Effecting of the Glass Filled Powder on Properties and Part Quality Produced by LS Processing

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Abstract—Polyamide (PA) using in selective laser sintering SL process, where Polyamide Duraform PA and Duraform GF polyamide of the 3D System Company and PA 2200 and PA 3200 GF of EOS Company are suitable for functional parts where GF composite material consist of polymer and glass filled. In these investigated aimed two methods was used, to find systematic approach to improving the quality of powder and quality of parts aimed to minimize the orange peel texture. LS of new powder (PA2200) EOS with a different proportion, volume of glass Beads (0 - 23%) was used. The microstructure of the mixture powders was analysed. The designed parts were built flat, in order to determine shrinkage values during sintering and curling and comparison between the normal pyramid which produced by PA3200 EOS parts. The highest density can be achieved at the glass bead ratio 50% and laser power of 15 Watt, owing to the melted PA2200 fills the gaps between the solid particles the high density solid glass particles may contribute in improving the density of the sintered part. Part quality can be affected by the change in parameters processing and orientation. The approach in this work described the surface quality affected by the continuing increase glass beads values, the best values of R_s singed with the lowest glass bead ratio 30% by weight and the worst was 3200 EOS powder. And the colour of the parts tended to darken when the value of glass beads increasing.

Index Terms: Selective Laser Sintering, polyamide, PA3200 Powders, Melt Flow rate and Viscosity.

I. INTRODUCTION

LS process is a rapid prototyping technology in which a 3D object is created layer by layer from heat powder materials with heat generated from a CO₂ laser, which was equipped with a nominal diameter of 0.45 mm, was continually focused on the powder bed. Polymer systems are widely used due to their unique attributes including ease of production, lightweight, and often-ductile nature. The polymer can be improved by the inclusion of fillers (fibres, whiskers, platelets, or particles) to from polymer matrix composites [processing and properties of glass beads] Two types of polymers have been extensively used in the LS process to build parts: amorphous polymers such as polycarbonate and crystalline polymer such as nylon/Polyamide, the later type of polymer has a latent heat and a melting temperature, while the former has only a glass transition temperature [1]. Polyamide (PA) using in SL process, where Polyamide Duraform PA and Duraform GF polyamide of the 3D System Company and PA 2200 and PA 3200 GF of EOS Company are suitable for functional parts where GF composite material consist of polymer and glass filled [2]. Composite materials can be defined generally as a macroscopic combination of two or more distinct materials, having a recognizable interface between them and mainly characterized by very high specific stiffness or rigidity, combined with an excellent resistance to the environment. The most commonly used reinforcement is glass Beads and carbon fibres [3]. Processing of polymer composites by SLS has been reported previously by Forderhase et.al. They investigated processing of Nylon-based composites with different volume fractions of glass fibre and glass bead reinforcements. Functionally graded materials (FGMs) by SLS has been reported by Jepson et al. created one dimensional FGMs by SLS processing of blends of tungsten carbide and cobalt powders. In this work FGMs were created on the plane of each layer by carefully placing different powder blends contiguously and scanning the entire layer using a laser. Beal et al. reported the SLS processing of one dimensional FGMs using blends of H-13 tool steel and copper powders and properties of glass bead particular filled SLS. In these investigated two methods was used, to find systematic approach to improving the quality of powder and quality of parts aimed to minimize the orange peel texture. LS of new powder (PA2200) EOS with a different proportion, volume of glass Beads (0 -23%) was used. The microstructure of the mixture powders was analysed. The designed parts were built flat, in order to determine shrinkage values during sintering and curling and comparison between the normal pyramid which produced by PA3200 EOS parts.

