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Reviewed Paper

Relationship between powder bed temperature and microstructure of laser sintered PA12 parts

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Abstract

The microstructure of a part from semi-crystalline polymers used in laser sintering gives a significant impact on the mechanical properties of the parts. The microstructure of laser sintering parts depends on powder bed temperature. If the powder bed temperature can be set in a wide range, it is also possible to control the microstructure of a part.
The authors have been introducing a modified laser sintering process, namely low temperature process. The process allows powder bed temperature being set wide range.
In this research, relationship between microstructure of PA12 parts by low-temperature process and powder bed temperature is investigated. As a result, high strength and crystallinity were obtained in high powder bed temperature, and high ductility and low crystallinity were obtained in low powder bed temperature.
This result indicates that parts having the desired mechanical properties can be obtained by controlling the powder bed temperature.

Introduction

Mechanical properties of the laser sintered semi-crystalline polymer parts are strongly dependent on their microstructures. As degree of crystallinity, a major index for the microstructure, increases, strength and stiffness increase, and ductility decreases [1]. Due to crystal formation and growth depend on time and temperature [2-3], crystallinity depends on cooling rate of solidification. In general, crystallinity increases as the cooling rate of solidification decreases. In a laser sintering process, powder bed temperature strongly affects in cooling rate and microstructure. Rietzel et al. investigated influence of temperature on crystallization and reported that cooling rate of laser sintering was slow [3]. The reason is that the powder bed temperature is maintained between melting point and crystallization temperature in order to suppress warpage during the process. If the powder bed temperature can be set in out of this range, it is possible to change cooling rate and control microstructure.

The authors have been introducing a modified laser sintering process, namely low temperature process. This process suppresses warpage during process by anchoring parts to a rigid base plate and allows processing in a wide range powder bed temperature, such as below crystallization temperature [4] and glass transition temperature [5] which cannot be set in normal laser sintering process.

In this research, relationship between microstructure of the laser sintered parts and powder bed temperature was investigated. Crystallinity and mechanical properties were measured and the effect of powder bed temperature for laser sintered parts was discussed.

Material and Method

Material and Machine

Vestosint® X1556 (Dicel-evonic) was used. Its melting and recrystallization temperatures are

184°C and 144°C, respectively. To avoid the effect of thermal history of powder, brand new virgin powder was used for all the experiment.

A commercially available laser sintering machine, RaFaEl300C® (Aspect Inc.) was employed. This machine is equipped with CO_2 laser. Laser spot diameter at powder bed surface is 350μm.

Experimental method

Table1 shows the process parameter set used in this study. To compare regular high temperature process and low temperature process, which the authors are developing, two powder bed temperatures of 170°C and 130°C were used. The higher one is between the melting and recrystallization points and the other is outside of the range. A commercial parameter set was used in high temperature process, and a parameter set which gave the highest density parts was used in low temperature process.

Table 1. Processing parameter set

Powder bed temperature	Scan interval	Laser power	Scan speed	Process classification
130°C	0.10mm	12.0W	2.0m/s	Low temperature process
170°C	0.14mm	24.5W	10.0m/s	High temperature process

Thermal analysis

Differential scanning calorimetry (DSC) was used to investigate the microstructure. The device used for measurement is DSC-60 (Shimadzu Corp.), and the software used for analysis is TA60. Under a nitrogen atmosphere, samples were heated from room temperature to 260°C at a heating rate of 10°C per minute. After holding the temperature at 260°C for 5 minutes, samples were cooled to room temperature at a cooling rate of 10°C per minute. Fig1 shows the DSC curve of Vestosint® X1556. The endothermic peak shows melting, and the exothermic peak shows crystallization.

