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The Application of Scanning Electron Microscope and Melt Flow Index for Orange Peel in Laser Sintering Process

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Abstract

In manufacturing, many products need to undergo increasing customisation, and a shortening of the manufacturing cycle time. This makes the time needed to produce prototypes one of the most important contributors to product development cycles. Rapid Prototyping (RP) offers the user the ability to optimise part design in order to meet customer requirements with few manufacturing restrictions. One of the most common RP processes is Laser Sintering (LS). A problem with LS is that sometimes the surface of the parts produced displays a texture similar to that of the skin of an orange (the so-called orange peel texture). This problem must be addressed before the technology can gain wider acceptance. The main aim of this research is to develop a methodology of controlling the input material properties that will ensure consistent and good quality of the fabricated parts. From the experiment, it was found that PA12 powder with high melt flow rate, low melting temperature, low glass transition temperature and low degree of crystallization temperature could improve the sintering process to produce a good Laser Sintering (LS) parts with lower shrinkage rate. The powder which has higher melt viscosity and lower melting heat becomes liquid more easily and therefore flows better during the sintering process due to a shorter chain molecular structure. The results of experimental work indicate that the melt viscosity, and part surface finish are correlated.

Keywords: *biometrics system, multimodal, facial recognition, system reliability*

1. Introduction

Rapid Prototyping refers to the physical modelling of a design using a special class of machine technology which allows manufacturers to improve product quality and reduce both times to market and cost. Because of the development and pre-production stages of rapid prototyping, it is often necessary to have small batches of parts for testing and evaluation purposes.

One of the most effective and versatile RP techniques available is Selective Laser Sintering (SLS) or the Laser Sintering (LS) process. In this technology, an object is created layer by layer from heat-fusible solid fine powdered materials with heat generated from a CO2 laser [1]. It is capable of producing very complex part geometry directly from three-dimensional CAD software by a quick, highly automated and high-process, flexible manufacturing process. [2].One of the major advantages of the LS process over other major RP processes is the ability to process almost any non-toxic materials, provided it is available as powder and that the fine particles tend to fuse or sinter when heat is applied [3]. The reason for having powders in particle size is that the finer particle size produces a thinner layer, better resolution and finer roughness. It also permits easier powder layer deposition and causes less shrinkage during the laser sintering process [4]. Polymer powders were the first, and are still the most widely employed, materials in LS [3]. The use of polymeric materials in the LS process offers some advantages which are related to the low processing temperatures, melting flow control and ease of production [2-4]. The advantage of using PA semi-crystalline as an LS material is that it has good physical properties with a high melting temperature due to the strong hydrogen bonding [2].

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Rapid prototyping has been significantly developed over the past two decades and is rapidly recognized as an alternative to manufacture real functional parts. One of the main advantages of employing Laser Sintering processes is that loose powder of the building chamber can be recycled. An insufficient amount of new powder results in poor quality and rough surface finishing known as orange peel of the produced parts [5-7]. This because of the longer build stage period in the LS process increases the chances that loose powder in the build cylinder may become badly deteriorated due to exposure to high temperature which is significantly influenced by the thermal history of the loose powder in the build cylinder.

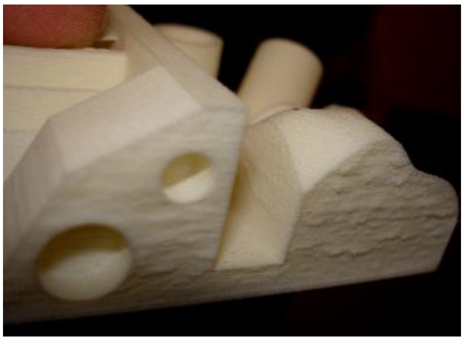

Figure 1. Laser Sintered part affected

Figure 2. Good part surface by orange peel texture

1.1. Past Research

At present, PA12 based powdered materials known as PA2200, supplied by EOS GmbH used to create functional plastic prototypes. Fabricating parts using only new powder, although providing the best quality, is significantly more expensive than using recycled powder and is impractical both in terms of time and cost especially in a production environment. On the other hand, using just recycled powder creates a problem in that a coarse, rough, and uneven surface texture is achieved and often referred to as orange peel (Figure 1) compare to good quality of LS sintered part as shown in Figure 2.

