EFFECT OF EMPLOYING DIFFERENT GRADES OF RECYCLED POLYAMIDE 12 ON THE SURFACE TEXTURE OF LASER SINTERED (LS) PARTS

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ABSTRACT

Laser sintering (LS) is one of the most versatile rapid prototyping (RP) processes currently available. One of the main advantages of employing this technology is that the non-sintered powder can be recycled and reused for further fabrication. Current powder recycling methodologies using a constant refresh rate with a very high portion of new material being added to the existing material reserve in order to maintain part quality and integrity. If the amount of the new powder is insufficient or if the recycled material is too “old” (i.e. has been recycled too many times), then the fabricated parts experience variation in their quality. Typical quality defects include; higher shrinkage rates and rougher than average surface textures often known as “orange peel”. This paper reports on an experimental study to investigate the significance of different deteriorated recycle Polyamide 12 (PA12) powders on the surface quality of products. The main aim of this research is to determine and acceptable ratio quantities of virgin to recycled powder that can be used before adversely affecting product surface texture. In this experiment, the melt flow rate (MFR) is chosen as a criterion to measure the recycled powder quality. The microstructures of external surface and cross sectional parts which employed the different grades of recycled powder quality were examined. The results of experiment suggested that the refresh powder target must be at least 27MFR in order to produce a LS good part surface.

KEYWORDS

Rapid Prototyping, Laser Sintering, Surface Texture, Polyamide 12, Recycling

1.0 Introduction

Laser sintering (LS) with Polyamide 12 (PA12) or Nylon 12 based powders allows the prototypes and functional parts to be produced using a dedicated machine, and is thus gaining popularity within the field of rapid prototyping technologies. 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 [1]-[2]-[3]. By using this technology parts are created directly from 3D CAD model, and then based on the slice data obtained from the STL file, a CO_2 laser draws the part cross section and sinters the powder particles. Than, the work platform moves down by one layer thickness (approximately 0.15mm). Following this a roller spreads a new layer on the part bed for the next laser scanning process (Fig. 1). This process is repeated until the part is complete.