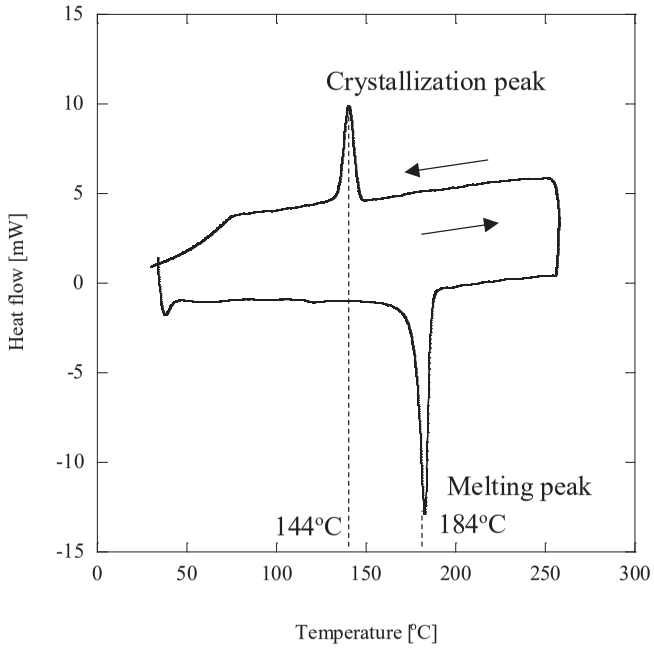

Fig1. DCS curve of Vestosint® X1556 fresh powder

The crystallinity of a semi-crystalline polymer is obtained by dividing the heat of fusion of

the specimen by the heat of fusion of the complete crystal [6]. As the heat of fusion of the complete PA12 crystals, 209.3J/g, which is reported by Gogolewski et al. was used for the calculation [7].

Tensile test

For the tensile test, Instron® 3365 mechanical testing system (Instron) was employed. Tensile test specimens were produced in the shape as shown in Fig2. The crosshead speed was set at 1mm per minute from 0% to 0.25% strain, which is the measurement range of the elastic modulus, and then 5mm per minute until fracture. The elastic modulus was measured by an extensometer attached to the specimen, and the elongation at break was calculated from the crosshead displacement at fracture of the specimen. Tensile strength was measured by a load cell attached to the measurement machine.

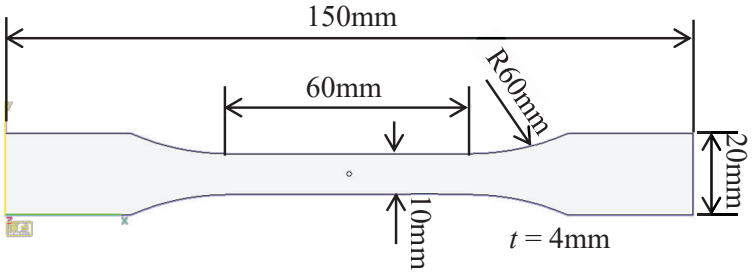

Fig2. Shape and dimension of tensile test specimen

Experimental result

Microstructure and crystallinity

Fig3 shows DSC measurement results for parts for low temperature process and high temperature process. Diagram for high temperature process had a large peak around 178ºC and a small peak around 185ºC. Diagram for low temperature process had a large peak around 175ºC and a small peak around 170ºC. The peak height for high temperature process was greater than the other.

Fig4 shows the obtained crystallinity. The crystallinities of parts were 31% and 35% for the low temperature process and high temperature process, respectively.

Fig5 shows polarized image of cross sections. In the cross section for low temperature process, stripes caused by differences microstructure was observed. On the other hand, in the cross section for high temperature process, there were region including insufficiently melted powder between the layers.