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II. LS PROCESS

In the LS process, there are processes parameters can be named in three categories: preheating, fusion, cooling rate control. The first two sets of parameters control part density, growth, and in-build curl; the last settings controls growth and post-build curl. The feed and part heaters control preheating. Before delivering powder to the part bed with the roller, the feed powder must heat to 10°C − 15°C below the melting point of the material at which the powder still flows freely and the part heater heats the powder to a temperature just a few degrees its melting point. Fusion occurs when just enough laser energy putting into the part to melt the powder. Cooling rate the parts position in the builds, and warm up height control the cooling rate. In general, the LS process involves three stages of the polymer: warm-up stage stabilizes the temperature in the process chamber, bed part, and feed beds. During this stage, the system slowly increases the part bed powder temperature to 10°C to 15°C below its melting point; the build stage maintains the part bed temperature and uses relatively low laser power to melt the powder in the part boundary which laser power, powder will be heated rapidly to a higher temperature above part bed temperature, the polymer will behave as a highly viscous liquid. Above T_m the polymer is in a liquid state and can flow due to this liquid flow, after laser scan over, the temperature will drop from T_m the viscosity increases and diffusion rate decreases, allowing the chains to rearrange themselves to form a nucleus and to from the whole part layer by layer [4]; the cooling stage allow the powder and the parts to cool slowly. During this stage, the system maintains the inert atmosphere to prevent oxidation (yellowing) of the powder and parts [5].

III. METHODOLOGY TESTS

In these investigated two methods were used, to find systematic approach to improving the quality of powder and quality of parts aimed to minimize the orange peel texture.

This experimental work in this section based on the fabrication of functionally graded materials FGMs by LS of new powder (PA2200) EOS with a different proportion volume of glass Beads (0 -23%). The different ratio of (PA2200 and glass beads) powders, the powder was weighed to the desired composition and blended in a rotary oblique mixer for 15 minutes. Glass used as the structural material and PA2200 used as the binder system and parts were analysis. These composite powders mixed to produce PA (glass filled powder) and parts were produced from these compositions. These are summarized in table2 and explained later. PA2200 powder was supplied from EOS GmbH and table 1 showed the Glass beads composition and has softening point 740 °C. HiQ machine was used to produce the test parts, which PA2200+10%glass beads was already configured with the parameters for polyamide to improve the quality of parts and intended to provide a broader range of data.

<table>
<thead>
<tr>
<th>Elements</th>
<th>SiO₂</th>
<th>Na₂O</th>
<th>CaO</th>
<th>MgO</th>
<th>Al₂O₃</th>
<th>FeO/Fe₃O₄</th>
<th>K₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>72.5</td>
<td>13.7</td>
<td>9.8</td>
<td>3.3</td>
<td>0.4</td>
<td>0.2</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Table 1. Shows The Chemical Composition Of Glass Beads

A. Preparation Of Powder

As mentioned earlier in this section the powder was prepared in the MEC LAB for building the parts, by obtaining the glass beads, PA2200 and PA3200 (GF) EOS weights, densities in order to estimate the proportion of the glass beads and PA2200 EOS by volume and weight, the results of these calculations was volume fraction of PA2200 86% 13% glass beads, and by weight 70% for PA2200 and 30% for glass beads (70:30), melt flow rate of this PA3200 EOS powder was 78 g/10min. According volume and weight ratio (70:30) by weight the calculations have been done by using different ratios of glass beads and it can be summarized in the table 2 and the powder were prepared according to this table.

In this work, a procedure for fabricating FGMs through the pressureless sintering of metal and ceramic powders is described. First, the selection and preparation of the powders are discussed. Then, the preparation of specimens with square and circular cross sectional geometries is presented. The steps are similar to the process outline by Rabin and Heaps [28], however, the focus is on pressureless sintering and overcoming the fabrication problems that result from this method of consolidation.