Prior to this study, Gornet [8-9] studied how the mechanical and thermal properties of LS parts from DuraForm (trade name of a PA12 powder produced by 3D Systems [10] material were affected by the number of builds or the number of times the powder was used. One of the conclusions was that after approximately 7-8 builds the properties of the processed material were so badly deteriorated that it was recommended that the remaining material be fully discarded. However, the explanation of the Orange Peel phenomenon in the Laser sintering process which related to thermal properties, melt viscosity, and part surface finish was not investigated. The aim of this research is to investigate the influence of glass transition temperature, crystallization temperature, melt flow rate, temperature, and the number of exposures on these properties is investigated.

2. Research Method

The aim of this experiment is to determine the quality of fresh PA12-based powder PA2200 and recycled PA2200 powder grades supplied by EOS, Germany. Variations in the new and used powder quality that are currently employed were tested, and the results were analyzed in order to investigate the thermal properties of the powders.

2.1. Samples Preparation for Scanning Electron Microscope (SEM)

Before gold coating, an interesting area that shows sintering between the powder particles was identified in the x-y (direction of laser scan) plane and y-z plane (perpendicular direction of laser scan). To obtain access to these areas, the sintered part was cut by breaking it in the y-z plane and y-z plane. As a similar procedure was applied for the powder specimen preparation, a small part of the sintered part sample was stuck on the specimen holder using glue. Afterwards, it was coated with gold and was ready for image capturing into the EMSCOPE SC500 Cam Scan.

2.2. Design and Fabrication of Bench Part

The model of the sintering bench part test using Pro-ENGINEER is shown Figures 3a and 3b. The special features incorporated in this model.The size of the benchmark part is 110mm (w) X 110mm (l) X 48mm (h) The file was then converted into STL format before it was transferred to the LS machine (Sinterstation 2500 HiQ).

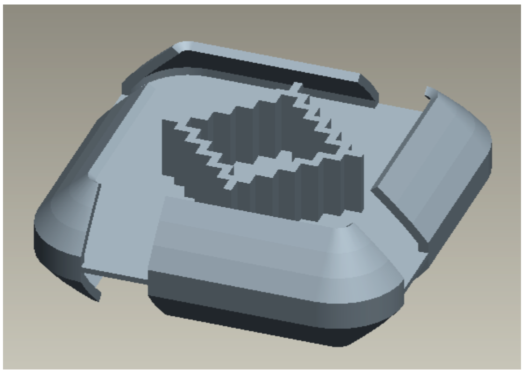

Figure 3a. Benchmark part (top view)

2.3. Melt Flow Rate Indexer (MFR)

The Melt Flow Rate (MFR) measures the flow viscosity of a molten polymer when extruded through a capillary die under specific temperature and load conditions. The flow ability of any polymer depends on its chemical structure. Polymer chains with simple geometry and short length slide past one another relatively easily with low flow resistance. By contrast, long chains of high molecular weight and complex structure yield greater flow resistance or viscosity [11].

The MFR was selected as a criterion because the flow characteristics of a molten polymer are very sensitive to changes in the basic polymer structure and its molecular weight [9]. The basic polymer property which is measured by this test is the molten plastic flow at a particular shear stress (related to the applied load) and temperature as illustrated in Figure 4. In this case, the MFR test provides a relatively fast and inexpensive method of measuring the rate of PA12 powder degradation because of the LS process.

For each sample, 6 MFR measurements were taken to calculate the (average) result. The coefficient of variation of this experiment is about ± 3 %. The MFR experiments were performed according to ISO1133 standard [12]. A small amount of PA2200 material (8-10 grams) was extruded for 10 minutes, at a temperature of 235 ºC under a weight of 2.16kg. The measured MFR units are in g/10 min [13, 14].

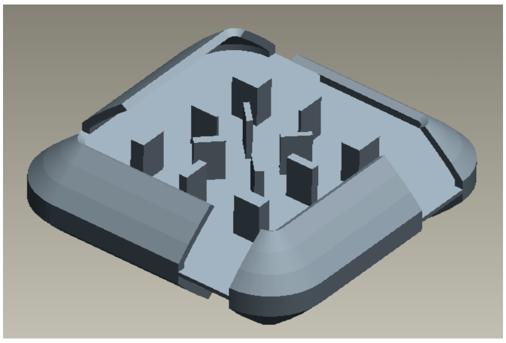

Figure 3b. Benchmark part (bottom view)

