Microstructural Observation and Mechanical Property Evaluation of Plastic Parts Obtained by Preheat Free Laser Sintering

Toshiki NIINO, Hisashi HARAGUCHI, Yutaro ITAGAKI, Kentaro Hara and Susumu Morita
Institute of Industrial Science, the University of Tokyo
4-6-1 Komaba Meguro Tokyo, 153-8505 Japan
niino@iis.u-tokyo.ac.jp
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ABSTRACT

Tensile test on preheat free (PF) processed part was performed it is shown that ultimate strength is the same as that from conventional process when relative density is the same. Microstructural observation showed that decomposition occurs during PF process. Microstructure of PF processed part is similar to those of amorphous. It is indicated that preheat free process can improve geometrical precision. PGA to which conventional process cannot be applied was successful processed by PF.

Introduction

Additive manufacturing (AM) has a great potential to be a major player of nonprototyping part production owing to its ability of fabricating difficult or even impossible parts due to shape complexity. In the variety of AM technologies, plastic laser sintering had been commercialized in the earlier periods for the both of prototyping and production purposes. The process selectively consolidates a thin layer of powder bed surface into a designated slice shape in melt and freeze fashion by exposing laser beam and repeats this layer by layer until whole slices are finished. In this procedure, the material property of the powder that is essential to layer formation and lamination is thermoplasticity. Since this property is common to most engineering materials and is utilized commonly by conventional fabrication methods such as casting and injection molding, laser sintering process is applied to conventional material and accepted by industries more easily than the other AM processes. Resultantly, plastic laser sintering is having been a major option in many rapid manufacturing applications until now, and is promising in the future as well.

Plastic laser sintering is one of the rare AM processes that do not require fixture of processing parts with sacrificial bodies known as “anchors” or “supports” to prevent the part from warping. The potential cause of the warping in additive manufacturing with selective consolidation basis is, primarily, a volume variation on consolidation;

shrinkage on crystallization of molten material and following thermal shrinkage of the top layer pulls previously finished layers and causes a curl distortion, resultantly. However, the layer-by-layer shrinkage is minimized in plastic laser sintering by a counter measure as following. Some semi-crystalline plastics have a wide gap between its melting point and crystallization temperature. The most commercialized plastic laser sintering processes employ such plastics and maintain the powder bed temperature in the gap until whole layers are processed. Therefore, the powder grains that have been once melted by laser exposure and adhered to each other do not solidify or crystallize but stay in supercooling condition. Consequently, thermal stress within the processing part does not occur during sintering process or layering process. After whole layers have been finished, slow cooling is performed to avoid distortion[1]. This anti-distortion measure is quite advantageous in terms of its unnecesity of removing anchors, but it also brings many disadvantages. From the view point of material development, the requirement for maintaining the gap between melting and crystallizing temperature, which is referred by "process window" in the following description, is a constraint on expansion of applicable plastic species and improvement of material property by adding agents on applicable species. From the view point of machine development, preheating the powder bed with its temperature maintained precisely in the process window, increases difficulty of temperature control as build envelope is expanding and raises the cost of machine components when application to higher temperature plastic is requested. From sustainability view point, exposing such heat on the nonsintered powder for long period may give some damage on the powder which leads to low recycle rate. Additionally, the power spent for powder preheating occupies large part of the total power consumption of laser sintering machine.

The authors are developing a laser sintering process that does not require powder bed preheating. In the previous presentation at SFFS 2011[2], feasibility of the *preheat free* plastic laser sintering is discussed. Parameters of a square mesh anchor structure were optimized to provide sufficient adhesion to the base plate and maximize ease of following anchor removal. Effectiveness of stress relief annealing was proven. Tensile and impact strength was roughly measured.