<table>
<thead>
<tr>
<th>Polyamide</th>
<th>Glass Beads</th>
<th>Ratio (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PA2200</td>
<td>90</td>
<td>10</td>
</tr>
<tr>
<td>85</td>
<td>15</td>
<td>85:15</td>
</tr>
<tr>
<td>80</td>
<td>20</td>
<td>80:20</td>
</tr>
<tr>
<td>75</td>
<td>25</td>
<td>75:25</td>
</tr>
<tr>
<td>70</td>
<td>30</td>
<td>70:30</td>
</tr>
<tr>
<td>65</td>
<td>35</td>
<td>65:35</td>
</tr>
<tr>
<td>56</td>
<td>44</td>
<td>56:44</td>
</tr>
<tr>
<td>50</td>
<td>50</td>
<td>50:50</td>
</tr>
</tbody>
</table>

Table 2. Shows The Mixing Ratio Of PA3200 With Glass Beads
B. Microstructure

The microstructure of the mixture powders were analysed by using an optical microscope with menmit software, samples of 10X10mm were analysed at room temperature, the image was taken in bright field to notify the uniformity of the powder during the mixing operation as shown in figure 4.6 and particle size distribution monitored.

C. Part Production

To investigate the influence of fabrication parameters and sintering between the powder particles PA2200 EOS and Glass beads for the powder that were mixed with the different ratio as mentioned in the preparation of powder section, the test parts were designed basis of the following goals; investigate and develop the materials processing techniques necessary to produce appearance of parts such as shape, X-Y plane dimensional accuracy of the parts and Z direction, surface quality measurements, density using above mentioned compositions default processing parameters on LS machine, figure 12 depicts the shape of three different specimens (pyramid test part, solid block 40X30X5mm and rectangle with circles and squares 114.3X25.4X5mm ) were built the parts by using the powder as in the table (3 in section 5.3). The parts were built flat, laid parallel to the machine x-direction in order to determine shrinkage values during sintering and curling and compare between the normal pyramid which produced by PA3200 EOS parts and test parts produced uniformity.

D. Testing Equipments

The experimental test equipments were used in this study LS machine HiQ machine, Meltflier MT thermohaake. With Haake software, oblique manual mixer, sand plaster, digital scale and optical microscope with Omnimet software.

D.1 Melt Flow Rate Indexer

In order to improve the quality of recycling powder PA3200 (RPGF) due to the effect of temperature and time after the LS process. New powder supplied by EOS Gmbh, once used, 1X recycled and 2X recycled powders (loose powder) were exam by using MFR and to determine the flow behaviour of the material. The results are indicated in g/10min by extruding molten material from the die under preset conditions of temperature and load. The MFR experiments were preformed according to ISO1133 standard a small of amount taken for testing 8-10 grams under testing conditions 5.0kg and at temperature 275 °C [10-12]. The weight and time recorded and the average of 6 MFR measurements then calculated.

Glass beads improved melt flow relative to other filler and up to 10 times’s compared to glass fibre reinforced resins. Glass beads increase stiffness while lower mould shrinkage and thermal expansion. Also enhance compressive strength, but have little effect on tensile strength [Melvin I. Kolan, 1995]. Solid fillers increase the viscosity in a manner consistent with the Einstein equation.

\[ \eta_c = (1 + 2.5\phi)\eta_0 \] .................................1

\[ \eta_c = (1 + 2.5\phi + 14.1\phi^2)\eta_0 \] .................................2

D.2 Sinterstation 2500HiQ Machine

The specifications of Sinterstation 2500 HiQ have CO₂ laser with the diameter 0.45 mm. The building chamber of the machine (320X280X457) mm, capacity volume of the 40950 cm³, the weight required for full load of the powder 19 Kg, this machine can be used PA and GF polyamide materials[7]. The whole process of powder preparation and loading into the LS machine is manual. A rotating drum cement Mixer is normally used for mixing. Automatic sifter with fine mesh used to sieve the powder after the LS process of using the powder once more.