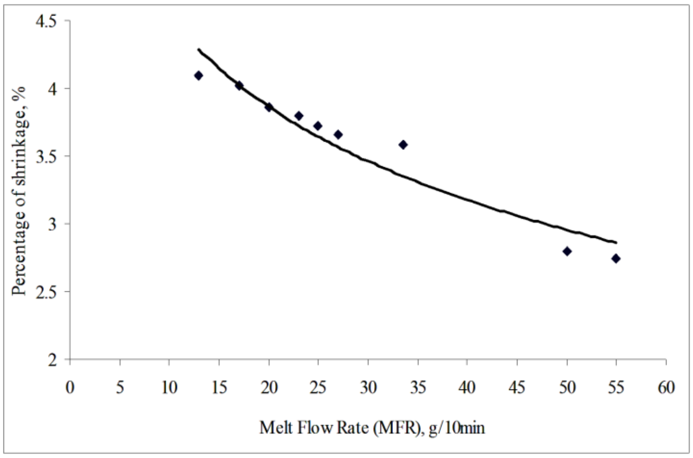

Figure 4. Effects of shrinkage and MFR to sintered part

3. Results and Analysis

As shown in Table 1, it is found that powder quality, thermal properties and melt viscosity are correlated. The glass transition temperature and melting temperature increase for PA12 powders used many times, while the MFR decreases. Although glass transition temperature and melting temperature change slightly, these changes are not very sensitive to the changes in powder deterioration. Therefore, it would be difficult to use any of these temperatures as a criterion for controlling the powder quality and powder recycling process. In contrast, the MFR responds significantly to small changes in PA12 powder quality due to deterioration. For instance, when 65% 1X Recycled material is mixed with 35% New material, the MFR changes from 28.9 to 33.13, while the glass transition temperature and melting temperature decrease by only 1ºC. The higher MFR means better PA12 powder quality. The lower the MFR and Crystallization temperature, and the higher the melting temperature and glass transition temperature the worse is the PA12 powder quality.

Each polymer displays unique thermal behavior, the most important characteristics that determine the application of thermoplastic polymers in the LS process are glass transition temperature, melting temperature and Crystalline temperature [13], [14]. This due to the polymer powder experience changes transition phases through the LS process cause changes its properties. Figure 4 depicts the average shrinkage of LS parts built from PA2200 powder with different grade and MFRs. This due to the more efficient packing of the PA12 polymer chains causes higher melt viscosity, which also leads to an increase in the LS part’s shrinkage.

Table 1. Comparison of different PA12 powder grades and their thermal properties

PA 12 powder	Melt Flow Rate g/10min	Glass transition Temperature (˚C)	Melting temperature (˚C)	Crystallization Temperature (˚C)
New PA2200 powder	51.50	53.95	186.49	149.97
35% new mixed with 65% once recycled powder	33.13	56.93	187.22	150.11
once recycled powder	28.90	57.78	188.53	148.82
Twice recycled powder	17.41	56.93	189.27	148.27
Three times recycled powder	13.50	58.11	191.48	147.26

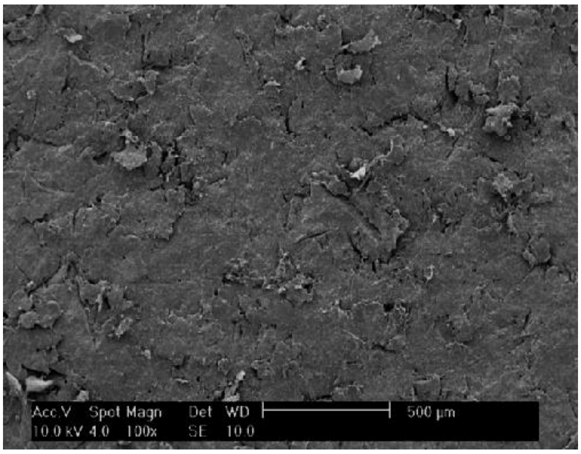

Figure 5a. Smooth surface

The external surfaces of good and orange peel parts are shown in Figures 5a and 5b, clearly differentiating the two surfaces finishes. It was found that the higher MFR used in the LS process, the lower the melting viscosity. This results in the polymer particles within the layers fusing easily which leads to a smooth surface. By contrast, employing the twice-times used powder, agglomerates of melted particles can be observed, which are in the direction of the laser scanning lines. Between the sintered lines, extended holes and cavities can be observed.