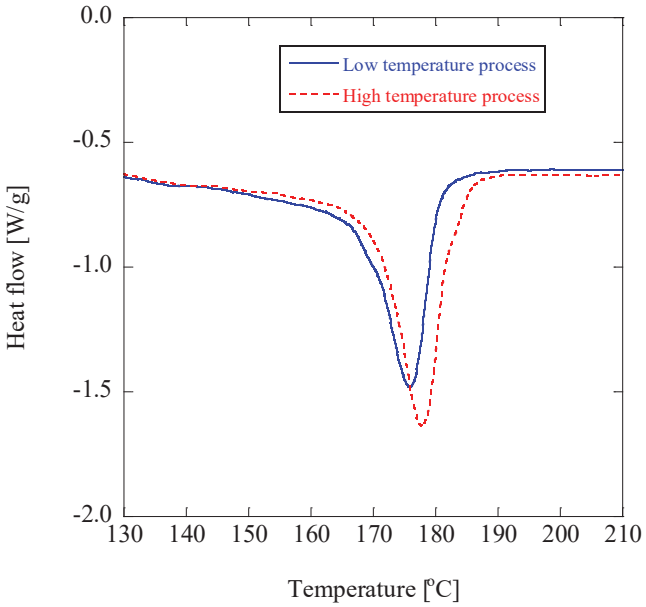

Fig3. DCS curve of parts by low temperature process (solid line) and high temperature process (dashed line)

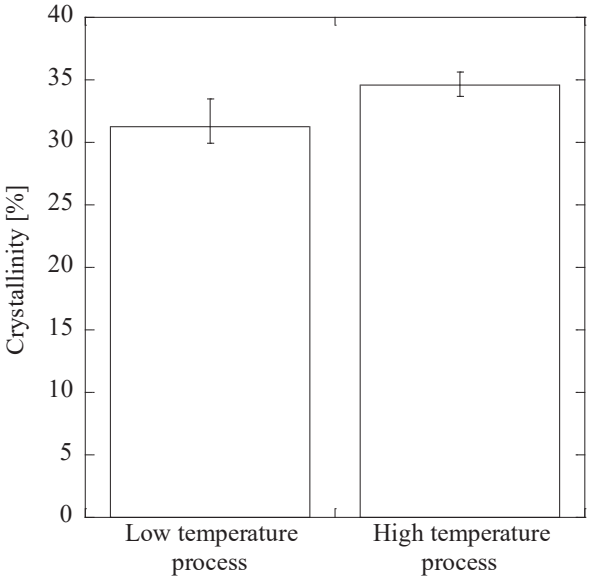

Fig4. Comparison of crystallinity of parts by low temperature process and high temperature process.

