

Low temperature selective laser melting of high temperature plastic powder

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ABSTRACT

In a typical plastic laser sintering or melting system, powder bed temperature is maintained above the recrystallization temperature of the powder material to prevent the parts under process from warping until the whole layers are processed. Although this countermeasure can elegantly suppress the part warpage, heating the powder bed to such a high temperature causes many problems. In case of high temperature plastic such as polyetheretherketone (PEEK), bed temperature should be more than 300°C. Due to this requirement, machine cost is extremely high and powder recyclability is very low. The authors had introduced another countermeasure for the part warpage that anchors the in-process parts to a rigid base plate instead of heating the powder bed above the recrystallization temperature. In the current research, application of this method to PEEK powder is tested, and a simple test piece of which relative density is more than 90% was successfully obtained with preheating temperature of 200°C. In this paper, mechanical performances of obtained parts are presented, and several problems with the process of PEEK powder are discussed as well.

INTRODUCTION

Plastic laser sintering (LS) is one of the most promising additive manufacturing processes that will be involved in direct parts manufacturing. In reality, several high value parts or products in aerospace and medical applications are produced by utilizing this technology already. Although these applications often require high performance plastics, so-called “super engineering plastics,” process of such material is not commercialized or much more expensive than those for standard materials such as PA11 and PA12. This limitation in material choice hinders the technology from expanding its application range. In the most layer manufacturing processes that obtain each layer by selective solidification of liquid or powder, one of the primary problems is part warpage caused by layer-by-layer shrinkage as the layers are consolidated and accumulated. In a typical plastic LS system, its powder bed is maintained above the recrystallization

temperature of the powder until the whole layers are solidified to minimize the layer-by-layer shrinks [1]. Although this anti-warp countermeasure can suppress the warpage elegantly, heating the powder bed to such a high temperature causes various problems. A normal plastic does not melt and recrystallize at points of temperature but in ranges with some widths, and the ranges are often overlapping. This overlapping leads to hard “cake,” a region of a powder bed that have not been solidified intentionally, and results in low powder recyclability. Additionally, the preheating requires LS machine with a heat resistance, and the requirement is quite high when high-temperature plastic is processed. Previously, the authors had introduced another anti-warp countermeasure that suppresses the warping by fixing the part to a rigid base plate instead of heating the powder bed to such a high temperature [2]. In the following discussion, we call this process as “low temperature process,” and normal process in which powder bed is kept above recrystallization temperature as “high temperature process.” In the current research, low temperature process of poly-etheretherketone (PEEK) super engineering plastic is preliminary tested. PEEK powder is processed with powder bed temperature of 200°C while it is more than 300°C in normal high temperature processed [3, 4]. Various parameters for consolidation of the powder is searched for, and relative density (part density) of the obtained objects are measured as primary index of their strength [5, 6]. Tensile, bending and impact tests are carried out. Mechanical performance in high temperature condition is tested as well.

MATERIAL AND METHOD

Material

PEEK powder (VESTAKEEP200FP produced by EVONIK) was employed. The powder was sieved to cut larger grains. The mean particle size, which is obtained by manual measurement from optical micrograph (Fig. 1) became 50 μ m, resultantly. Thermal and mechanical performance of the material is as summarized in Tbl. 1.

Tbl. 1 Parameters of employed material

Type of material	PEEK(Polyetheretherketone)
True density	1.3 g/cm ³
Bulk density	0.28 g/cm ³
Average particle size	50 μm
Melting point	340°C
Tensile strength	100 MPa
Tensile modulus	3700 MPa

