EFFECT OF SINTERING PARAMETERS AND FLOW AGENT ON THE MECHANICAL PROPERTIES OF HIGH SPEED SINTERED ELASTOMER

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Abstract

High Speed Sintering (HSS) is an Additive Manufacturing process that creates parts by sintering using inkjet and infra-red lamp technology rather than laser systems employed in Laser Sintering (LS). This research investigated the effects of machine parameters (sintering power, bed temperature) and the addition of fumed silica flow agent on the tensile properties of thermoplastic elastomer parts processed using HSS. The results showed improved elongation at break values by a factor of more than 2X compared to reported values for LS of the same thermoplastic elastomers. At constant parameters, improved tensile strength and tensile modulus were observed with the addition of flow agent into the sintering mixture.

Keywords

High speed sintering, thermoplastic elastomer, tensile properties, sintering power, flow agent

Introduction

Additive Manufacturing (AM) is defined as "the process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing technologies" (1). There are currently over twenty recognised Rapid Prototyping (RP) processes based on the "additive" principle, though method of layers consolidation may differs from one another. Classification is made according to the physical state of raw material used in the process; liquid-based systems (eg: Stereolithography), solid-based systems (eg: Fused Deposition Modelling) and powder-based systems (eg: Laser Sintering, High Speed Sintering) (2). Depending on the type of process and hardware, a range of metal, polymer and ceramic materials can be used with AM to create end-use parts with minimal post-processing.

The main advantage of AM is the design freedom that comes with its tool-less and mould-free processes, enabling consumers to produce parts of almost any geometric shape or feature (3). Design customisation is also made easy and cost-effective with AM as it only takes place within the CAD design. This eliminates many restrictions of conventional design for manufacture and assembly (DFMA) such as the need for undercuts, draft angles, reduced part counts, etc and therefore improves the design process as a whole (2).

Laser Sintering (LS) is a popular additive process used for making polymer parts. It uses a high power CO_2 laser to sinter powder raw material on a surface bed to create desired shape. Nylon-based materials have been widely used with LS for many years in both RP and Rapid Manufacturing, particularly the nylon polyamide (Nylon-12) for its superior properties (4). Material suppliers such as 3D Systems and ALM specialise in materials development and has manufactured a range of polyamides (eg: filled and unfilled nylon) and elastomers (eg: Thermoplastic elastomer, TPE) to be used with LS (5).

High Speed Sintering (HSS) is a relatively new AM process invented at and patented by Loughborough University in the UK, with its process developments detailed by Hopkinson et al (6). It utilises inkjet print head and infra-red technology to manufacture products layer by layer from polymer powder material. The process incorporates the same technique as LS with the exclusion of laser disintegrated into the following; IR lamp acts as the heating source and inkjet print head defines the part geometry. HSS has several advantages over LS – reduced machine cost by laser elimination and reduced build time as it sinters layer by layer. In common with all AM processes, HSS is capable of producing complex geometry parts with superior mechanical properties.

Application of AM is widespread across aerospace, automotive and consumer industry. LS is used to manufacture air ducts for Boeing F-18 military jets. New Balance, a major footwear manufacturer has adopted LS in producing high-performance customised running shoes for elite athletes.

High Speed Sintering

Figure 1 shows the High Speed Sintering machine and its key parts; powder bed heater, infrared lamp, roller system, inkjet printhead and powder bed.

The build procedure on the HSS machine can be broken down into three main stages; pre-processing, building and post-processing. The pre-processing involved modelling of 3D part on a CAD software (Solidworks) then subsequently sliced into a stack of 2D layer files or slice files (SLC), each of 100μm thickness in preparation for the HSS machine. The layer files were transferred to the machine where build parameters were set.

Once the prepared powder was fed into the feed chamber and build parameters were set, the building process began with deposition of a layer of raw powder onto the part bed by roller action, with an IR preheat stroke to warm up the powder prior to sintering. Monochromatic bitmap images of desired part were printed onto the powder bed using a Radiation Absorbent Material (RAM), which upon heating by IR lamp will absorb thermal energy thus melted and sintered the powder underneath. Unprinted powder will remain unsintered and acted as a support to the build. New layer of raw powder will be redeposit and this process was repeated until all layers have finished building. Blank layers may be included in the build to separate different parts.

The parts were left to cool with all heating parts switched off, before the cake was removed from the machine. Post-processing involved powder removal through bead blasting process to obtain finished parts. Other properties enhancement technique such as infiltration of parts may follow if needed.