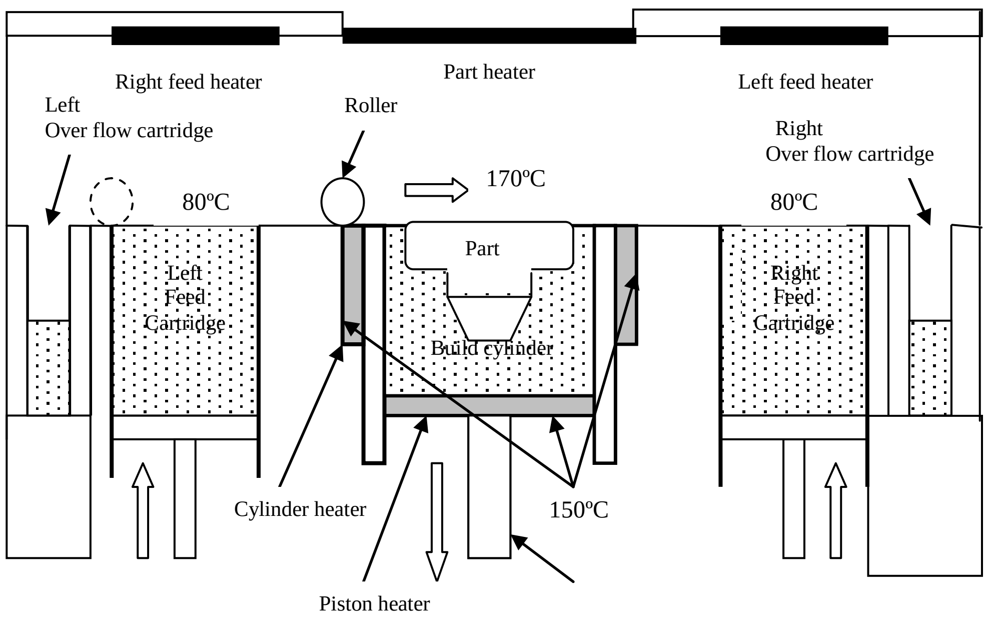


Fig. 1 Sinterstation 2500 HiQ LS machine

At present, there are two PA12 based powdered materials known as PA2200, supplied by EOS GmbH, and Duraform, supplied by 3D Systems Corp. used to create functional plastic prototypes. A major constraint that limits the application of LS as a viable Rapid Manufacturing process is the inconsistency in the part quality due to variation of the powdered material properties. This is due to as 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 the high temperatures (which can range between 140ºC to 175ºC) (Fig. 1). For instance the loose powder located around and close to the sintered part that is the hottest, is therefore exposed to the longest period of high temperature through the LS process. This is significantly influenced by the thermal history of the loose powder in the build cylinder.

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" (Fig.2).

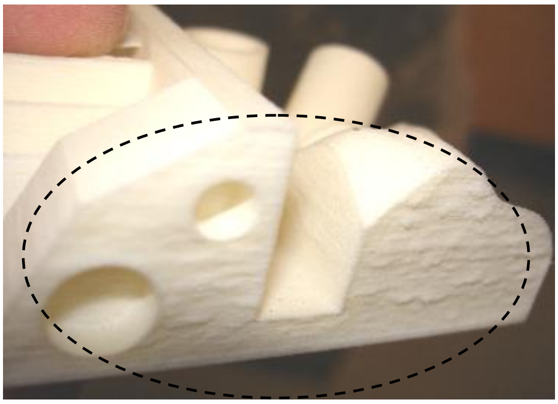


Fig.2 LS part affected by "orange peel" texture

Prior to this study, Gornet [4-5] studied how the mechanical and thermal properties of LS parts from DuraForm (trade name of a PA12 powder produced by 3D Systems [6-7] 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, how to control the input material properties in order to provide consistency in quality on the powder and part quality was not investigated.

1.1 Current powder management practice and its limitations

A normal practice is to use a constant refresh rate and to monitor the quality of the fabricated parts. If the part quality deteriorates then the parts are scrapped and the build is repeated with a higher ratio of new material to recycled material. The typical refresh rates recommended by these suppliers are shown in Fig 3 and Table 1. In addition, the current powder recycling practices are not able to determine how deteriorated the recycled powder has become in service and also, the operator of the LS machine is unable to exactly determine the quality of the material used in different builds. When a full system refresh is ordered then this raises the production cost enormously due to the high portion of the material used within the manufacture of the product and creates huge amount of scrapped PA12 powder with consequent environmental problems.

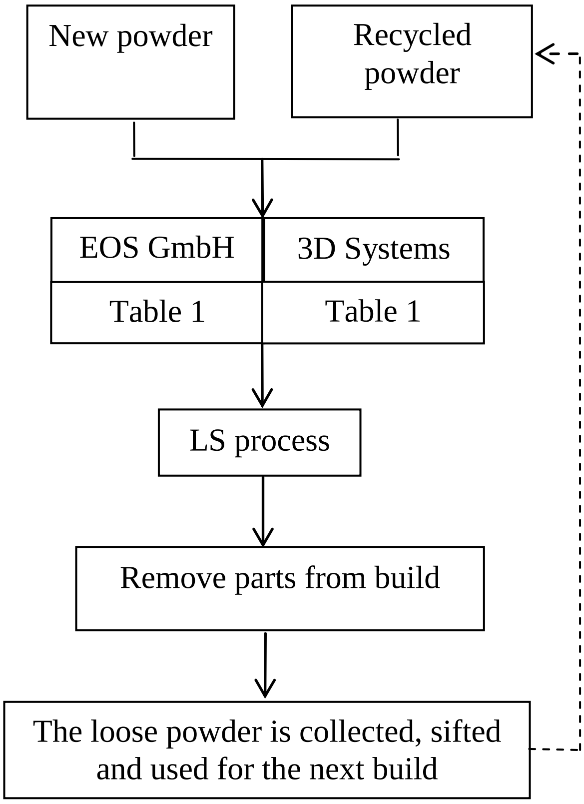


Fig. 3 The current LS recycling practise

Table 1 Recommended refresh rates

Manufacturer / Material Name	Refresh rate new powder, %	Additional recommendations
EOS GmbH		
PA2200 fine polyamide	30% to 50%	Scrap the powder if there is severe "orange peel" texture
PA3200 GF polyamide	50% to 70%	
3D Systems Corp.		
Duraform^{TM} (polyamide)	30% + (30% overflow)	Scrap the powder if there is severe "orange peel" texture.