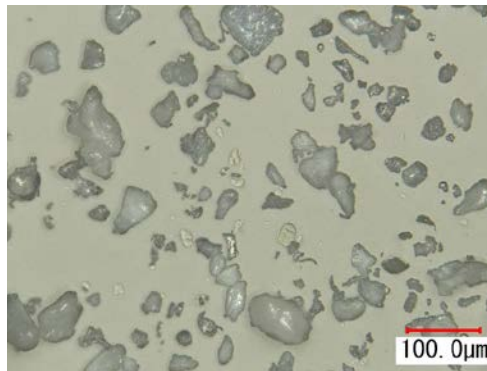


Fig. 1 Optical micrograph of powder grains

Laser sintering apparatus

An experimental set up used in this research had been developed by the authors. The machine consists of building system, laser unit and their controller (Fig. 2, 3). The laser unit is installed with a CO₂ laser that can generate an infrared with a wavelength of 1064μm. The maximum power at the source is 30W. Irradiated beam is focused by a dynamic focusing system equipped with Z-galvanometer into a very small spot of 130μm in diameter while it is 400 to 500μm in typical commercial systems. The spot diameter is modified or defocused by adjusting the distance between laser unit and the bed as illustrated in Fig. 4. A pair of galvanometers scans the beam in x and y direction in a range of approximately 100×100mm. The beam shoots the powder bed through a laser window of ZnSe, which seals the build chamber. The chamber is purged with nitrogen gas to inhibit the bed from oxidization. The maximum power at the bed is attenuated to 15W by transmission and reflection loss of the optics. Powder coating system employs a coating roller of which rotational and transverse speeds can be controlled independently. Two heater systems can heat surfaces of feed stock and part bed up to 200°C. In this research, the highest powder bed temperature of 200°C For the base plate a 12mm thick PEEK plate was used. Parameters of the LS apparatus is summarized in Tbl. 2