In the present research, deeper investigations into the effect of the novel process especially on microstructure and mechanical property were carried out. Strength data are updated and elongation is also measured. Discussion on pore formation which strongly affects various mechanical properties is performed on the basis of microstructure observation. Precision of preheat free process is also investigate. A biodegradable plastic that does not fit traditional laser sintering due to its high melting

point and low powder flowablity at high temperature is tested on preheat free process. Additionally, effect on high cooling rate of preheat free process on mechanical property is demonstrated.

In the following description, we refer preheat free process and normal with-preheat process with PFP (preheat free process) and HTP (high temperature process), respectively.

MATERIAL AND METHODS

POWDER, MACHINE AND BASE PLATE

A commercially available polyamide 12 powder (DuraForm PA, produced by 3D Systems) and poly Glycol acid (PGA) powder were used. PGA is a semi-crystalline biodegradable thermoplastic. Molecular weight of the tested material is varying between 170,000 and 200,000. The melting, recrystallization and glass transition temperatures in terms of heat flow peaks are 220°C, 95°C and 40°C, respectively. This plastic performs differently when it is in crystalline or amorphous state. For example, the densities of crystalline and amorphous are 1.7 and 1.5, respectively. Granules for regular injection molding were ground and sieved between 100μm and 150μm. Though the powder flows very well at room temperature, it becomes so cohesive in high temperature range more than 130°C that regular flat surface cannot be recoated.

A machine developed by the authors [4] was used. The machine is equipped with CO_2 laser source (GEM-30, Coherent Inc.), which can emit a single mode laser with a wave length of 10.6μm at the maximum output power of 30W. The laser beam is focused into a 130μm diameter ($1/e^2$) spot on the powder bed with a zinc selenide lens of 109mm in focal length. The beam can be scanned by a galvanometer mirror system in a range of 100mm × 100mm. Powder recoating is performed with powder feed cylinder-piston mechanism and a roller system. The roller is 20mm in diameter and rotates with a DC servo motor. Being placed on a stepper-motor-driven linear actuator, this roller-motor system can rotate the roller at any designated speed in any direction independently to the roller traversing speed. In this research, a counter rotation mode, which is applied in typical commercially available system, was used.

Adhesion between a constructed parts and base plate should be strong enough to resist the warping force. For this purpose, the materials of the powder and the base plate should desirably be the same. For PA12, a base plate was fabricated by the normal with-preheat selective laser sintering from the same material [2]. To strengthen the plate, it was backupped with a rigid aluminum plate. Since PGA could not fit the normal sintering process, a 2mm thick plate was prepared by injection molding. Though

the PA12 plate was screwed to the backup plate, the injection molded PGA plate was glued with epoxy adhesive since the available thickness out of injection molding was not enough to hide the screw head.

MECHANICAL PROPERTY MEASUREMENTS AND PREPARATION

Tensile tests following ISO 527 were carried out on PA12 parts. Since standardized specimen for tensile test could not be obtained due to size limitation of build envelope, specimen design as displayed in figure 1 was used. Build direction of each specimen was rectangular to the pulling direction in the tensile tests. The results were compared with those out of HT process. The reference specimens were fabricated with a beta prototype version of Semplice produced by ASPECT Inc. On PGA parts a standardized three-point bending test (ISO 178) was performed. The process parameters were tuned so that the designated relative densities could be obtained. Process parameters for the both of PA12 and PGA, HT PA12 process as a reference are summarized in table 1.

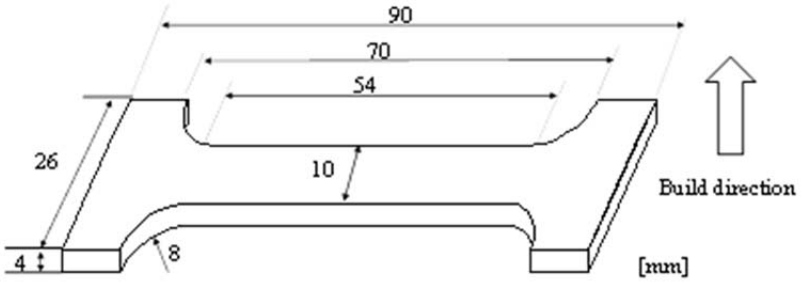


Figure 1 Design of the specimen employed in tensile tests

Table 1 Parameters for 90% relative density from preheating process. Relative densities for PFP will be reviewed in the following result section.