2.0 Experimentation

The objective of this experiment is to investigate the significance of the deterioration level of PA12 powder on part surface quality through using the LS process. The deterioration level is determined by the number of cycles the powder has undergone in the system. The goal is to find a threshold or level of acceptable powder quality which would guarantee a relatively good surface finish with the absence of "orange peel". The influence of different powder quality to melt viscosity, part surface and part shrinkage is investigated.

2.1 Methodology and equipment used

Figure 4 shows a standard experimental methodology applied in this instance. The first step is powder preparation where the different qualities of recycled PA2200 powder material were utalised. A Melt Flow Rate (MFR) test is then conducted to determine the powder quality. A high MFR means that the material is likely to be virgin material and will have better thermal and processing properties. Through experience, the part affected by "orange peel" texture is associated with shrinkage problem. For this reason, the score system is used to evaluate the physical surface of benchmark part produced and the shrinkage measurement is conducted. The SEM is employed to examine and to compare the microstructures of the benchmark parts.

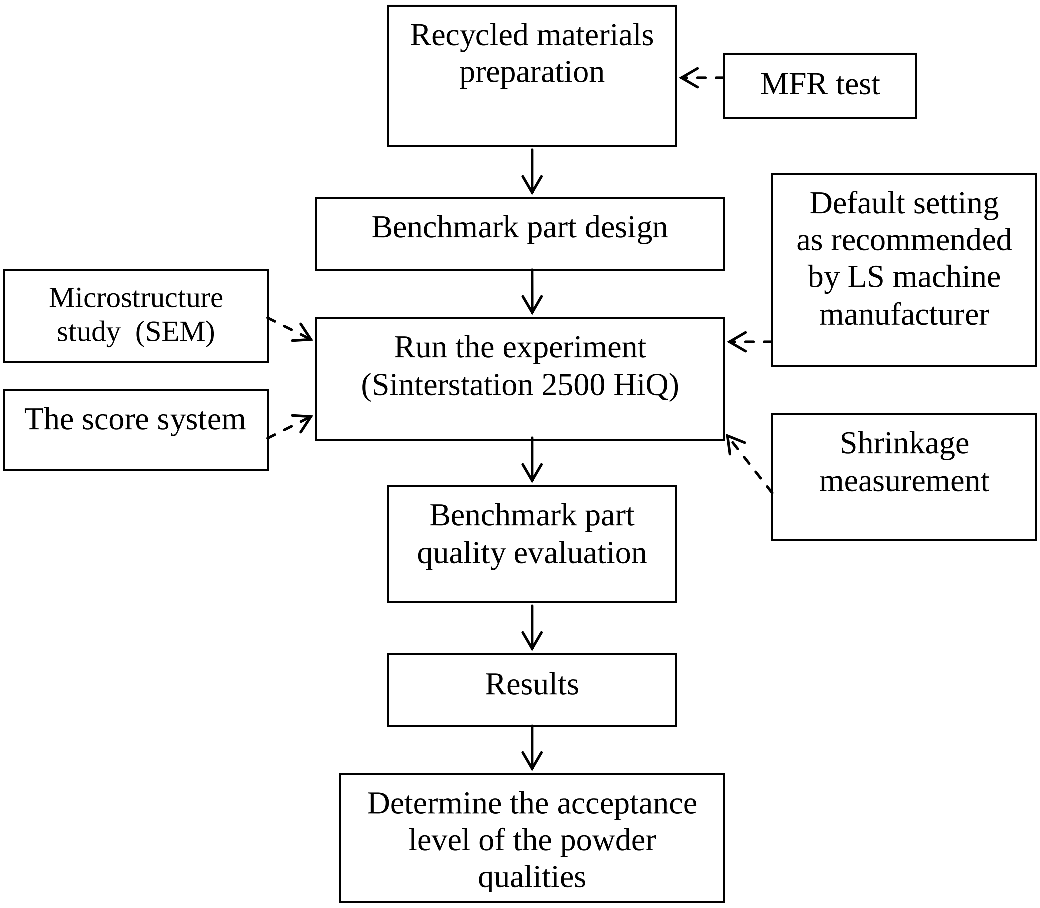


Fig.4 Experimental approach to determine acceptance level of the powder qualities