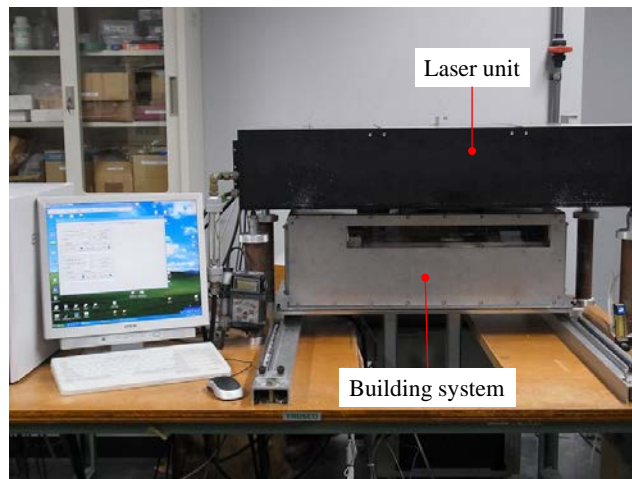


Fig. 2 Laser sintering system

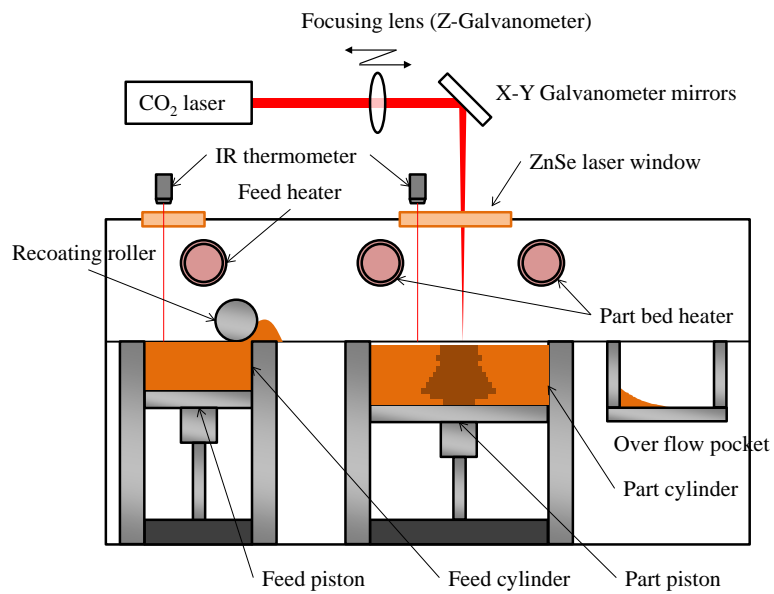


Fig. 3 Schematic view of the laser sintering system

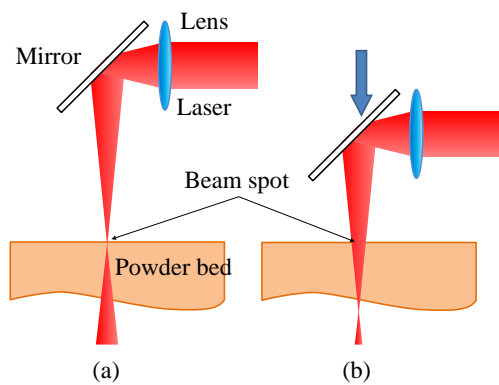


Fig. 4 Beam spot adjustment. (a) the minimum spot, (b) greater (defocused) spot

Tbl. 2 Parameters of the LS apparatus

Maximum beam power	12 W
Minimum Spot diameter	130 μm
Maximum scanning speed	3.81 m/s
Minimum scan interval	21 μm
Maximum powder bed temperature	200°C
Work volume	100 mm \times 100 mm \times 100 mm

Observation and measurement

Optical transmission observation was carried out using a digital microscope (VHX-2000 and VH-Z100, KEYENCE.) Specimens with thickness of 20 μm to 50 μm were prepared with large scale microtome (ERMA, INC.) Tensile test (ISO527-2) and bending test (ISO178) were performed by using multipurpose test machine (Instron 3365.) For impact test DG-UB from Toyoseiki was used. For tensile tests, a small test piece (JIS-K7161-2) was used due to part size limitation of the LS system employed in this research. For high temperature test, AG-100kNX (SHIMAZU) was used.

EXPERIMENTAL RESULTS

Minimum spot diameter and typical layer thickness

A parameter set as shown Tbl. 3 was tested. The beam was focused as small as possible, i.e. 130 μm . For the layer thickness, a typical value in commercial LS system of 100 μm was used. The powder bed is preheated to the highest temperature of 200°C. As soon as laser scanning was started, sparking and smoking was observed as shown in Fig. 5, although the chamber is well purged with nitrogen gas. Surface of the obtained parts are burnt as shown in Fig. 6. Although such sparking occurred, powder bed was rarely melted as shown in Fig. 7, and obtained part density was only 68%.

Tbl. 3 Build parameter for the minimum spot diameter and 100 μm layer thickness

Laser power	Scanning speed	Scanning interval	Energy per unit area	Beam diameter	Layer thickness
6.6W	1.53 m/s	27 μm	159 J/m ²	130 μm	100 μm

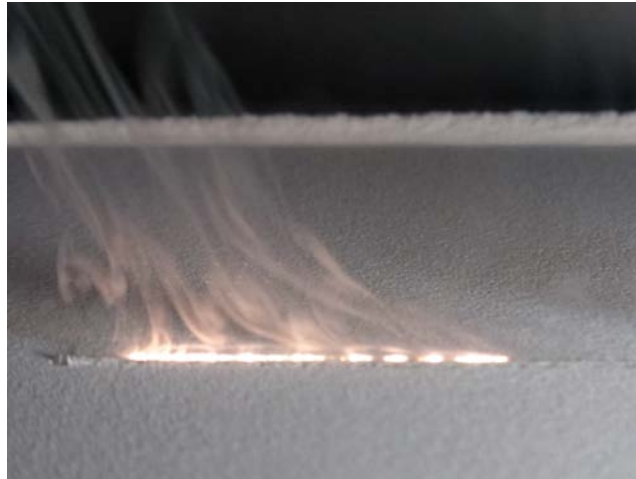


Fig. 5 Spark and smoke

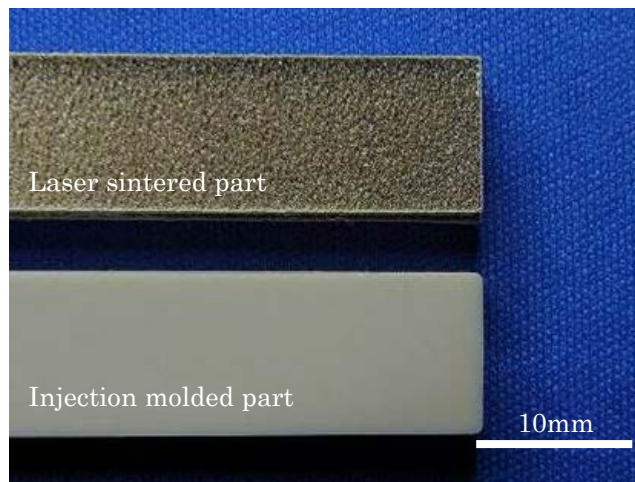


Fig. 6 Burnt surface of the obtained part. (top) A part from injection molding is also displayed for comparison. (bottom)