Process	HT	PF	
Material	PA12	PA12	PGA
Beam spot size [μm]	550	130	130
Beam power at powder bed surface [W]	8.0-14.0	7.5	13.7
Scan Speed [m/s]	3.78	1.53	3.81
Scan Spacing [μm]	150	30	30
Powder Bed Temperature [°C]	177	–	–
Relative Density [%]	87-96	87	70

MICROSTRUCTURE OBSERVATION

Microstructures of specimens' cross sections were observed with a transmission optical microscope (VH-5000 for microscope and VH-Z100 for objective lens both of which were KEYENCE Inc.) A polarizer facilitated the microstructural observation. A specimen which is equivalent to ISO 180 impact test was prepared. Thin film is sliced from it by manual cutting with a utility knife as shown figure 2. Thickness of the slices is

supposed to varies approximately between 20 and 40μm.

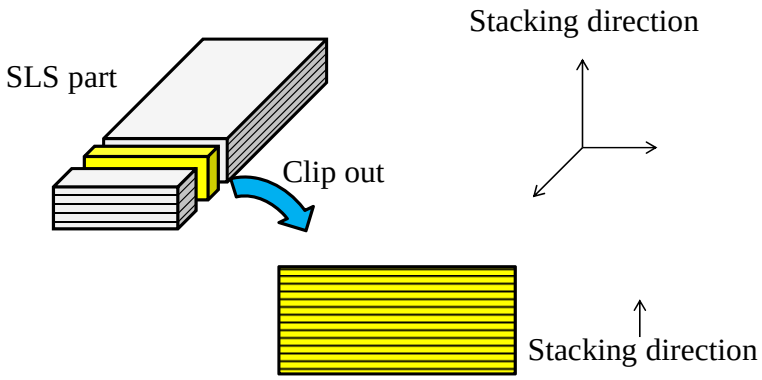


Figure 2 Preparation of specimen for transmission optical micrographs

EVALUATION OF PRECISENESS

We will discuss the preciseness of plastic laser sintering by evaluating the amount of excessive sintering. Excessive sintering naturally means unintended sintering or sintering where laser did not shoot. However, the issue is that we cannot distinguish exposed area and nonexposed one due to Gaussian laser distribution. In the case of present research, the laser intensity gradually decreases in the range of 70μm, i.e. half of the beam diameter of 130μm, approximately from the beam spot center and still remains at very low level even out of the range. Our problem is that precision we wish to talk about is in the equivalent dimension to the range or the spot diameter. To avoid this confusing situation, we will adopt the idea of nominal exposure area where the center of beam ran over. In present research, evaluation of precision in forming thin wall and small hole in vertical direction; thickness of excessive sinter out of nominal exposure area as shown in figure 3.