2.1.1 Recycled materials preparation

The LS material investigated in this study is PA12 based powder PA2200 supplied by EOS GmbH. The description of the different recycled PA2200 powder grades which based on melt flow rate (MFR) used to in these experiments is given in Table 2. The MFR was selected as a criterion for the powder "age" evaluation.

2.1.3 Benchmark part design

The benchmark part was fabricated several times using the same default LS parameters but different grades of PA2200 powder. The design and special features incorporated in this model are shown in Fig 5a,b and Table 3. 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).

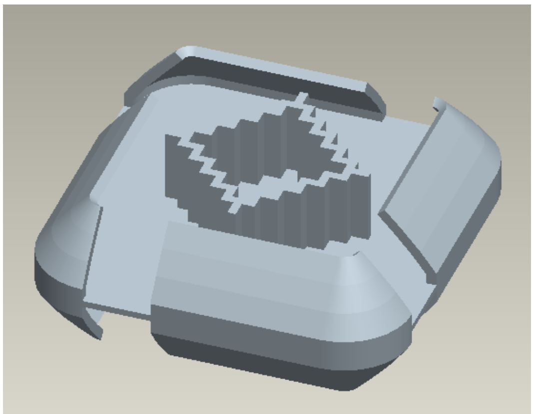


Fig 5a top view

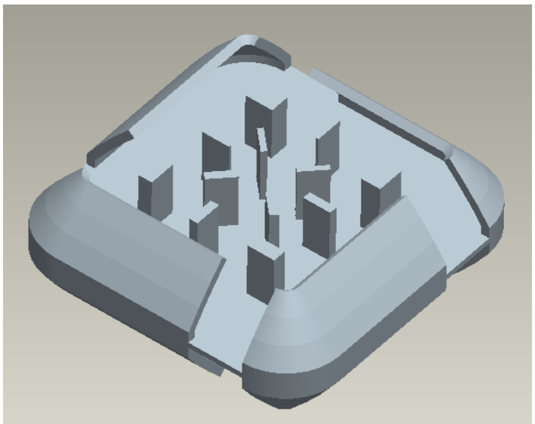


Fig. 5b bottom view

2.1.2 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 [8]-[9]-[10].

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 [4-5]. 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. 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 [11-12]. 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.

Table 2 Recycled PA12 powder grades

Recycled PA2200 Powder qualities	Description of Recycled PA2200
1	3 times recycled
2	2 times recycled
3	20% once used powder mixed with 80% 2 times recycled powder
4	40% once used powder mixed with 60% 2 times recycled powder
5	48% 2 times recycled powder mixed with 52% fresh powder
6	50 % Once used powder mixed with 2 timed recycled
7	Once used powder mixed with 65% fresh powder

Table 3 Benchmark part features

Features	Description	Function
"Pyramid"	Located on top of benchmark part sample	Shrinkage measurement
Angled surfaces	Different angles 50º, 54º,57ºand 90º	To study the effect of "orange peel" texture at different angles of surface
Vertical plain surfaces	Located at the bottom of bench part. Different size of 1mm, 2mm, 3mm, 5mm and 7mm	To study the effect of "orange peel" texture at different thicknesses and orientations
Conical surfaces	Different angles 50º, 54º,57º and 90º	To study the effect of "orange peel" texture on cone-shape surfaces

2.1.4 The score system

As shown in Fig.3, the score system is introduced in order to evaluate the benchmark part surface finish. Each part is individually inspected. The evaluation of the part quality from the point of view of the “orange peel” occurrence was done in accordance with a scoring system as described in Table 4

Table 4 Scoring system for evaluation of the part surface quality

Description	Score	Quality
Good surface finish, NO “orange peel”	1	Acceptable
Slightly rougher surface finish, NO “orange peel”	0.5	Acceptable
Small signs of “orange peel”	0	Not Acceptable
“orange peel” texture	0	Not Acceptable

The benchmark part surface quality is calculated based on the total number of scores divided by 96, which is the total number of surfaces and the accepted score of benchmark part surface quality is 70%.