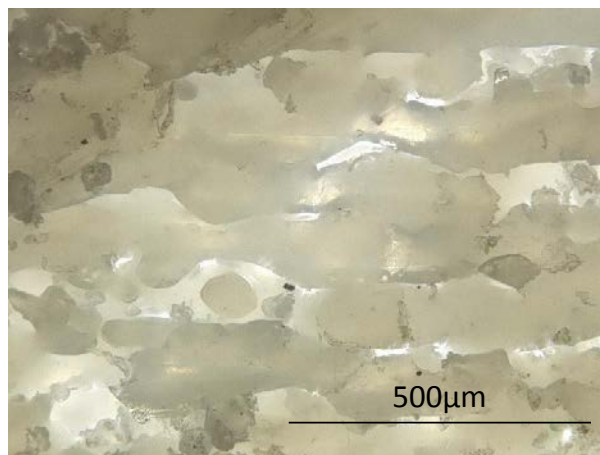


Fig. 7 Cross-sectional view of the part obtained from the parameters in Tbl. 3

Effect of Defocusing

To supply light energy more mildly, a larger laser spot was tested. The laser unit was lowered so that the beam shoots the powder bed before being focused completely as illustrated in Fig. 4. The beam diameter was expanded to 560 μm , which is typical focal diameter of commercial machine. Energy supply per unit area (energy density) is adjusted to be similar to the previous case. Tbl. 4 summarizes the parameter.

Sparking and smoking was suppressed as shown in Fig. 8, and burning was also eliminated as shown in Fig. 9. Part density is still very low as 71%.

Tbl. 4 Build parameter for expanded beam spot

Laser power	Scanning speed	Scanning interval	Energy per unit area	Beam diameter	Layer thickness
10.0W	1.53m/s	40 μm	165kJ/m ²	560 μm	100 μm

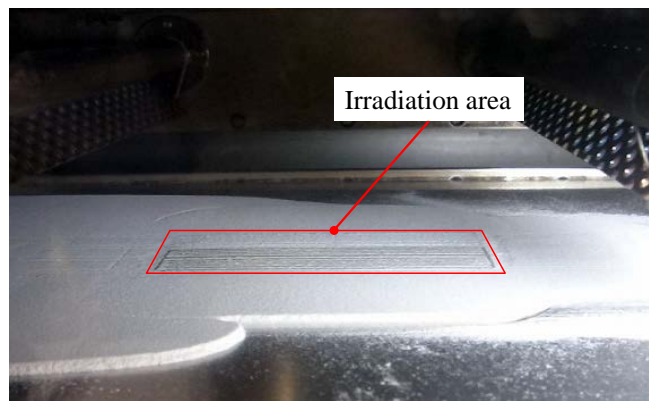


Fig. 8 Laser irradiation with expanded beam spot at the same energy density

Sparking and smoking can also be suppressed by simply reducing laser power or energy density without defocusing. Tbl. 5 summarizes the parameter set that can suppress the sparking and smoking without defocusing. Though reducing energy density could suppress the sparking or smoking, doing this decreased the part density to 60.4%, resultantly.

Tbl. 5 Build parameter for fully focused spot which does not cause sparking or smoking

Laser power	Scanning speed	Scanning interval	Energy per unit area	Beam diameter	Layer thickness
7.65W	1.53m/s	40 μm	125kJ/m ²	130 μm	100 μm

Effect of reducing layer thickness

Smaller layer thicknesses as summarized in Tbl.6 were also tested. Layer thickness of

50, 40 and 30 μ m, was used while a typical system uses 100 μ m, and part densities of 73.0, 76.5 and 81.0% were obtained, respectively. By reducing layer thickness, part density was improved by a factor of 10% to 20%.