This research highlights the effects of three main build parameters on the mechanical properties of finished parts, using a TPE material. Previous researches concerning the HSS process include the study of excess powder hardness using Nylon with HSS (6) and the effect of Infra-red power level on sintering behaviour of Nylon-12 (7).

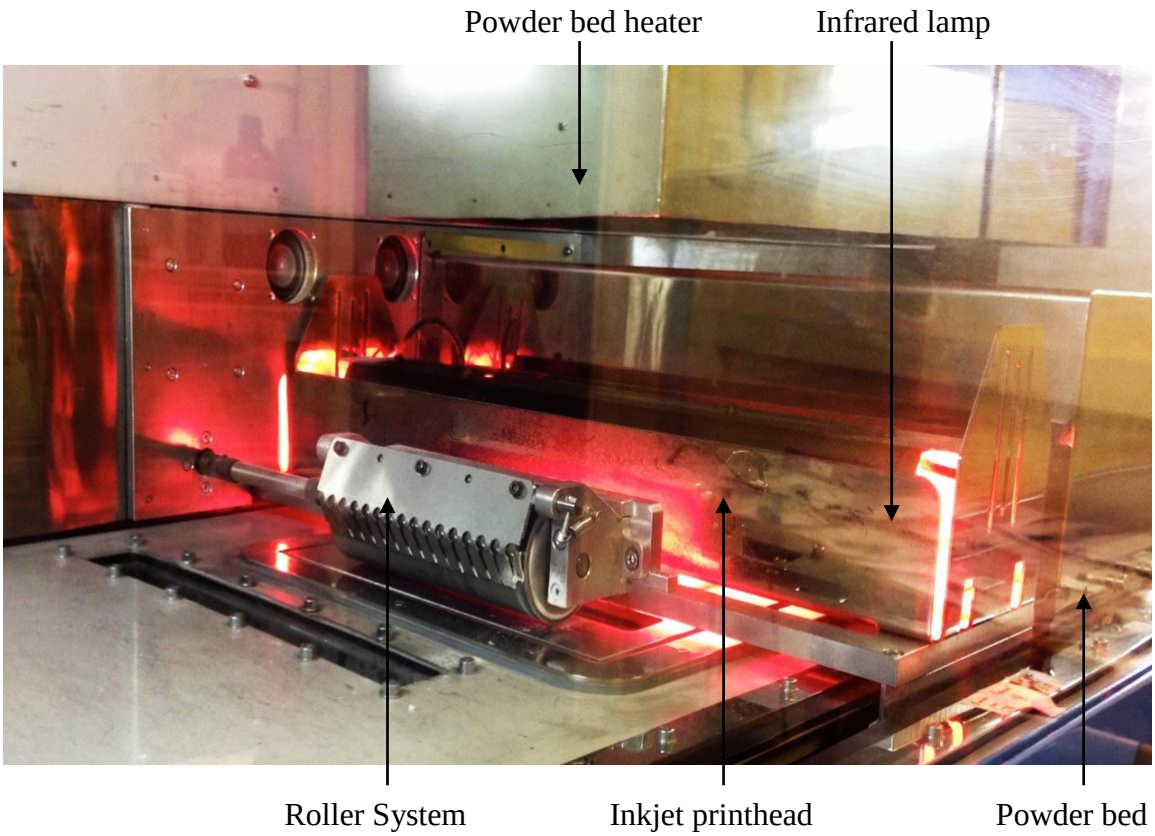

Figure 1 – High Speed Sintering machine and key parts

According to Kang (8), sinterability and the sintered microstructure of a powder compact are mainly determined by material variables and process variables. A set of key HSS build parameters are divided into the two groups and defined in Table 1:

Material variables	Process variables
• Layer Thickness – The thickness of each powder layer is determined by the lowering of build platform.	• Build Bed Jacket & Overhead Temperatures – The base and overhead temperatures of bed determines the powder temperature at sintering.
• Powder Ratio – The composition of powder material which may include additives or fillers.	• Sintering Power – The sintering rate is determined by the sintering speed.
	• Surface Preheat – The rate at which powder is preheated before sintering.

Table 1 – Sintering parameters

Elastomer

Thermoplastic elastomer (TPE) is elastomeric with the properties of thermoset rubber. It softens when heated to a flowable state but does not cure or set under heat as does thermoset hence its chemical properties were unchanged thus promoting recyclability (9). This study will focus on the manufacturing and testing of TPE210-S, a flexible elastomer sintering material supplied by ALM. The ASTM definition of elastomer is “a material which at room temperature can be stretched repeatedly to at least twice its original length, and, upon

release of the stress, will return immediately to its approximate original length" (10). The general mechanical properties for TPEs include low modulus and high yield strain. Table 2 below listed the main properties of TPE210-S as provided by the manufacturer, ALM when processed using LS.