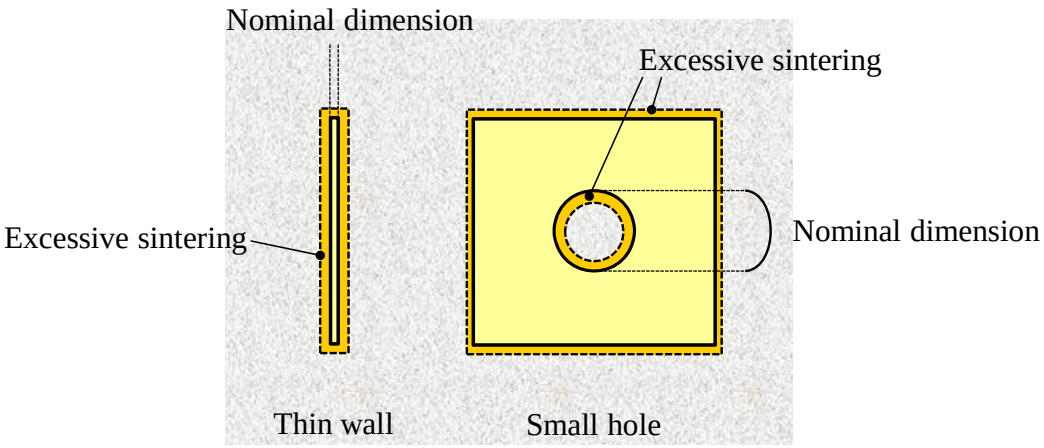
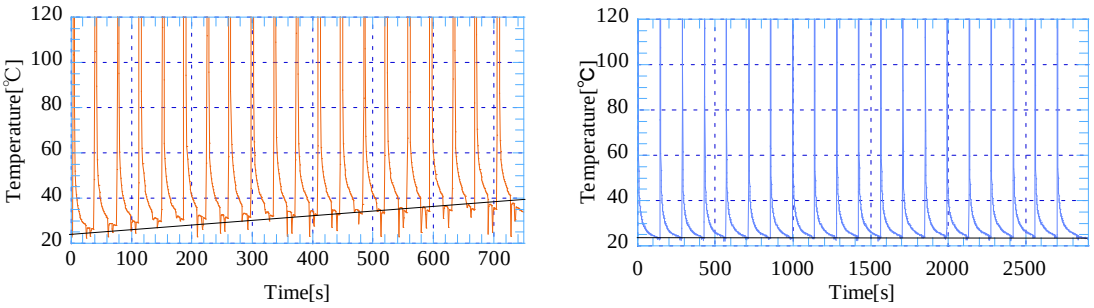


Figure 3 Definition of excessive sinter

EFFECT OF HIGH COOLING RATE

In PF process, it is possible to cool molten plastic to almost room temperature at higher rate than HT process which requires intentional slow cooling spending some hours. Figure 4 shows a temperature measurement of PF process of PGA powder. To avoid temperature increase during layer formation, layering cycle was extended to 120s. As a result, powder bed temperature was kept less than 25°C while the t_g of PGA is 40°C. The obtained part is supposed to be amorphous. To confirm it, heat treatment at 120°C for 3hours followed by low rate cooling over 3hours to 30°C was performed, and bending test results were compared before and after.



(a) layering cycle = 50s (b) layering cycle = 120s

Figure 4 Powder bed temperature for high cooling rate tests.

RESULTS

TENSILE TESTS

Relative density of tensile test parts obtained by PF process was 87.4%, and UTS (ultimate tensile strength) was 29.1±0.7MPa (mean±SD). This strength is slightly greater than those parts from HT process that have the equivalent relative density. UTS of HT processed parts increases as their relative density increase and reach the datasheet equivalent value of 43MPa when the density is 95%(Figure 5). There is a clear difference in stress strain characteristics between PF and HT processes. Modulus of PF processed parts is roughly half of those of HT process. In figure 6, stress strain charts for a PF part and HT one with various relative densities are displayed. Elongation at break was tripled by PF process, and the top value reached more than 26%. It is even greater than those out of higher density HT process and datasheet value.

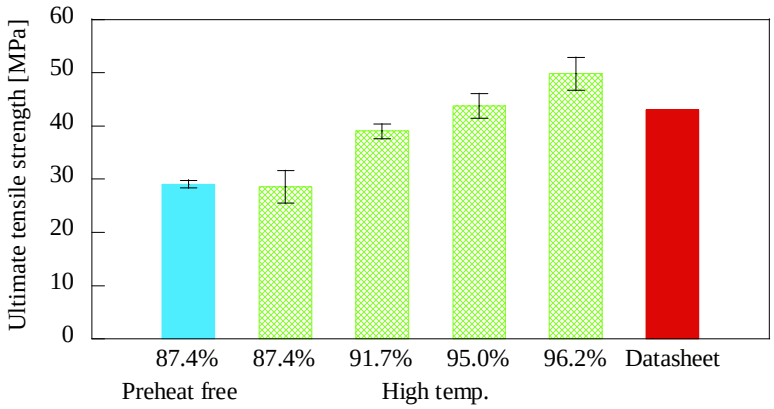


Figure 5 Ultimate tensile strengths for PF process and HT process with various relative densities