Tbl. 6 Build parameter for narrow layer thickness

Laser power	Scanning speed	Scanning interval	Energy per unit area	Beam diameter	Layer thickness
7.65W	1.53m/s	40 μ m	125kJ/m ²	130 μ m	50, 40, 30 μ m

Mechanical tests

The above-mentioned results indicate that defocusing beam can reduce the sparking and smoking problem, and that part density can improve when the layer thickness is reduced. On the basis of these guidelines, a build parameter set for the following mechanical tests was searched in the range as shown in Tbl. 7. Part densities out of the various parameter were measured, and resultant relationship between the part density and energy density is shown in Fig. 9. Although an energy density does not represent a unique parameter set since different parameter can make the same energy density, energy density is still the most dominating parameter for the part density. The highest part density of 96% was obtained from the parameter shown in Tbl. 8. Fig. 10 is a transmission optical micrograph of the cross-section of the specimen. Contrarily to the previous case which is shown in Fig. 7, the cross-section was quite smooth except that some voids and dusts indicated by dark stains. There is no vestige of grains that has not been melted. Tbl. 9 summarizes mechanical performance of the part. Those for PA powder (ASPEX PA, ASPECT) and typical performance of molded parts are also displayed as a reference. Tensile and flexural strengths are roughly 80% to 90% of those for injected parts. This difference is almost the same or smaller than that for laser sintered PA. Impact performance was much worse than molded parts. In comparison with LS parts from PA, PEEK parts was much stronger as expected. Fig. 11 shows the result of flexural tests at high temperature. PEEK parts maintain a good mechanical performance in high temperature condition, while PA loses its performance at 100°C.

Tbl. 7 Parameter sets for density measurement for mechanical tests

Laser power	Scanning speed	Scanning interval	Energy per unit area	Beam diameter	Layer thickness
11.5-12.6W	0.93, 1.53m/s	21-40 μ m	194-361kJ/m ²	560 μ m	50 μ m