D.3 Omninet Software And Optical Microscope

Particle size can have a fundamental effect on the physical properties of colloidal dispersions. For many systems, the measurement of average particle size is not sufficient; the presence of different size populations will have a strong influence on properties and could be related to the production process. The particle size distribution of the powders was measured using random area.The size of particles computed from image analysis of the two dimensional projection of the particles. The image analysis system consisted of an optical microscope fitted with camera for obtaining the particle image as a pixel representation and the high-resolution monitor screen to display the image. The software used to obtain the diameter (omninet software) magnifying (Ax5), the particle profile data were collected from at least 75 particles of each powder sample [13 manual]. This method produces information about the average particle size and the distribution of particle size samples with broad particle size distributions for (PA2200). Glass beads and Glass filled powder mixed with different ratios of glass beads.
IV. EXPERIMENTAL RESULTS AND DISCUSSION

A. Effects Of The Ratio Of Glass Beads On The Quality Of Powder

The selection of materials suitable for the fabrication by LS was based on the following criteria; material must be available in powder form for good spreading on the powder bed. Powder should flow freely, even at elevated temperature, to form each new layer in LS processing. In this experiment the effects of the volume fraction of Glass Beads on the quality of powder studied owing to it becomes necessity from economic points of view. Comparison of new PA3200 EOS powder with different mixed ratio of PA-glass filled powder produced in Manufacturing Engineering Center (MEC) LAB using a rotary cement mixer, melt flow rate and image analysis optical microscope to obtain the equivalent powder of PA3200 EOS. Uniformity of the powder owing to mixing was observed as showed in figure 7 for (50:50) mixed powder then compared with PA3200 EOS powder in figure 6. Figures 8 and 9 showed the particle size distributions of the PA2200 EOS and the glass beads obtained by image analysis before the mixing operation. The majority of the new PA2200 EOS powder particles were in the 2.5-66 μm range and the average 42 μm while the majority of glass beads were in the 34-67μm range and the average 53 μm. It can be noticed from the figures unsymmetrical histograms – skewed towards larger sizes this means medium are not equal the modal value.

Figure 1. Uniformity Of Particle After Mixing With Ratio PA2200 EOS And Glass Beads (56:44) By Weight Powder

Figure 2. Particle Size Distribution Of PA2200
B. The Effect Of The Content Of The Glass Beads On The Mer And The Viscosity

As mentioned earlier the experiments in this work based on the different mixing ratio, table 2 shows the results of this observation of mixing, Figure 10 showed the effect of glass beads on the MFR values. It was observed from these measurements, by continuing the increase of the glass beads to 50%, the value of MFR decrease due to quantity of melted PA2200 reduced and the gap between the solid glasses becomes narrow and brittle material. Figure 11a, it is also shown that melt viscosity increased with increasing the volume fraction of glass beads as showed in figure 5b.

C. Effects Of The Ratio Of Glass Beads On The Equality Of Parts And Their Density

The test Parts of the study were produced by an LS process by HiQ machine and as shown in Figures 6 and 7, built for various laser powers12, 15 Watt, scanning speed 4000 mm/s and scan spacing 0.15mm with ratio by weight (70:30), (50:50). Figure (7.c) showed the image of new PA3200 EOS powder volume fraction (70:30) and weight% (86:13) was built at laser power 15 Watt, distinct as the normal part has optimal conditions and good results. The parts were produced from the powder ratio mentioned above in Table 3 PA2200+13% glass beads (70:30) by weight and PA2200+23% glass beads (50:50) by weight distinct as test ports as shown in figure 7.a, b. Comparing the results data for normal pyramid part obtained from EOS powder with the results data for the test parts was produced by weight% (70:30) and (50:50). Figure 8 showed that the density of the normal pyramid part is higher than the density of test part (70:30) by 0.1 (g/cm3) and less than (50:50) by 0.13. Also the analysis as in density of part made from PA2200+23% glass beads (50:50) higher than that of...
PA2200+13% glass beads (70:30) part by 0.23 when laser powder 15 watts and slightly less in 12 Watt 0.225 g/cm³.