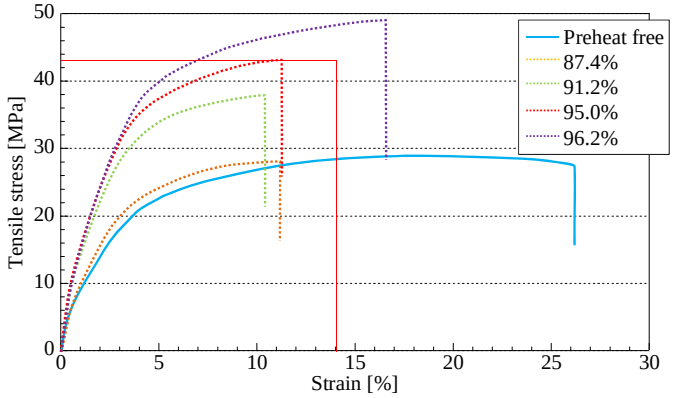


Figure 6 Stress Strain charts for PF process and HT process with various relative densities. Red strait lines show UTS and elongation at break of PA12 (Duraform PA) in datasheet

MICROSTRUCTURAL OBSERVATION

Figure 7 is a typical transmission optical micrograph of a HT processed part. We can find unmelted grains and spherulites surrounding them[3]. There are pores with irregular shapes which are forming strata with a regular thickness of 100μm. Figure 8 is a transmission micrograph of a PF processed part. We do not find unmelted grains but small spherical pores as well as irregular shaped pores. The spherical pores also form strata, but whose thickness is 200μm. Figure 9 is a polarized transmission micrograph of a PF processed parts. We can see some striped contrast near the irregular pores but they are less than the HT process's case. When the laser energy is high enough to give a yellow color to the specimen, it is observed that the spherical pores increase in both size and number.

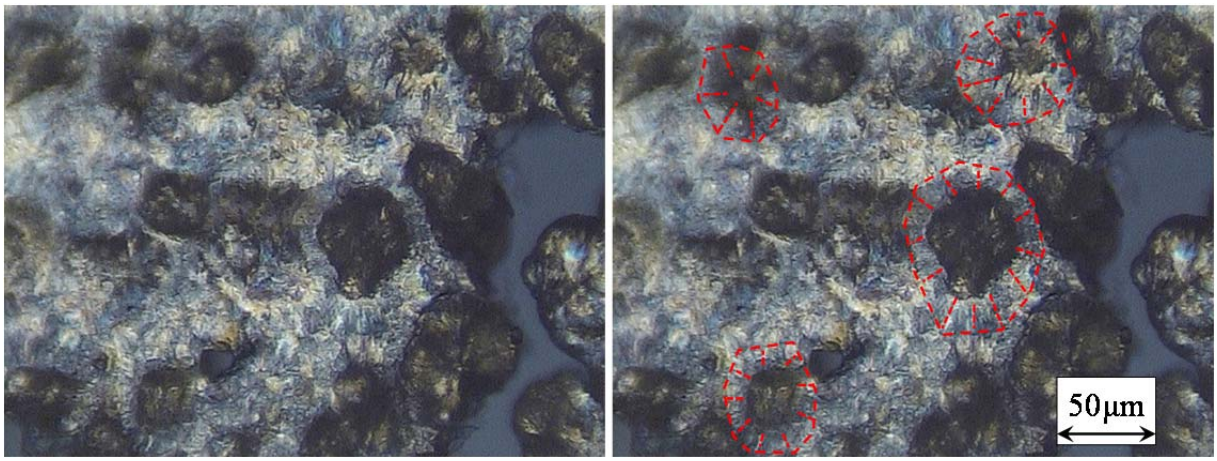


Figure 7 Polarized optical transmission micrograph of an HT processed part. A pore of residual air and nonmelted grains are observed. Around each of the grains, shperulite are formed as illustrated by red lines in the right side picture.