2.1 5 Scanning Electron Microscope examinations (SEM).

The objective of this experiment is to investigate the external and internal microstructure (cross sectional) of good parts and parts affected by an “orange peel” texture. To obtain access into internal microstructure, the sintered
part was cut by breaking it. For all examinations, a thin layer of gold was sputtered on substrates using an auto sputter apparatus. Two pieces of equipment have been used consecutively. The first is a Bio-Rad SC500 for gold coating of the specimens and the second is an EMSCOPE SC500 for image capture. It is employed to characterise the individual powders and to analyse the surface morphology and microstructure of the sintered bench part. All LS fabricated bench parts were examined under high vacuum conditions. A low voltage (10kV) was chosen to minimise heat damage to the sample [13]

3.0 Results

3.1 Determination of acceptance level of the powder qualities to produce good finish part surface

This section presents a series of experiments is to find the refresh rate for the two different recycled powder blending at different ratios.

Table 5 Different mixed recycled powder quality

Description of Recycled PA2200	Powder grades MFR (g/10min)
3 times recycled	13
2 times recycled	17
20% once used powder mixed with 80% 2 times recycled powder	20
40% once used powder mixed with 60% 2 times recycled powder	23
48% 2 times recycled powder mixed with 52% fresh powder	25
50 % Once used powder mixed with 2 timed recycled	27
Once used powder mixed with 65% fresh powder	33

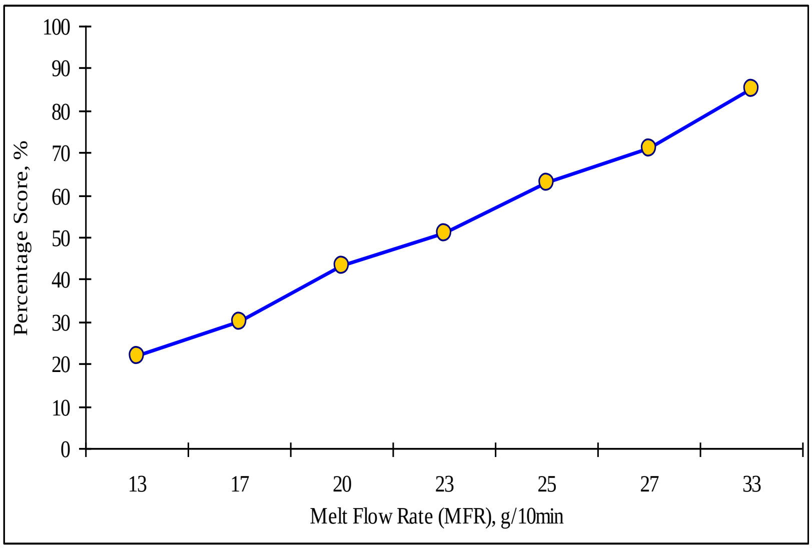


Fig. 6 Percentage score and MFR

As shown in Fig.6 suggest that the higher no of recycled times increased the melt viscosity due to badly deteriorated. However, it's the melt viscosity become improved when the less deteriorated powder was blended. It can be seen how the percentage score increases progressively with the increase the MFR value. It has been found that the parts build with the "2-3 times used PA12" (17 MFR) had worst surface finish and "orange peel" texture. After blending this material with 20 percent once used PA2200 powder the MFR was raised to 20. However, it was still not sufficient ratio to overcome the problem of poor surface quality. Even though another 20 percent once used PA2200 powder was blended to produce 23MFR powder quality, the rough surface, and "orange peel" texture were still affecting the thicker part features. After blending with 48% new PA in total and bringing the MFR to 25 the signs of "orange peel" disappeared. However, several part surfaces were observed rougher then normal. Only after using once PA powder with 27MFR or higher the part surface quality improved much more than before and was considered acceptable. These results suggest that a PA2200 blend having 27MFR could be used as a reference point for LS fabrication of parts with good quality. This due to the lower entanglement with a shorter molecule chain causes a low resistance to flow.