Table 3. Default Values For Processing Parameters For PA2200, PA3200EOS And That Used For Different PA2200EOS/ Glass Beads Compositions.

<table>
<thead>
<tr>
<th>Processing parameters</th>
<th>PA2200 EOS</th>
<th>PA3200 EOS</th>
<th>PA2200EOS 86%+13% glass beads</th>
<th>PA2200EOS 77%+23% glass beads</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting point</td>
<td>185</td>
<td>185</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Laser power (Watt)</td>
<td>15</td>
<td>15</td>
<td>12 &amp;15</td>
<td>12 &amp;15</td>
</tr>
<tr>
<td>Scan space (mm)</td>
<td>0.15</td>
<td>0.15</td>
<td>0.1</td>
<td>0.15</td>
</tr>
<tr>
<td>Scan speed (mm/s)</td>
<td>5080</td>
<td>5080</td>
<td>5080</td>
<td>5080</td>
</tr>
<tr>
<td>Layer thickness (mm)</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>Ambient temperature (°C)</td>
<td>174</td>
<td>174</td>
<td>174</td>
<td>174</td>
</tr>
</tbody>
</table>

Figure 6. Test Specimens Of LS 1-Pyramid Test 2- Block 3- Rectangle

Figure 7. Normal Test Specimen Of LS Test Parts a- (50%:50%) PA2200, b- (70%:30%) c- Normal Part PA3200 EOS.

Figure 8. Density Of Parts Against Ratio Of Mixing

D. Effects Of The Ratio Of Glass Beads On The Microstructure Of The Parts

Figures 9-14 show optical microscopic observations of how the internal structure of the parts changed as the laser power, energy density and ratio of glass beads increased. It confirms that the microstructure of the top surface of the PA3200 EOS specimen built on the laser power 15 Watt and energy density 0.025 J/mm².

In this case, the particles are fused slightly of the points of contact and voids between the glass particles can be identified the cross section of the specimen still similar to the top surface individual particles and voids exists along the depth of the specimen and the glass particles are still solid in the form of a sphere. The cross section images shows there are area have some polymer particles which unmolten have fused in the core of the parts, usually that occurring when the particles do not receive enough heat to viscose, melt and flow the powder. A microscopic image of the cross section and the top of the specimens at laser power 12 and 15 Watt are shown in Figure 9 and 10. The existence of voids between particles less apparent in all ratios, at laser power 15 than that 12 due to apparent of density of parts fabricated a as mentioned in figure 8 when the dense part increased as the laser power increasing the (50:50) % more compact structure than that ratio (70:30) %.

The microscope image of a PA2200 EOS mixed with a glass bead ratio (50:50) % and (70:30) % parts fabricated at 15 Watt laser power is shown in Figure 11,12,13 and 14. The top of the image shows the part unmolten particles fused to the edge of the part for all cases. The presence of voids at the surface in ratio (50:50) % similar apparent that exhibited in the cross sectional area and less apparent than that in ratio (70:30) % and normal PA3200 EOS. This is owing to the flow of powder, heating and cooling rate. Figure 13 and 14 show this and also shows a number of peaks and holes at the surface for both of ratio mixing, the presence of these peaks are believed to the lawyer and to be important to the properties and mechanical influences of LS processed powder. Also, it was signed at ratio 50:50) % 15
Watt at cross section and near of the part there was tear and holes.

A microscopic image of the cross section and the top of the specimens at laser power 12 and 15 Watt are shown in Figure 11, 12, 13, and 14. The existence of voids between particles less apparent in all ratios, at laser power 15 than that 12 due to apparent of density of parts fabricated a as mentioned in Figure 8 when the dense part increased as the laser power increasing the (50:50) % more compact structure than that ratio (70:30) %.

Figure 11. Shows The Laser Power 15 Watt And Energy Density 0.025 J/mm² Inside Of The LS Parts Using 50:50 Ratio Of Powder.