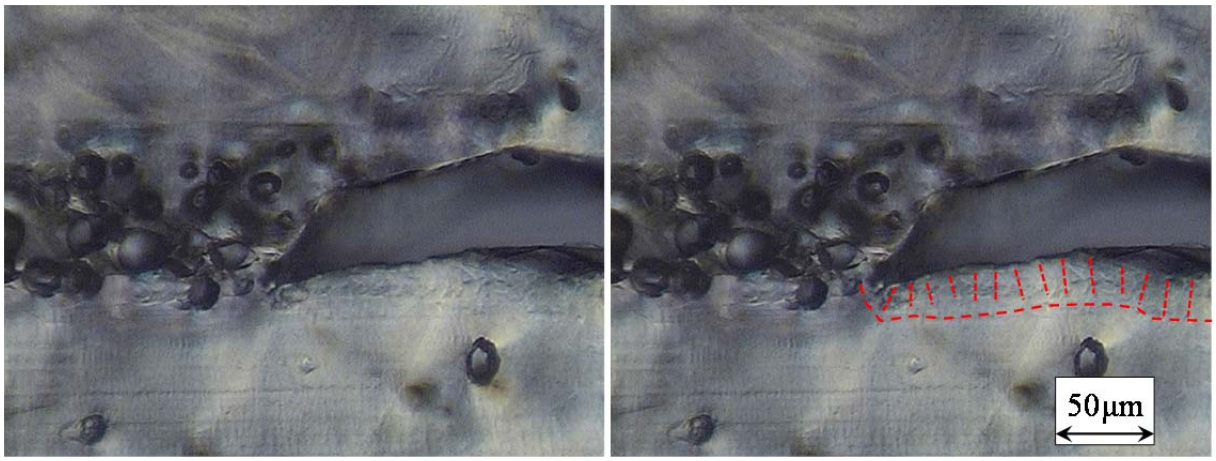


Figure 8 Polarized optical transmission micrograph of a PF processed part. Striped contrasts are found near the pore edge as shown in the right picture.

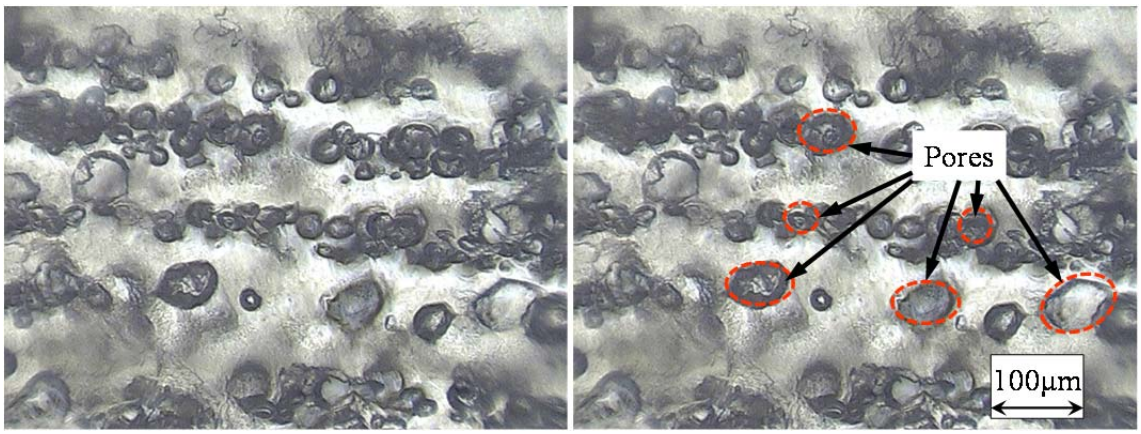


Figure 9 Optical micrograph of an excessively exposed part

DIFFERENCE IN PRECISENESS BETWEEN PF AND HT PROCESSES

Table 2 summarizes nominal thicknesses of a vertical thin wall and their actual thicknesses obtained by PF and HT processes. The difference between nominal thickness and actual thickness, which is expansion in thickness, can be an index of excessive sinter. For every nominal thickness, excessive sintering was smaller when PF process was used. The excessive sinter decreased as the nominal thickness increased for the both of PF and HT processes.