Properties	**Value**
Average Particle Size (D50)	85 μm
Melting Point	178 °C
Tensile Modulus	8 MPa
Elongation at Break (uninfiltrated)	110 %
Shore Hardness (uninfiltrated)	40

Table 2 – TPE210-S Material Properties

TPE has existed for 40 years and was favourable to conventional rubber for its ease of processibility and flexibility. Injection moulding is the principle process used in the fabrication of TPE parts, followed by extrusion process. Injection moulded TPEs are advantageous of costs at rapid production rates. However in comparison, LS is more economical for low volume part production (less than 14000 unit) (2). TPEs have replaced thermoset rubbers in most of its former applications. TPE parts are widely used in automotive applications for both exterior (bumpers) and interior (dashboard) parts, including under-the-hood automotive applications such as front-wheel drive components and air ducts (9).

Methodology

1. Manufacture of test parts

To investigate the influence of build parameters, test specimens were manufactured with variation in build bed overhead temperature, powder ratio and sintering power. A Design of Experiment was created as followed. Table 3 lists the complete build parameters used throughout the project. Table 4, Table 5 and Table 6 were build parameters with variations in sintering power (speed), build temperature and powder composition (presence of flow agent), respectively. Figure 2 shows the tensile piece used throughout the experiments.

Parameter	**Value**
Build bed jacket temperature	35 °C
Build bed overhead temperature	90/93/94/100/110 °C
Feed bed jacket temperature	30 °C
Feed bed overhead temperature	35 °C
Layer thickness	0.1 mm
Maintenance layers	1
Orientation	XYZ
Powder temperature	Room temperature
Powder ratio	100% Virgin/ 100% Recycled/ 50% Virgin 50% Recycled/ Virgin+0.2% Cab-o-sil/ Virgin+0.5% Cab-o-sil/ Virgin+1.0% Cab-o-sil
Sintering power @ speed	100% @ 50/55/60/70/80 mm/s
Surface preheat	50% @ 100 mm/s
Warm up time	30 minutes

Table 3 – HSS build parameters for manufacture of test specimens

Overall length :	115 mm
Gage length :	25 mm
Overall width :	19 mm
Neck width :	6 mm
Depth :	4 mm

Figure 2 – ASTM Type IV Tensile Test sample (left) and dimensions (right) processed by HSS

- Investigating the effect of sintering speed at two sets of constant temperature and powder composition parameter

Build	Powder ratio	Build bed overhead temperature (°C)	Sintering speed (mm/s)
1	50% Virgin 50% Recycled	90	60
			70
			80
2	50% Virgin 50% Recycled	100	50
			60
			70

Table 4 – Variation in sintering speed

- Investigating the effect of bed temperature at two sets of constant sintering speed and powder composition parameter

Build	Powder ratio	Sintering speed (mm/s)	Build bed overhead temperature (°C)
1	50% Virgin 50% Recycled	70	90
			93
			100
			110
2	50% Virgin 50% Recycled	60	90
			100

Table 5 – Variation in temperature

- Investigating the effect of flow agent addition at two sets of constant sintering speed and temperature parameter

Build	Build bed overhead temperature (°C)	Sintering speed (mm/s)	Flow agent level (% by weight to virgin TPE)
1	100	50	0
			0.2
			0.5
2	110	70	0
			0.2
			0.5
			1.0

Table 6 – Variation in flow agent level

2. Tensile testing

Tensile tests were carried out according to ASTM D638 Standard Test Method for Tensile Properties of Plastics (11) using a Tinius Olsen H5K-S Universal Testing Machine equipped with model 500LC Extensometer and HT36 grip under ambient conditions. The crosshead speeds of 5mm/min and 10mm/min were maintained during the tests. The method provided calculations of the elongation at break (%), young's modulus (MPa) and ultimate tensile strength (MPa). Figure 3 shows a typical stress-strain curve obtained by the tensile test.