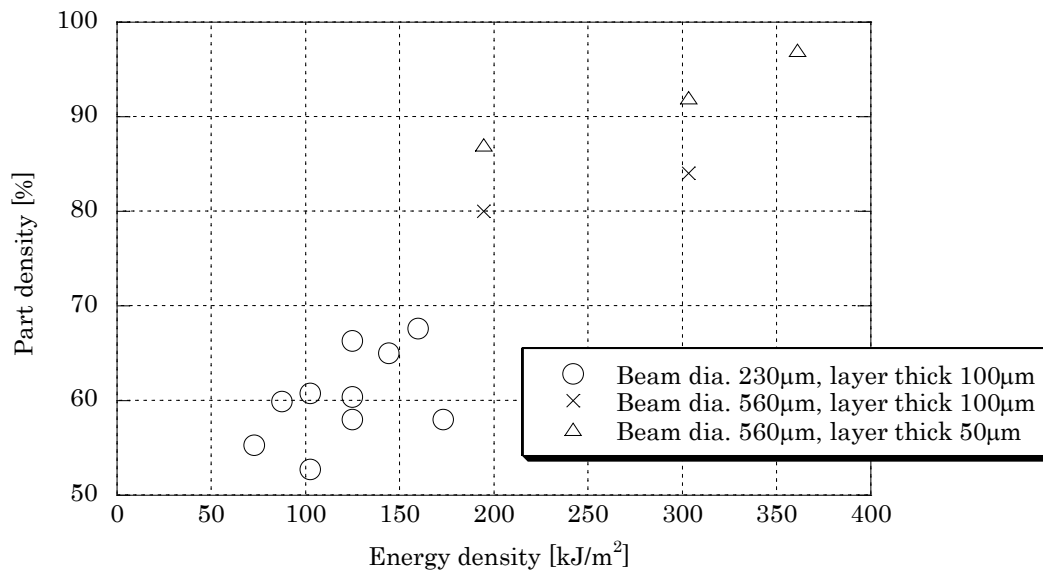


Fig. 9 Relationship between part density and energy density

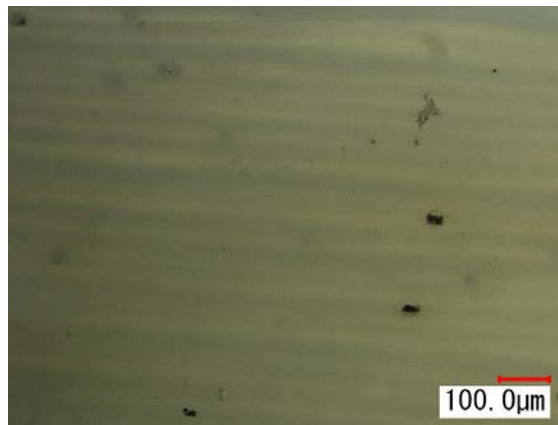


Fig. 10 Cross section of a part with 96% of part density

Tbl. 7 Mechanical performance summary for PEEK and PA

	Process	Stress at yield [MPa]	Flexural stress [MPa]	Notched Impact strength [kJ/m²]	Impact strength [kJ/m²]
PEEK	Laser sintering	79 ± 3	143 ± 6	1.36 ± 0.02	9.1 ± 0.6
	Injection molding	100	150	-	No break
PA	Laser sintering	47	58.5	2.36	-
	Injection molding	70	NR	9	No break

NR=Not reported

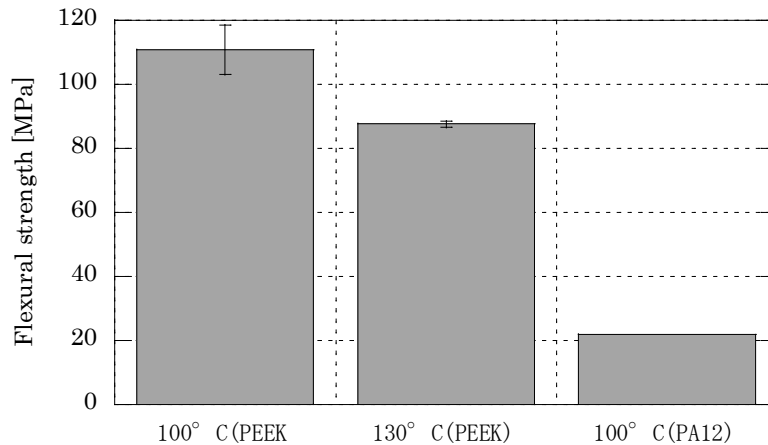


Fig. 11 Flexural strength at high temperature process

DISCUSSION

Sparkling and smoking is a new problem that was not observed in low temperature process of PA powder. Since the problem is suppressed by reducing the light intensity even if total energy supply is larger, cause of the phenomenon is not as a result of long term exposure to the heat or high temperature but direct reaction between light and resin or decomposition by instantaneous heat at very high temperature. The reason why thinner layer thickness improved the part density is that penetration of the infrared beam is shorter than typical layer thickness of 100 μ m. In other words, the light-heat exchange occurs only near the bed surface.

In typical plastic laser sintering process, which uses high temperature process, each powder grain is melted partially [7]. Disappearance of the vestige of grains in the obtained parts shows that the powder is completely melted. Thus we should call this process “selective laser melting” rather than “laser sintering.” In case of low temperature process of PA, each grain melts completely as well, or the density becomes very low. In low temperature process, it is assumed, the period in which the material stays melted and fluidic is much shorter than in high temperature process since surrounding powder bed is colder. As a result the viscosity of the molten material should be lower to obtain high part density. It is surmised that the low temperature process requires the whole powder in a layer to be melted totally, and this increases the significance of light penetration through the powder layer.

Obtained mechanical performance is quite satisfying. However adjustment of the parameter was quite difficult. Coating such thin layer of the powder requires improvement in powder flowability and coating mechanism. Build parameters depends

very much on various conditions such as parts shapes, their arrangement in the bed and scan pattern. More investigation is still required for practical use of this technology. Powder bed temperature of 200°C is easy to achieve from the view point of machine development, and this means that the low temperature process can facilitate the laser “melting” of high performance plastic in the near future.

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CONCLUSIONS

PEEK powder was successfully processed with low powder bed temperature of 200°C. Heat resistance against the temperature is achieved relatively easily, and the result indicates that PEEK can be processed practically in the near future. Sparking and smoking phenomena were observed, but the problem can be avoided by reducing light intensity of the laser. Part density reached a high value of 96%, which is a standard value of commercialized process for PA powder. Reduction of mechanical performances from those of typical injection molded parts is around 10 to 20%. This reduction is almost the same as the case of PA powder. Additionally, good performance is obtained in a high temperature condition as well. Various conditions such as shape of objects, their arrangement and scan pattern affect the performance of the parts. These problems are remaining to be solved in the future.

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