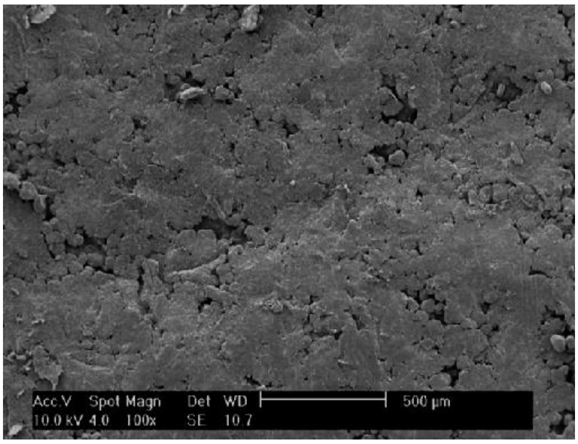

Figure 5b. Uneven and rough surface

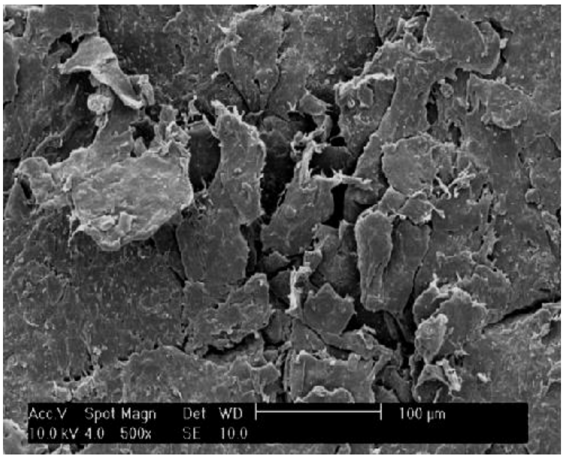

Figure 6. Small voids

Figure 6 shows a close-up view of the good surfaces. They were most likely caused by the contact with unsintered particles while the surface was still molten. The formation of microstructures with highly dense morphology, and only small voids, can be observed.

The higher melt viscosity is related to a bigger spherulite size. Figure 7a shows i the centres of the single spherulites, the origins of crystallisation are clearly visible but nucleating particles cannot be detected. This is considered to be a sign of the deterioration of the polymer material. This means that it is not possible to seal off the voids due to inefficient melting of the polymer. As shown in Figure 7b, it is observed that the cavities have different shapes and sizes and it is estimated that the average cavity size is approximately 100μm to 200μm, as may clearly be seen.

PA12-based powders are the most used materials in the LS process for RP of plastic parts. An average of 80% to 90% of the powdered material in the building chamber is not sintered during the LS and could be reused. However, the PA12 powder properties deteriorate due to the high temperature, close to the material melting temperature, for a long period of time through the LS building and cooling cycles. The recycled powder needs to be blended with a sufficient quantity of new material in order to produce parts with good quality. Adding too much new powder would increase the cost because of the high cost of the powder within the total manufacturing cost. Using too old recycled powder or less new powder would result in poor quality and waste. In all LS machine designs, the temperature in the building chamber after LS is maintained very close to the PA12 crystallisation temperature. However, in the 3D Systems LS machines, the build starts to cool down after a certain build height while in the EOS LS process, this temperature is maintained high until end of the build. This means that in the second case the un-sintered powder would be subjected to higher temperature for longer time, which ultimately would reduce its quality more than in the first case.

Causes of the orange peel phenomenon related to changes in the properties of the LS material, in particular its melt viscosity, due to exposure to high process temperatures have been presented. The quality of the powder used in the LS process is one of the most important parameter determining the final part quality. The deterioration rate of the recycled PA2200 varies depending on the LS machine process parameters, on build height and part volume. The deteriorated powder properties are the function of time and temperature through the LS process. This causes the quality of the recycled powder to deteriorate due to changing the morphology chain structure, which affects the amorphous and crystalline regions. Ultimately the powder ageing affects the PA12's thermal and mechanical properties.

PA12 powder qualities refers to a method of utilising used powder to gain better control of used powder quality. For instance, refreshed powder quality after the deteriorated PA12 powder is blended results in a 10% to 50% better powder quality. However, not all grades of powder can be recycled. The powder which has higher melt viscosity and lower melting heat becomes liquid more easily and therefore flows better during the sintering process due to a shorter chain molecular structure. SEM micrographs show that the morphology of the external surfaces and cross sections of parts affected by the orange peel texture exhibits a very inhomogeneous structure after laser sintering. On the basis of the experimental results, a qualitative LS mechanism has been proposed to describe the orange peel phenomenon.