3.1.2 The relationship between MFR and recycled PA2200 powder quality

In Table 6, the result shows that by adding the higher amount of better quality PA2200 increases gradually the MFR value. This means that the powder quality and melt viscosity are correlated. This could be due to the less deteriorated PA2200 powder has lower entanglement with a shorter molecule chain causes a low resistance to flow (higher MFR value). This could produce a better powder melting and fusion during the sintering process which results in a good surface finish.

Table 6 Quality of different recycled based on MFR

Description of Recycled PA2200	Powder grades MFR (g/10min)
3 times recycled	13
2 times recycled	17
20% once used powder mixed with 80% 2 times recycled powder	20
40% once used powder mixed with 60% 2 times recycled powder	23
48% 2 times recycled powder mixed with 52% fresh powder	25
100 % Once used powder	27
Once used powder mixed with 65% fresh powder	33

3.1.3 The influence of MFR on part shrinkage

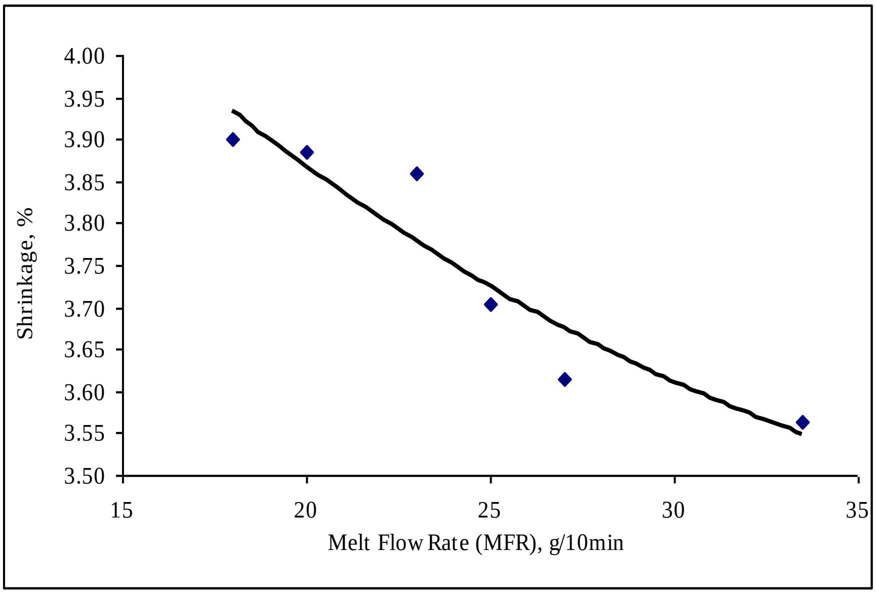


Fig. 7 The shrinkage and MFR

Fig.7 depicts the average shrinkage of LS parts build from PA2200 powder with different grade and MFR. This could be due to the higher melt viscosity causes more efficient packing of the PA2200 polymer chains which leads to increase in the LS part shrinkage.

3.1.4 The influence of PA12 powder quality to the microstructures of external surface and cross sectional parts

The objectives of these experiments were to find out the relationship between the PA12 powders MFR and, the microstructures of external surface and cross sectional parts and also to determine the minimum MFR required to maintain acceptable and consistent part quality.

Three different batches of PA12 powder were used to build the same design benchmark employed in Fig. 5a and 5b on the Sinterstation 2500 HiQ LS machine. The MFRs of these batches were 27 g/10min., 25 g/10min., and 17 g/10min. The part quality and surface finishing and its microstructure of the benchmark parts were evaluated and the results are shown in Fig.8a to 8f.