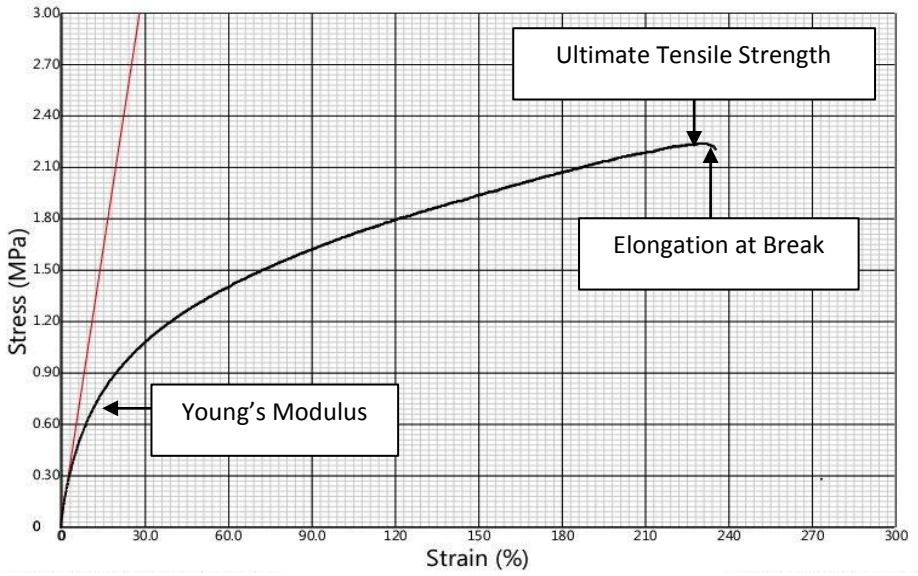

Figure 3 – EaB, YM and UTS determined from ASTM D638 Tensile Test

3. Thermal analysis

The thermal analysis of raw powder material was carried out as outlined by ASTM D3418 Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry (11). The tests were performed using a double furnace PerkinElmer DSC8500 instrument. The powder sample tested were 100% Virgin TPE210-S (0.0113g) and Virgin TPE210-S mixed with 0.2% Cab-O-Sil (0.0167g). The standard reference material was an empty pan.

The samples were heated from 20°C to 210°C at the rate of 20°C/min. The temperature was held isothermally at 210°C for 1 min. Then it was cooled back to 20°C at the rate of 20°C/min. The temperature was held isothermally at 20°C for 1 min. The cycle of heating, temperature hold and cooling was repeated at the same rate . The DSC curves were plotted for all samples.

4. Surface roughness testing

Surface roughness testing was done using a Mitutoyo Surftest contact profilometer rig to quantify the quality of high speed sintered TPE parts surface. Constant speed of 0.5mm/s was maintained and evaluated along 20.0 mm length of the type IV ASTM test specimen during the test. The test was performed on both top and bottom surfaces of specimens. Values of roughness parameter; average roughness, Ra and average maximum height, Rz were obtained through this method.

Results and Discussion

1. Mechanical Properties

a) The effect of sintering speed at two sets of constant temperature and powder composition parameter.

