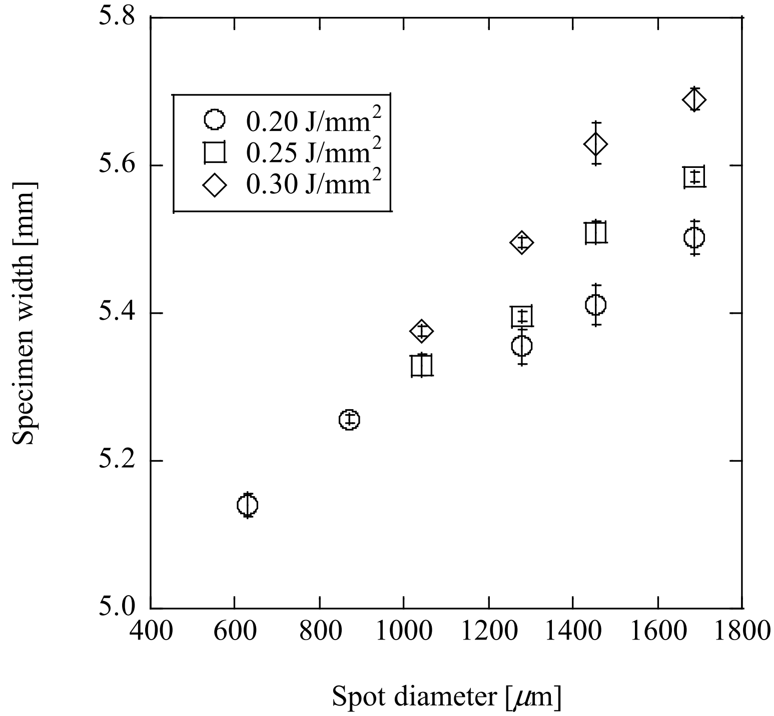


Figure 4. Relationship between specimen width and laser diameter. Nominal width of specimen is 5.00 mm.

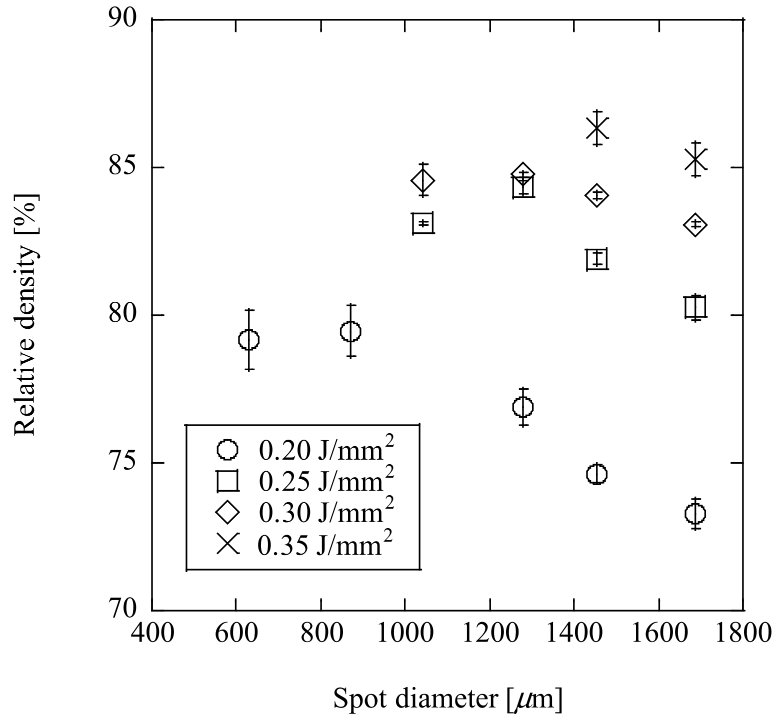


Figure 5. Relationship between laser diameter and relative density.