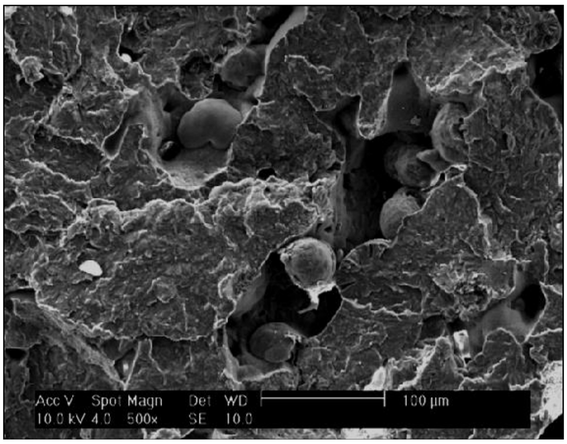

Figure 7a. Particle core of orange peel microstructure

The explanation of the orange peel phenomenon can be understood by a multistage model. At first, nearly all laser energy is absorbed by particle powders, causing local heating. Then, a part of the particle is melted, which leads to complete melting of the surrounding polymer and the formation of sintering necks. At the build stage, the melt viscosity of the polymer is significantly influenced by the molecular chain entanglement (spherulites), the higher molecular weight, and longer molecular chains, leading to higher viscosity and to the formation of agglomerates of melted particles. Lowering melting viscosity generally leads to a more highly dense microstructure and exhibits dimensional stability and is non-porous. The physically refreshed powders that were sintered exhibited good interconnectivity. This results in an inhomogeneous structure and a poor coherence sintered region.

From the observation of Figure 7a, sintering effects among the particles are clearly noticeable. It can be seen that the overall voids are smaller than those in Figure 7b. The sizes of cavities are found approximately to be the size of a single particle (50µm to 80µm). However, a lot of partial core (unmolten) adheres to the surface, which leads to the formation of many small cavities. This could be because the particles do not receive sufficient heat during the sintering process.

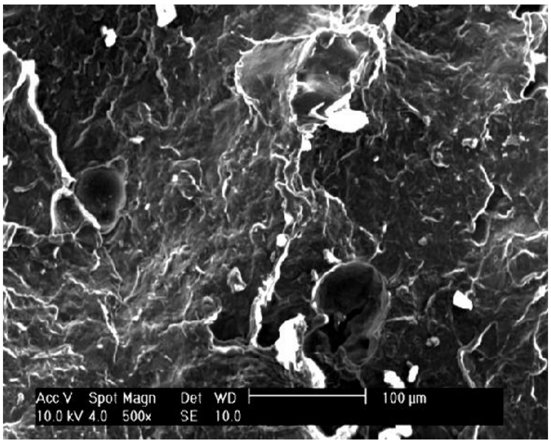

Figure 7b. Particle core of good part microstructure

4. Conclusion

One of the challenges which LS faces is the Rapid Manufacturing of plastic parts with good consistent quality. This is due to the fact that plastic powder properties deteriorate during the long periods of time through the LS building and cooling cycles. The influences of temperature and time are found to be of significant influence on the deterioration of PA12 powder.

The orange peel phenomenon is one of the main constrains in the LS process as it causes unacceptable LS parts' surface quality. It happens when deteriorated PA12 powder is employed in the LS process. This paper reported on the effect of the properties of the PA12 polymer on the microstructure of the parts which are affected by the orange peel phenomenon.

It is important to predict the temperature distribution and its effects on powder deterioration in the LS process by using a simulation model. It is useful to collect loose powder of acceptable quality from different positions in the build chamber. It will be more efficient and practical if LS machine users could determine regions of the build chamber with less powder deterioration. As a result, powder of acceptable quality would be more fully utilised due to the fact that its location is known. This can be achieved by developing a Finite Element simulation model. The method undertaken in model development might be mainly focused on specifying temperature changes expected to reflect those experienced during LS processing. Mathematical equations could be developed in order to predict temperature changes experienced at different points in a build during processing. This takes into consideration the fact that temperature distribution in both liquid and solid phase through the LS process causes the powder deterioration which leads to the orange peel phenomenon. Once the model has been developed, the density of solid parts could also be predicted.

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