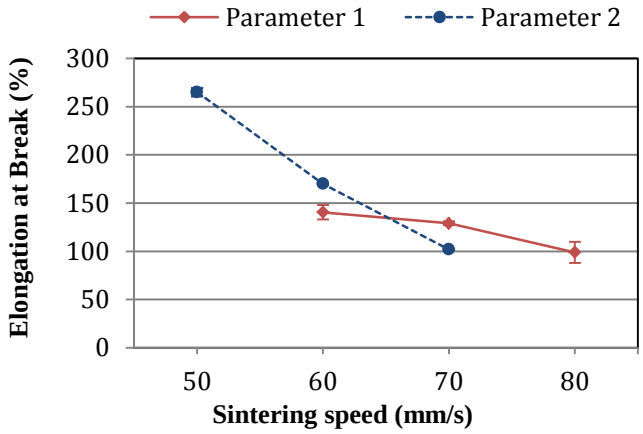

Figure 4 – Variation of EaB with sintering speed

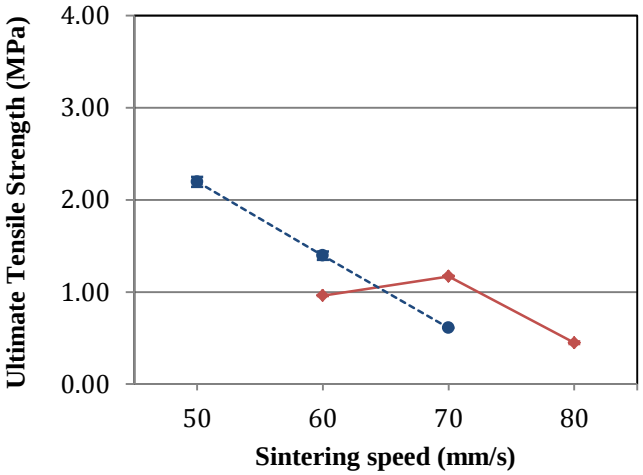

Figure 5 – Variation of UTS with sintering speed

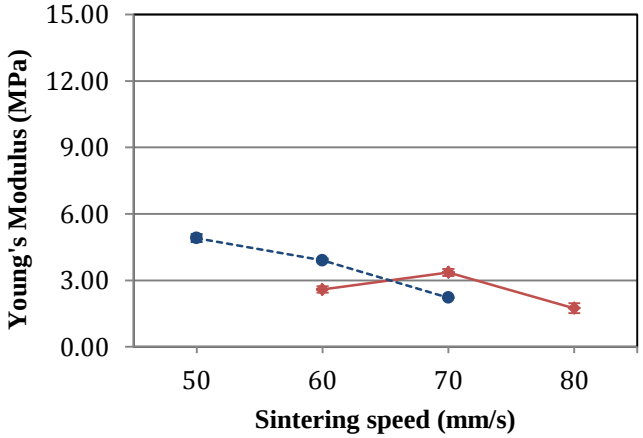

Figure 6 – Variation of YM with sintering speed

Figure 4, Figure 5 and Figure 6 show the graphs of mechanical properties EaB, UTS and YM plotted against increasing sintering speed at two constant bed temperature 90°C (Parameter 1) and 100°C (Parameter 2), respectively.

The maximum value of EaB, UTS and YM are observed at the lowest sintering speed of 50mm/s, which are 265%, 2.20 MPa and 4.91 MPa respectively

From these figures, it can be concluded that EaB increases with decreasing sintering speed. UTS increases with decreasing sintering speed, and at a higher rate at low temperature compared to high temperature. The value of YM decreases with increasing sintering speed at both set of temperatures. The variation is not linear and remains low across all set of build parameter.

Though at different intensities, it can be observed that the mechanical properties of HSS printed TPE increase with decreasing speed; highest properties achieved at lowest speed and conversely true. The influence of this parameter on mechanical properties can be related to energy density and sintering behaviour. According to Andrew Number, an established model formulated for LS energy density calculation, a decrease in sintering speed results in an increase in energy density and directly mechanical properties.

b) The effect of bed temperature at two sets of constant sintering speed and powder composition parameter.