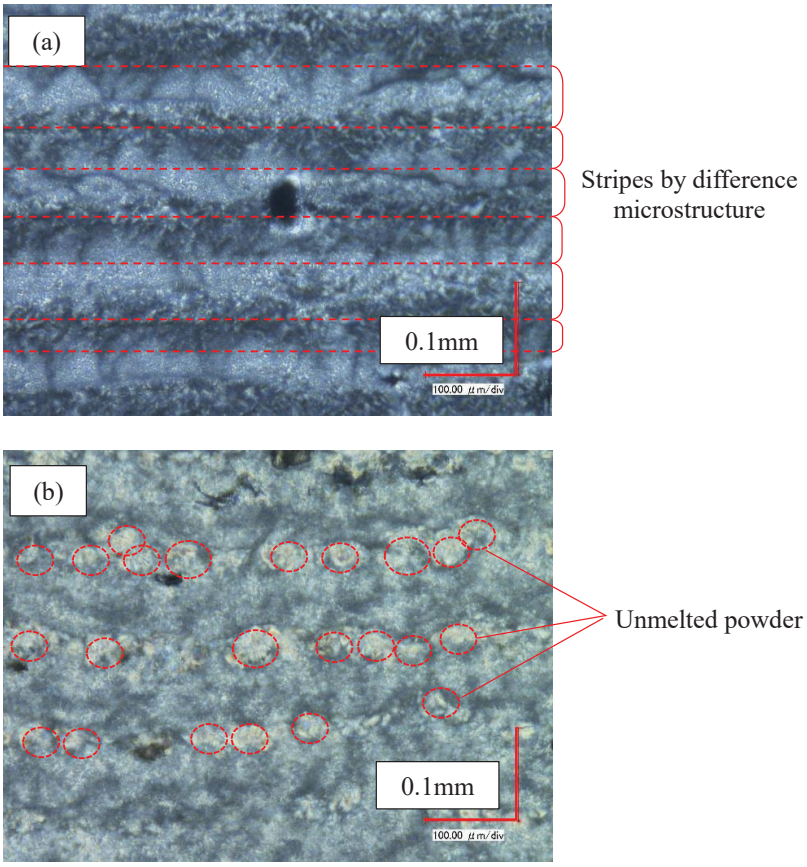

Fig5. Polarized cross section image of parts. (a) is low temperature process and (b) is high temperature process, respectively.

Material properties

Fig6 shows typical stress-strain curves for specimens obtained by the two powder bed temperatures. The stress-strain curve of the specimen for low temperature process showed yield behavior and large elongation. On the other hand, that for high temperature process did not show yield behavior.

Fig7 shows specimens after tensile test. Specimens for low temperature process necked and showed ductile fracture, specimens for high temperature process did not neck and showed brittle fracture.

Fig8 shows the tensile strength (α), elastic modulus (β), and elongation at break (γ) obtained from the tensile test. The tensile strength and elastic modulus were higher at parts for high temperature process, and the elongation at break was larger at parts for low temperature process.

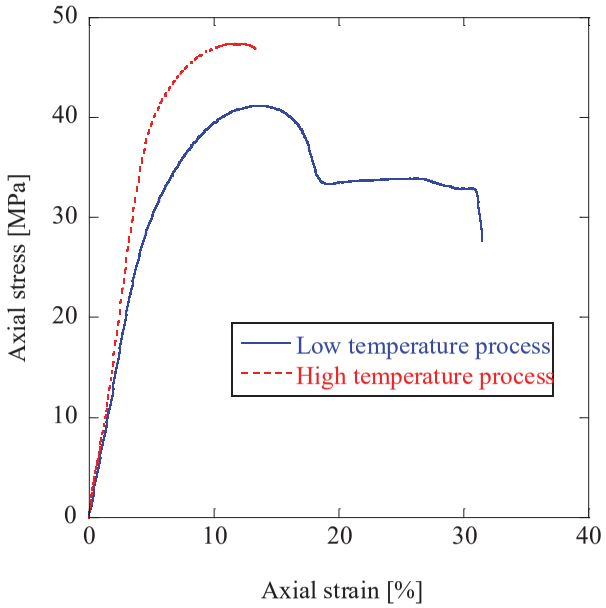

Fig6. Stress strain curve of parts for low temperature process (solid line) and parts by high temperature process (dashed line) obtained by tensile test.