Figure 12. Shows The Laser Power 15 Watt And Energy Density 0.025 J/mm² At The Surface Of The LS Parts Using 50:50 Ratio Of Powder.

Figure 13. Shows The Laser Power 15 Watt And Energy Density 0.025 J/mm² Inside Of The LS Parts Using 70:30 Ratio Of Powder.

Figure 14. Shows The Laser Power 15 Watt And Energy Density 0.025 J/mm² At The Surface Of The LS Parts Using 70:30 Ratio Of Powder.

E. Effects Of The Ratio Of Glass Beads On The Surface Roughness

Surface finishing the evaluation of the surface finishing depends on the visual inspection of test parts. Some of the very poor surface can be classified by orange peel texture depends on the degree of texture, this effect is supposed to be reduced or completely eliminated by improvement of the quality of the powder and LS parameters or by secondary finishing operations on the parts such as sanding. The theoretical values of Ra and Rt roughness relative to angle inclination $\theta$ \[14\].

$$Ra = \frac{p \sin \theta}{4} \text{ and } Rt = p \sin \theta \ldots .3$$

Where ; $p$: layer thickness

In the present study Ra value were measured the better surface was at the lowest ratio of glass beads (70:30) and the worst was at the highest ratio of glass beads (56:44) owing to the changing of amount of the quantity of PA2200, two levels of finishing were considered, upper surfaces smoother than the bottom surfaces and the best values in horizontal surfaces Ra 3.2 $\mu$m in (70:30). it was signed that the amount of glass beads an important factor influences in the surface quality and effect of the reduction of surface quality of these parts and the colour of the parts tended to darker when the value of glass beads increasing.

$X-Y$ plane of test parts showed uniform linear dimensions and circular diameter and no curl and distortion occurrence. Parts dimension after building measured internal and external features X and Y directions in order to determine the accurately the scaling factors and the offset values compare with normal part and to track dimensional changed that can occur as the powder ratio change and variations.
powder quality. Test parts measuring is indicated, no significant shrinkage occur compared with normal part.

Table 4. Roughness Surface Of Different Powders

<table>
<thead>
<tr>
<th>Powder</th>
<th>Roughness surface $R_a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>PA2200 EOS</td>
<td>3.2 $\mu$m</td>
</tr>
<tr>
<td>PA3200 EOS</td>
<td>3.6 $\mu$m</td>
</tr>
<tr>
<td>PA (50:50)</td>
<td>3.6 $\mu$m</td>
</tr>
<tr>
<td>glass filled</td>
<td>3.6 $\mu$m</td>
</tr>
<tr>
<td>PA (70:30)</td>
<td>3.2 $\mu$m</td>
</tr>
<tr>
<td>glass filled</td>
<td>3.2 $\mu$m</td>
</tr>
</tbody>
</table>

V. CONCLUSION

Material properties play an important role in determining fabrication parameters, mechanical properties of LS parts and $T_g$ and $T_m$ to determine part bed temperature and laser powder also the relationship between them to obtain good quality parts.

The density of the sintered part is a function of the laser power and glass bead ratio; it increases when the laser powder increases and the glass beads ratio increases.

The highest density can be achieved at the glass bead ratio 50% and laser power of 15 Watt, owing to the melted PA2200 fills the gaps between the solid particles the high density solid glass particles may contribute in improving the density of the sintered part. For case dimensions and geometrical prototypes tolerances are important, the manufacture of the parts must be fitted with data of CAD during the preparation for the partial stage. The surfaces of parts produced from the LS process are investigated in order to determine the part quality. Part quality can be affected by the change in parameters processing and orientation. The approach in this work described the surface quality affected by the continuing increase glass beads values, the best values of $R_a$ sinned with the lowest glass bead ratio 30% by weight and the worst was 3200 EOS powder. And the colour of the parts tended to darker when the value of glass beads increasing.

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