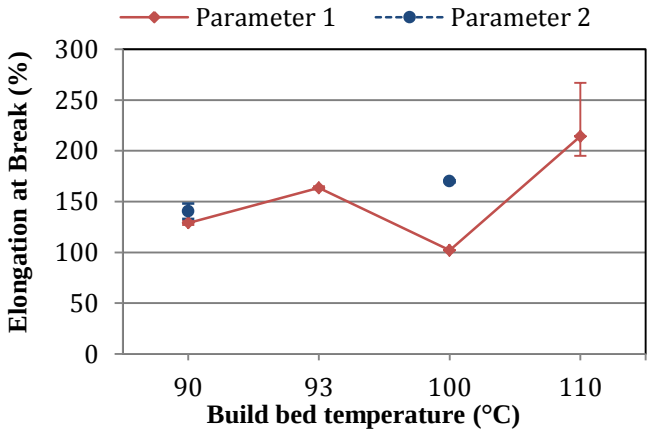

Figure 7 – Variation of EaB with bed temperature

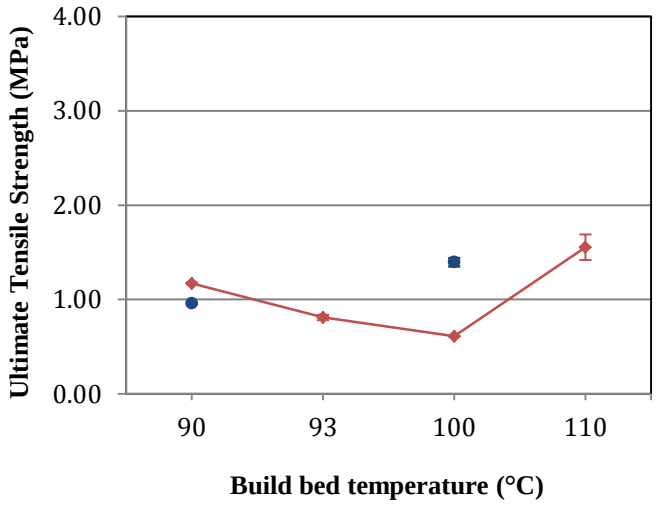

Figure 8 – Variation of UTS with bed temperature

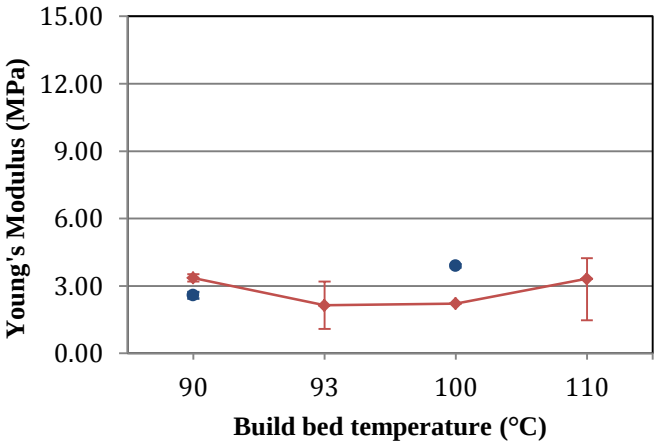

Figure 9 – Variation of YM with bed temperature

Figure 7, Figure 8 and Figure 9 show the graphs of mechanical properties EaB, UTS and YM plotted against increasing build bed temperature at two constant sintering speed 70 mm/s (Parameter 1) and 60 mm/s (Parameter 2), respectively.

The maximum value of EaB, UTS and YM are observed at the highest build bed temperature of 110°C, which are 214%, 1.55 MPa and 3.32 MPa respectively.

A slight fluctuation can be observed in values of EaB from 90-100°C, which can be regarded as constant before a rapid increase at 110°C. The same variation exist in tensile properties; an almost horizontal gradient that shows little effect of build bed temperature on UTS and YM until reaching 110°C.

Build bed temperature largely determines the powder temperature which are set across the bed surface. According to DSC in Figure 13, the temperature range of 100 – 110 °C is where glass transition occurs for TPE210-S. During this critical temperature range, the material softens and flows at low viscosity before it melts completely, which provides the best characteristics for sintering. It is possible that high degree of sintering with full coalescence took place at this temperature, thus provided improved tensile properties of the sintered parts.

c) The effect of flow agent addition at two sets of constant sintering speed and temperature parameter