As shown in Fig.8a, the part surface of employing 17MFR powder grade was found rough texture, coarse and uneven surface finishing everywhere, severe “orange peel” and very bad quality. The microstructure of the sintered twice-used PA2200 powder is shown in Fig 8b. It can clearly be seen that there are a quantity of unmolten particles creating cavities in the sintered region. The high melt viscosity could cause the variations of the viscous flow may be expected, which lead to the formation of unmolten particle cores as clearly observed in the microstructure of the “orange peel” texture. As shown in Fig.8c, employing of 25MFR powder causes some vertical surfaces have “orange peel” texture, uneven surface finishing, however the part surfaces better than the previous part (Fig.8a), but it is still consider as unacceptable quality. It can be seen that the overall voids are smaller than those in Fig.8b. 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. In this experiment, the accepted part surface quality was established when the 27MFR powder was employed. It can be seen that no indication the presence of ‘orange peel’ texture, surfaces is smooth, even, acceptable quality (Fig.8e). The cross-section microstructure of good part which still have some small cavities with larger fully sintered region compare to image shown in Fig.7f. This because of less deteriorated powder was employed thus improves the sintering mechanism which causes the particles easily melted.

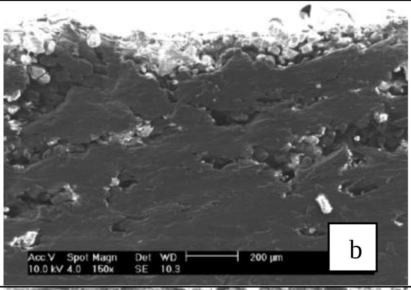
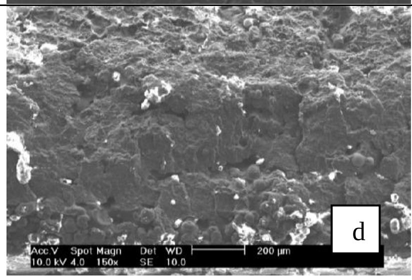
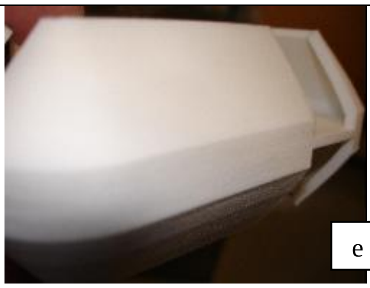
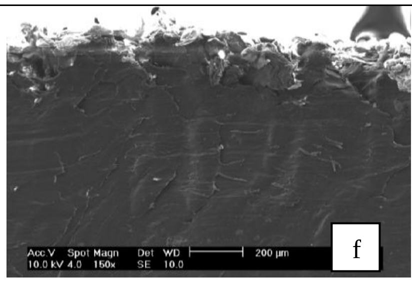
Recycled PA12 powder quailty	External surface	Cross sectional surface (150 X magnifications)
Type 2 (17MFR)		
Type 3 (25MFR)		
Type 5 (27MFR)		

Fig.8 The influence of MFR to the part surface finish and its microstructure

4.0 Conclusions

This paper suggests a methodology for identifying the optimum ratio between Virgin and Recycled PA12 material that can be used in the LS process that achieves consistent and good quality for the LS fabricated parts. The LS part quality is significantly influenced by the PA12 powder properties. It has been found that the powder with a higher melt viscosity (lower MFR) produces a poor "orange peel" texture due to poor sintering mechanism which cause inhomogeneous of external and internal sintered part microstructures.

The experimental results shown that the "orange peel" can be eliminated when a greater quantity of virgin material is mixed into the recycled material mix where the MFR is subsequently raised beyond a critical level. .An acceptable level of PA12 powder quality which would guarantee a relatively good surface finish and absence of "orange peel" is a powder blend with an MFR higher than 27g/10min. This could be used as a reference point when mixing and blending used and new PA12 material in the LS process. In this way the user would be able to control the input powder quality and therefore the final quality of the sintered parts. This result can be used as a basis for the development of a strategy for systematic recycling and a set of rules for more efficient powder management. The eventually benefit of this research is the manufacturing cost of LS parts can be reduced and the usage of new material can be optimised according to the properties of the recycled powder. The results of this research also contribute in energy saving thereby making a contribution to the environmental outcome.

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