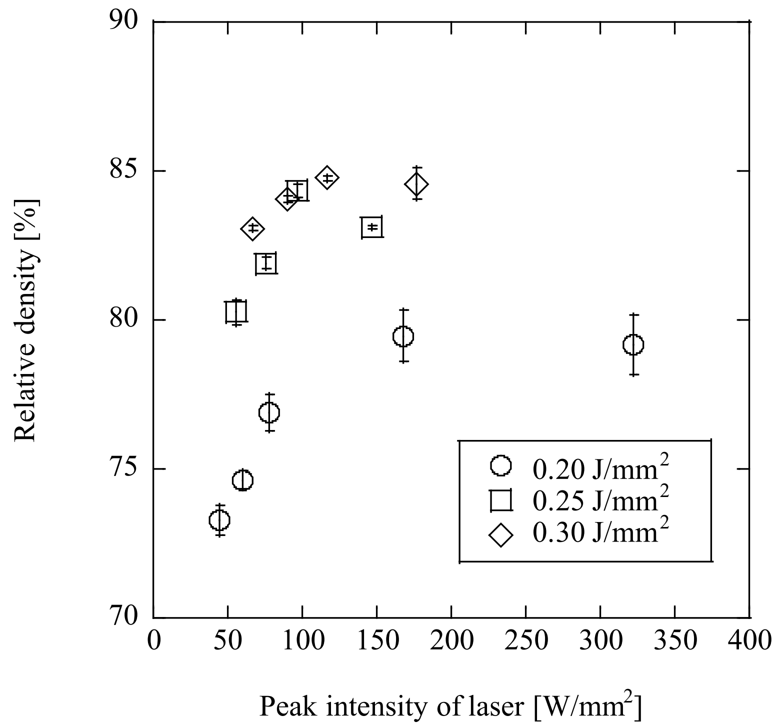


Figure 6. Relationship between peak intensity of laser and relative density.

Figure 7 shows the time variation of the rough estimation of surface temperature at each spot diameter obtained by analysis. When the spot diameter is large, the temperature rise starts from an early stage. In addition, as the spot diameter increased, the temperature rise showed a small behavior, and the maximum temperature reached tended to decrease.

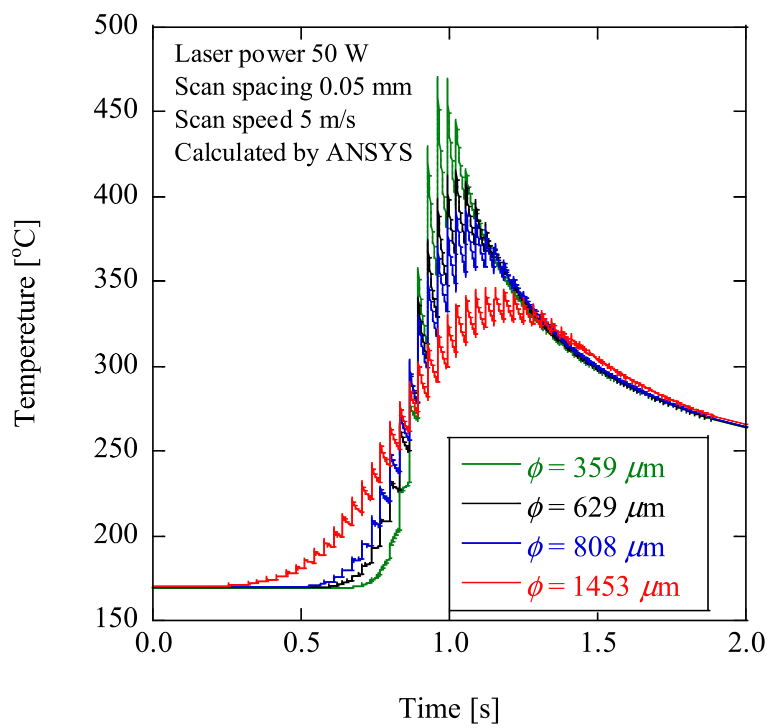


Figure 7. Temperature variation on surface by rough estimation.

Figure 8 shows the relationship between the maximum temperature shown in Figure 7 and the spot diameter. The temperature decreased with spot diameter increase and the behavior of this relationship was not linear, but a downwardly convex curve was drawn. The solid line indicated approximate curve by $a/x^2 + b$. The approximate curve and plot points did not match.