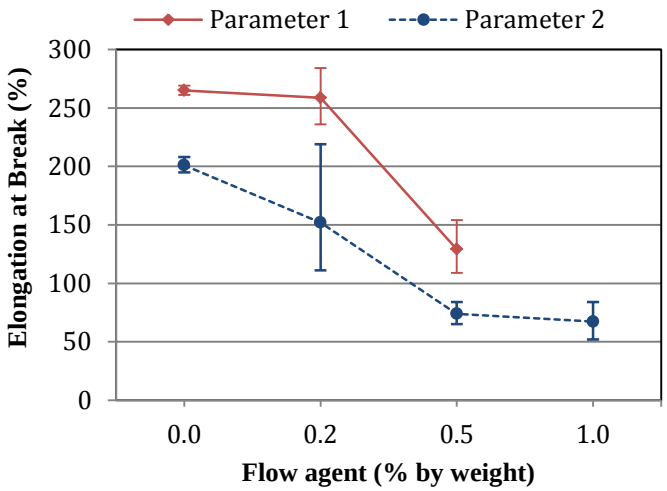

Figure 10 – Variation of EaB with flow agent level

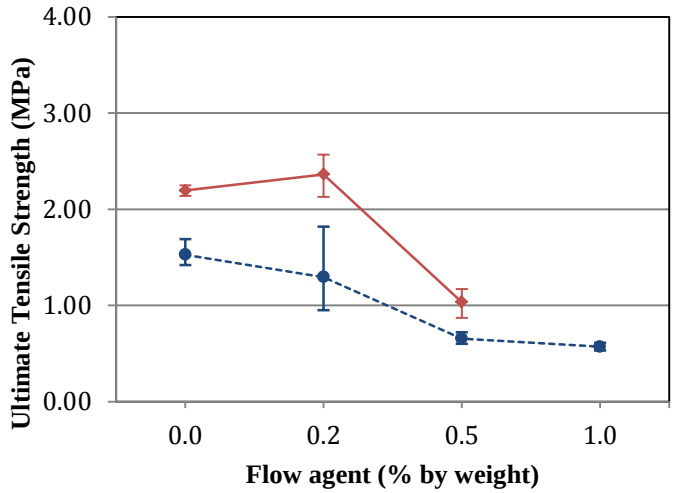

Figure 11 – Variation of UTS with flow agent level

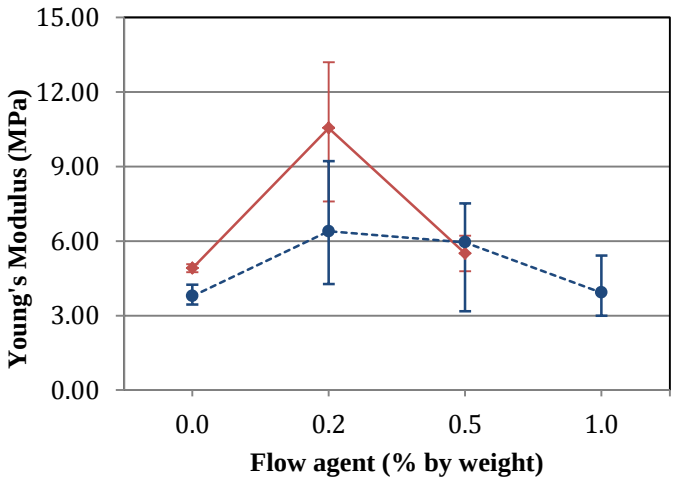

Figure 12 – Variation of YM with flow agent level

A flow agent (fumed silica) was added to the following two best set of parameters to further enhance their finished parts properties;
Parameter 1 : (100°C bed temperature, 50 mm/s sintering speed) & Parameter 2 : (110°C bed temperature, 70 mm/s sintering speed).

The influence of flow agent addition on EaB, UTS and YM are plotted in Figure 10, Figure 11 and Figure 12 respectively, with comparison to 100% virgin-powder sintered parts.

The maximum value of EaB, UTS and YM are observed at 0.2% flow agent by weight, which are 284%, 2.57 MPa and 10.90 MPa respectively.

All figures indicate a steady decrease in EaB, UTS and YM when more flow agent is mixed with virgin powder pass 0.2% level.

The influence of polymer additives on HSS part performance can be explained by Frenkel's Model of Sintering which relates particle viscosity to predicted sintering rate. Zero shear viscosity rate for TPE210-S in unknown. However, it is relatively high compared to Nylon-12. It can be speculated that fumed silica promotes fluidity and lowers viscosity of the TPE210-S mix. A decreased viscosity lowers sintering time, thus allowing less energy addition. As low thermal energy is absorbed and tensile properties depend largely on the amount of heat, this will subsequently result in parts of inferior properties

d) Comparison between mechanical properties of parts processed by High Speed Sintering, Selective Laser Sintering and Injection Moulding

Process	% by weight of fumed silica mix	EaB (%)	YM (MPa)	UTS (MPa)
HSS	0.2	259	10.55	2.36
HSS	0	265	4.91	2.20
LS (5)	0	110	8.00	N/A
Injection Moulding (9)	0	495	4.48	1.06

Table 7 – Part performance: Comparison between HSS, LS and IM

2. Thermal Properties

Figure 13 shows the thermal scan of TPE210-S mixed with 0.2% by weight Cab-O-Sil compared to the thermal scan of virgin TPE210-S**.** The two curves are identical in shape, with physical transitions occurring at constant temperatures T_g (105°C), T_m (140°C) and T_c (50°C). Minor distortions are observed which may be due to the effect of impurity during mixing or irregular sample composition in the pan.