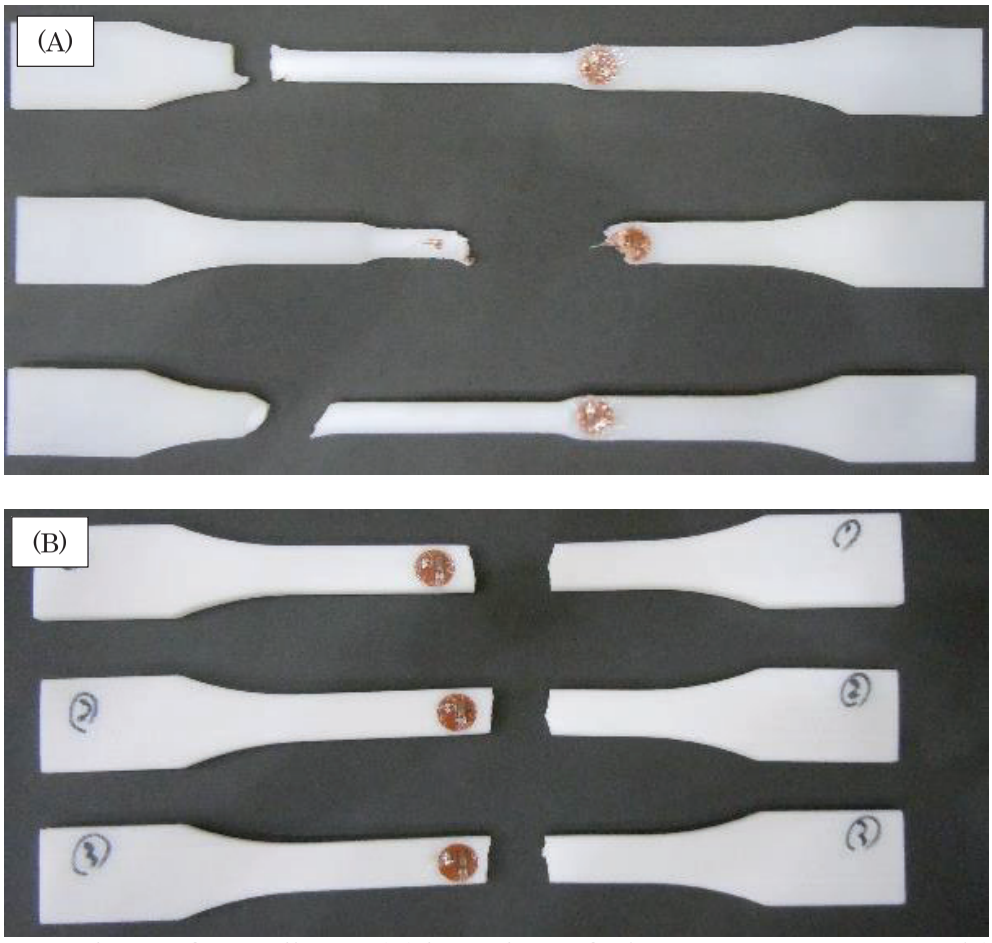

Fig7. Specimens after tensile test. (A) is specimens for low temperature process and (B) is specimens for high temperature process, respectively.