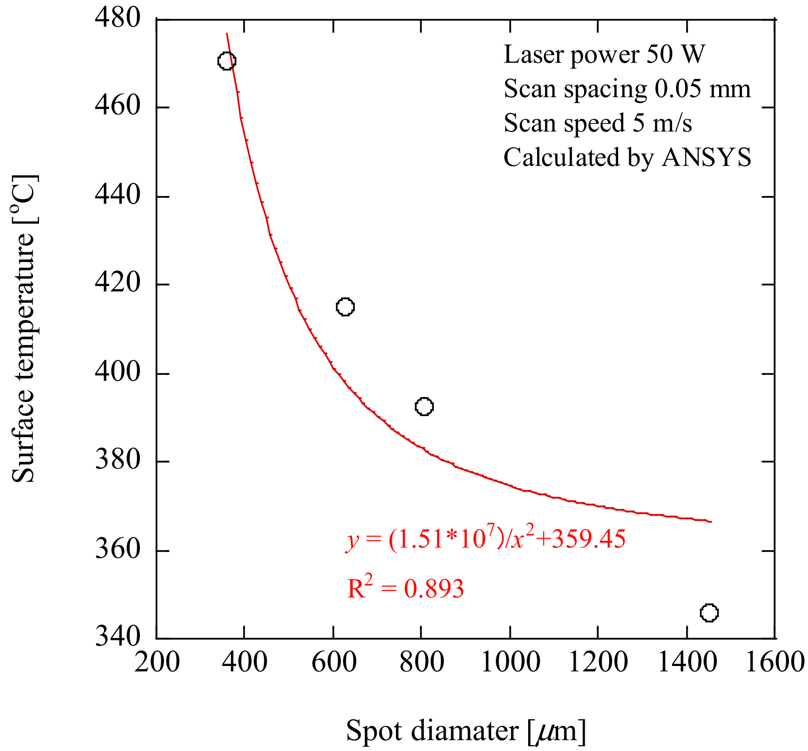


Figure 8. Relationship between rough estimation of surface temperature and laser diameter.

Discussion

The results shown in Figures 2-6 show that not only the amount of energy delivered, but also the peak value of the laser energy is an important factor in the melting and filling of the material in laser sintering. The parameters that affect the peak energy value are the laser power and radius, as shown in equation (1). The results in Figure 6 suggest that an optimal intensity value exists between 75 and 125 W/mm^2. This energy intensity range is one of the parameters for irradiating the laser without causing thermal decomposition in PEEK. It also shows that by controlling the spot diameter so that the laser intensity does not exceed this range, large amount of energy can be supplied without causing thermal decomposition. However, the scanning speed is deeply related to the heat generation during the laser irradiation, so more detailed examination is required including this factor.

From the cross-section shown in Figure 3, elongated voids were observed in all specimens, but the spot diameter of the irradiated laser changed the direction of the voids. The voids of the specimen with spot diameter of 1453 μm and 1685 μm spread laterally, and unmelted powder was confirmed in the voids. Since the energy intensity is small at large laser diameter, it is assumed that the temperature was not sufficient for the melting and filling, and only the part near of the surface was melted and solidified. On the other hand, at 1042 μm, which had the smallest spot diameter in this paper, the voids were slanted, and almost no unmelted powder was found in them. It is assumed that the smaller the spot diameter, the higher the laser intensity, and the higher the temperature of the melted material in the laser irradiated area, causing excessive melting.

The repeated temperature rises and falls in Figure 7 indicate heat dissipation from the surface. Assuming that heat dissipation is independent of laser diameter, the maximum temperature shown in Figure 8 depends on the maximum intensity of the supplied energy and should be inversely proportional to the square of the spot diameter, as in equation (2). However, as is clear from the approximation curve, it is difficult to say that this relationship holds true, and it indicates that the heat dissipation differs depending on the spot diameter. This phenomenon cannot be ignored when controlling the spot diameter to optimize the process.

Conclusion

In this study, the effect of the difference in the spot diameter due to defocus was investigated by measuring the parts density and rough estimation analysis in low temperature laser sintering process of PEEK. As a result, it was indicated that increasing the spot diameter is an effective way to liaises both suppression of thermal decomposition and large amount of energy supply. From the relationship between the density of parts and the laser intensity, it was indicated that consideration of not only amount of the energy supply but also the energy intensity is necessary to desired process. Especially in low temperature process, it was suggested that irradiation with high energy intensity causes excessive flow of melted material and causes increase of the voids. A rough estimation of surface temperature found that increasing the diameter of the laser slowed the heat supply and that the diameter of the laser affected the heat dissipation.

These knowledges can contribute to the improvement of the laser sintering process.

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