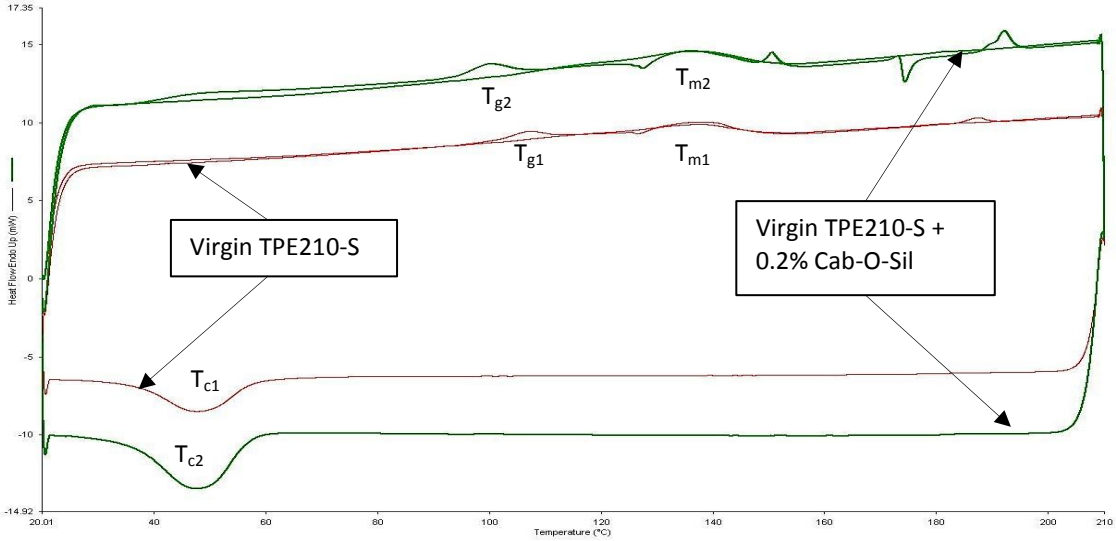

Figure 13 – DSC scan of TPE210-S mixed with 0.2% Cab-O-Sil and virgin TPE210-S

The mix powder curve (outer) have significantly shifted in the direction of heat flow at a value of around 5 mW from the virgin curve (inner). Both melt peak (T_{m2}) and crystallisation peak (T_{c2}) appear sharper in the mix powder curve compared to T_{m1} and T_{c1}. This suggests the addition of flow agent has increased the amount of energy required to change the material from solid to liquid i.e heat of fusion, ΔH_m and the amount of energy released during crystallisation, ΔH_c. As both transitions take place at the constant temperatures of 140°C and 50°C, consequently higher thermal energy is involved in both phase changes. The presence of silica atoms in between the TPE atoms may have an effect on the molecules arrangement, making them closely packed together. This subsequently strengthens the intermolecular bonds thus more energy is needed to break them. It is also observed that the range of T_g in both curves differed slightly, with ΔT_{g2} higher than ΔT_{g1}, by approximately 5°C. The process window is widened thus providing advantage to the sintering process at lower temperature range. In conclusion, the addition of flow agent in powder does not alter the original physical properties of powder, instead enhance its thermal properties.

3. Surface Roughness

The effect of adding flow agent into the powder mix can be observed through the following graph of average roughness variation in Figure 14.

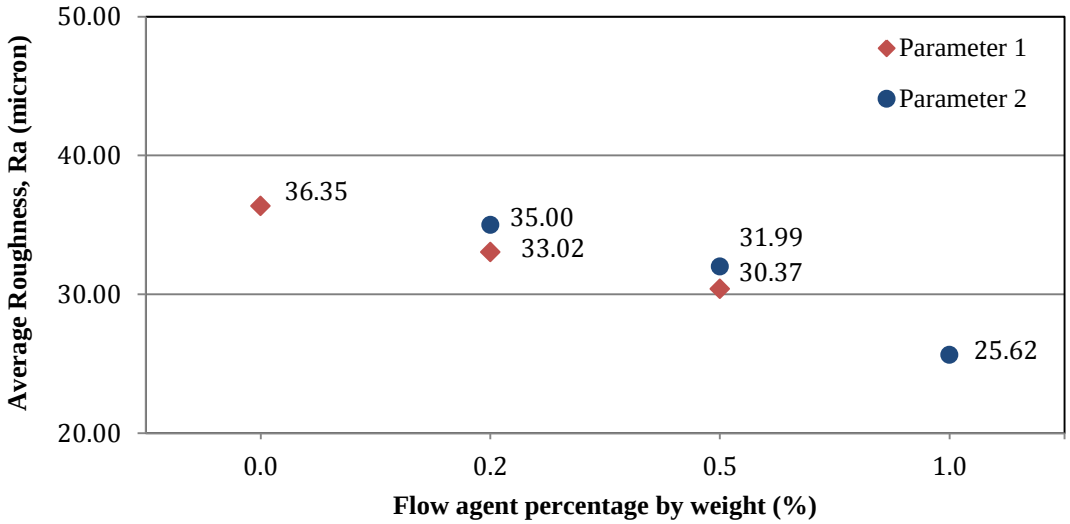

Figure 14 – Average roughness values against flow agent addition

The addition of flow agent generally reduces the surface roughness by promoting better flow between the particles during sintering. However the values of R_a are still relatively high compared to parts produced by other manufacturing technologies and materials.

Conclusions

The experiments have shown that High Speed Sintering process produces more superior parts compared to equivalent Laser Sintered parts. The addition of fumed silica decreases part's surface roughness. However further addition of fumed silica results in a decrease of Elongation at Break, evident increase of Young's Modulus and mixed results in Ultimate Tensile Strength.

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