Table 3 summarizes nominal diameter of a vertical hole and their actual diameter obtained by PF and HT processes. Similarly to thin wall case, PF process decreases the excessive sinter. The relationship between the excessive sinter and nominal hole diameter is again similar to thin wall case. We can visually observe the edge of the hole is sharper and amount of not removed powder grain is fewer when PF process is applied.

Table 2 Nominal and actual thickness of 3.5mm high vertical wall

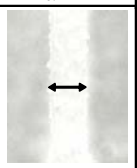
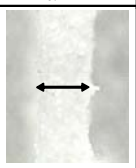
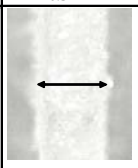
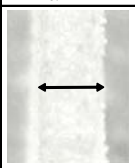
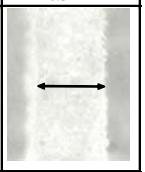
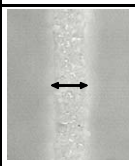
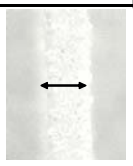
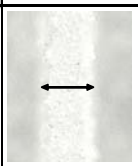
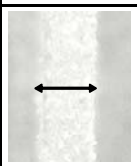
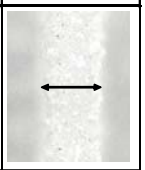
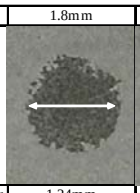
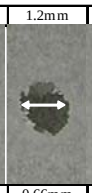
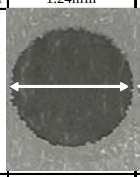
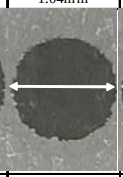
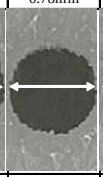
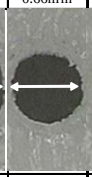
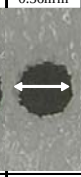
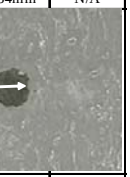
	Nominal Thickness	0.1mm	0.2mm	0.3mm	0.4mm	0.5mm
High Temperature						
	Actual Thickness	0.32mm	0.43mm	0.50mm	0.54mm	0.60mm
Preheat Free						
	Actual Thickness	0.24mm	0.37mm	0.40mm	0.45mm	0.51mm

Table 3 Nominal and actual diameters of 3.0mm high vertical hole. Holes were not pierced when the nominal diameter < 0.6mm with the both process.

	Nominal Diameter	1.8mm	1.6mm	1.4mm	1.2mm	1.0mm	0.8mm	0.6mm
High Temperature								
	Actual Diameter	1.24mm	1.04mm	0.78mm	0.66mm	0.56mm	0.34mm	N/A
Preheat Free								
	Actual Diameter	1.58mm	1.36mm	1.14mm	0.94mm	0.70mm	0.48mm	N/A

FABRICATION FROM PGA POWDER AND EFFECT OF HIGH COOLING RATE

Preheat free laser sintering process successfully work on PGA powder to which conventional process cannot be applied. Obtained relative density was as low as 71% at the maximum. Figure 10 shows examples of stress strain charts for three-point bending

test before and after heat treatment, and various parameters are summarized in table 4. As shown here, UBS and bending modulus were increased, and strain at break was decreased after heat treatment. The difference was roughly 10% for each parameter.