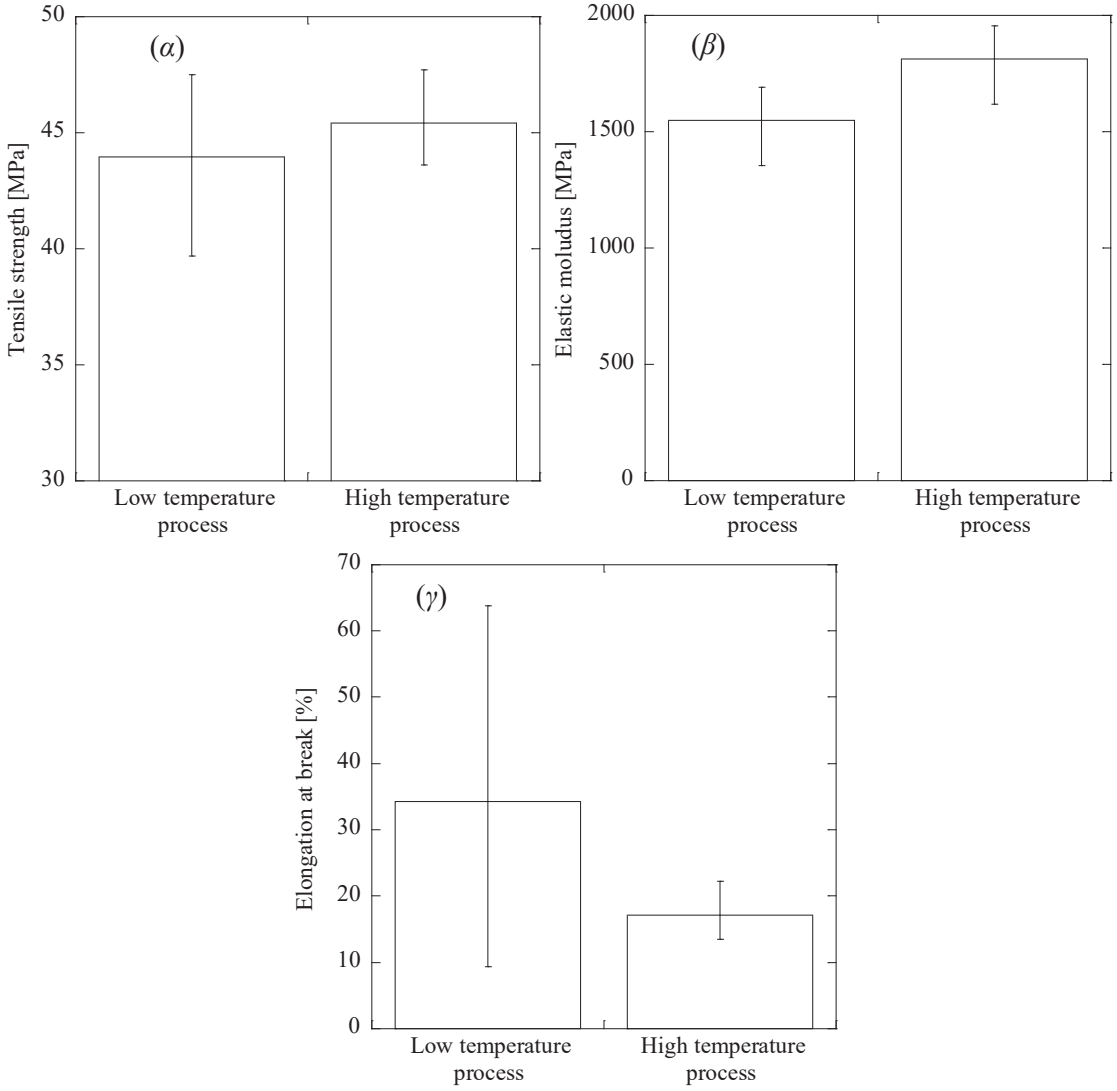

Fig8. Comparison of mechanical properties of parts for low temperature process and high temperature process. (α) shows tensile strength, (β) shows elastic modulus, (γ) shows elongation at break, respectively.

Discussion

The parts for low temperature process had lower strength than the parts for high temperature process. However, it was better in elongation at break. This indicates that solidification at low temperature quenches the melted polymer to lower crystallinity and increases high ductility resultantly. The large deviations shown in strength and elongation at break of parts for low temperature process are assumed to be caused by factors other than crystallinity, such as crystal size and void distribution, and further investigation is required.

The second peak observed at 185°C in DSC curve for high temperature process shows that some powder grains remained not melted. On the other hand, the small peak at around 170°C for the low temperature process indicates that each crystal grain of the parts is smaller than those solidified at a higher temperature. The melting point of semi-

crystallized polymer depends on the thickness of lamellar crystals that is component of the crystal [8]. When the thickness of lamellar crystal is thinner, melting point becomes lower. The low melting peak temperature indicates that thin lamellar crystals were formed when the powder bed temperature is low.

When the parts are processed at high powder bed temperature, powder grains remains not completely melted [9]. Those grains remaining not melted become nucleus of crystallization, and crystallization speed is lower when bed temperature is higher. As a result crystallization is enhanced. When powder bed temperature is lower, in contrast, all the powder grains are completely melted. Thus, no nuclei exists when crystallization is initiated. In addition, crystallization speed is much higher because of the low bed temperature. As a result, molten polymer was quenched.

These results lead different crystallization and microstructure in deferent powder bed temperature.

Conclusion

Parts for high powder bed temperature had high crystallinity and strength, parts for low powder bed temperature had low crystallinity and high ductility. These results indicate that the powder bed temperature affects the mechanical properties and microstructure of laser sintered parts. It is suggested that by changing the powder bed temperature, the crystallinity and mechanical properties of the laser sintered part can be controlled to make desired parts.

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