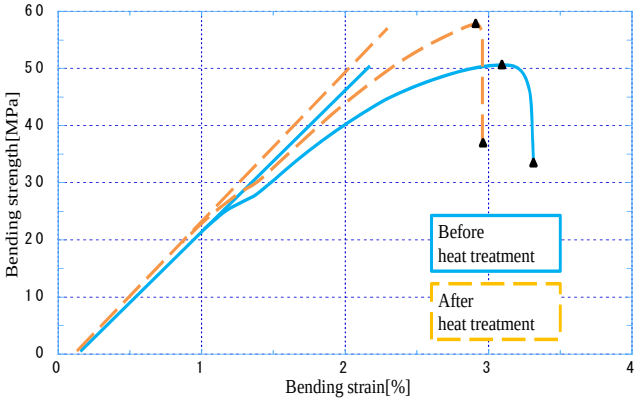


Figure 10 Stress-strain diagram (bending) of a PF processed PGA part before and after heat treatment.

Table 4 Summary of bending tests on a PF processed PGA part before and after heat treatment

	Before	After
Maximus Load [N]	84.1	98
UBS [Mpa]	45.4	51.9
Maximum Bend [mm]	5.3	4.3
Maximu Strain [%]	3.2	2.7
Modulus [Mpa]	2300	2550

DISCUSSIONS

TENSILE TESTS

The same tensile strength was obtained by preheat free process when the same relative density as conventional process is obtained. This indicates that binding force of the powder is the same. To obtain such high strength as datasheet value for conventional process, we should lower the porosity. To achieve this less viscous material is required, and such material must have different mechanical property. To go further we need discussion from more chemical side.

MICROSTRUCTURAL OBSERVATION

Absence of nonmelted powder grain in a PF processed part shows that the process is not sintering process but melting.

Smoother microstructure observed in polarized transmission images indicates that crystalline rate is much lower than in the case of conventional process. The cause of this is supposed to be high cooling rate.

There were two types of pores in a PF processed part. One is irregularly shaped and the other is spherical. Irregular one is obviously residual air. The other one is supposed to be decomposition gas from the material. The average energy given to the powder in PF

process can roughly be calculated as 1.8kJ/g. This value is equivalent to the heat which required for decomposition of the PA from room temperature [4], while it is roughly estimated the temperature in conventional process reaches 220°C. Additionally, increase in number and size of the pore strongly supports our supposition. In the fabrication process, cross hatch scanning strategy was adopted. In this strategy, scanning interval in time is alternating every two layer because of the difference in scanning path length. Consequently, the highest temperature of each layer should change alternatingly at every two layer. In the transmission micrograph of a PF processed part, we can find formation of strata at every 200μm which is the same interval as the same scanning strategy occurs. This result also supports our supposition with respect to occurrence of decomposition.

Having compared thin walls and holes out of PH and HT, we found clear difference in the amount of excessive sinter. Cause of this seems the difference in temperature gradient of part edge. In preheat free process, the highest temperature of exposed area is as high as decomposition temperature while the temperature of conventional process is only 220°C as mentioned above. On the other hand, temperatures of unexposed region are room temperature and 180°C for PF and HT processes, respectively. Thus, there is a great difference in temperature gradients.

Difference of mechanical property before and after heat treatment of PGA indicates effect of the fast cooling rate. We need more investigation about this topic such as microstructure observation and fabrication in various thermal conditions and histories.

CONCLUSIONS

It is shown that preheat free process can provides the same tensile strength when the same relative density is obtained. However, more material development is required to obtain the strength as high as datasheet value of Duraform PA. Microstructural observation provided us with various information. Preheat free process is melting process. In the process, temperature of the powder might be elevated to high temperature that can cause decomposition. Microstructure is similar to amorphous that was obtained by high cooling rate of preheat free process. This is also supported by comparison of mechanical properties before and after heat treatment. PGA to which conventional process cannot be applied was successful processed by preheat-free. Preheat free process is advantageous in terms of precision.

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