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Examining Mechanical Strength Characteristics of Selective Inhibition Sintered HDPE Specimens Using RSM and Desirability Approach

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Abstract. Selective inhibition sintering (SIS) is a powder based additive manufacturing (AM) technique to produce functional parts with an inexpensive system compared with other AM processes. Mechanical properties of SIS fabricated parts are of high dependence on various process parameters importantly layer thickness, heat energy, heater feedrate, and printer feedrate. In this paper, examining the influence of these process parameters on evaluating mechanical properties such as tensile and flexural strength using Response Surface Methodology (RSM) is carried out. The test specimens are fabricated using high density polyethylene (HDPE) and mathematical models are developed to correlate the control factors to the respective experimental design response. Further, optimal SIS process parameters are determined using desirability approach to enhance the mechanical properties of HDPE specimens. Optimization studies reveal that, combination of high heat energy, low layer thickness, medium heater feedrate and printer feedrate yielded superior mechanical strength characteristics.

1. Introduction

Selective Inhibition Sintering (SIS) is one of the novel and inexpensive AM techniques to fabricate parts on a layer-by-layer basis that has been introduced at University of Southern California, USA [1]. The crux of the SIS process is cohesion of polymer powders through sintering and inhibition at the part boundaries. SIS process has attractive features like rapid fabrication, use of wide range of processing materials (polymer, ceramic, and metal), enhanced sintering capability, and cost effective that leads to a great impact to RP community [2]. The following steps are involved in the production of SIS processed part:

1. Polymer powder is initially spreaded over the build platform through roller mechanism to build a first layer,
2. Inhibitor is deposited at the selected boundaries of built layer through print head nozzle, which is moving in X-Y plane,
3. Formulated layer is heated by an inexpensive heating system to perform sintering of polymer powder particles,

4. Layer by layer addition of materials with the above sequence will be repeated until the desired part profile is obtained. Finally, fabricated part is extracted from build chamber and post processing including removal of inhibitor, and cleaning of surplus materials from the part completes the SIS process.

Although SIS process is an economical and rapid way to fabricate functional parts, full-scale commercialization of this process has not gained much attention because of compatibility issues on the selection of process parameters. Asiabanpour *et. al.* [3] conducted few experiments in SIS process evaluation resulted that, layer thickness, heater energy and heater feed rate have affected the mechanical properties. In order to obtain maximum mechanical strength, it is essential to have complete control over the relevant process parameters [4]. Statistical design of experiments and empirical modelling of process parameters are efficient methodologies to improve the quality and performance characteristics [5-6]. Therefore, in this analysis an attempt has been made to model and optimize the SIS process parameters for maximizing mechanical strength of high density polyethylene (HDPE) specimens using response surface methodology and desirability approach.

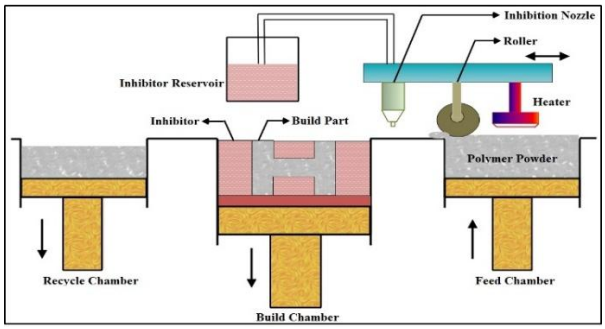

Figure1. Selective inhibition sintering process

2. Experimental methods

HDPE powders of particle size ranging from 25-40 μm is used as base powder and potassium iodide is considered as inhibitor throughout the study. Based on the existing literature [3], various SIS process parameters and their levels are accounted for part fabrication which is given in Table 1. The parts are built using a self-developed laboratory SIS machine. The schematic layout of SIS process is depicted in Fig.1. The working range of each process parameters is analyzed through inspecting the degradation of polymer and thermal stress distribution on specimen surface.

Table 1. SIS process control factors and their levels

Level	Layer thickness (mm)	Heater energy (J/mm^2)	Heater feedrate (mm/sec)	Printer feedrate (mm/min)
	A	B	C	D
Level 1	0.1	22.16	3	100
Level 2	0.15	25.32	3.25	110
Level 3	0.2	28.48	3.5	120

In order to plan and conduct the experiments, four-factor three-level response surface methodology (RSM) based box-behnken design [7] is considered and the corresponding design matrix is shown in Table 2. The tensile and flexural test specimens are fabricated with reference to ASTM standards D638 and D790, respectively. Structural strengths of SIS specimens are assessed under ambient conditions through Zwick/Roell automated material testing system with crosshead speeds of 2 mm/min. Experiments are performed and SIS part specimens mechanical strength characteristics are estimated. It is observed from Table 2 that, maximum tensile strength (TS) of 26.44 MPa and flexural

strength (FS) of 64.88MPa is attained at a layer thickness of 0.1mm with heat energy of 28.48 J/mm^2, heater feed rate of 3.25mm/min and printer feed rate of 110mm/min.

Table 2. Experimental layout using box-behnken design to evaluate mechanical strengths

Run	Layer Thickness (mm)	Heater Energy (J/mm^2)	Heater Feed rate (mm/sec)	Printer Feed rate (mm/min)	Tensile Strength (MPa)	Flexural Strength (MPa)
1	0.20	25.32	3.50	110	23.05	40.46
2	0.15	28.48	3.00	110	21.29	56.64
3	0.10	25.32	3.50	110	22.53	36.34
4	0.15	25.32	3.25	110	25.55	55.63
5	0.20	25.32	3.25	100	24.39	46.35
6	0.10	25.32	3.25	120	24.94	47.18
7	0.15	25.32	3.50	120	21.53	47.2
8	0.15	25.32	3.00	120	20.41	43.11
9	0.15	28.48	3.50	110	25.92	53.57
10	0.15	25.32	3.25	110	25.12	54.55
11	0.15	28.48	3.25	100	24.64	53.13
12	0.15	22.16	3.25	100	23.97	48.9
13	0.20	25.32	3.00	110	20.86	44.18
14	0.15	22.16	3.25	120	21.29	45.04
15	0.15	22.16	3.50	110	19.34	39.24
16	0.20	22.16	3.25	110	23.77	52.57
17	0.15	25.32	3.25	110	25.29	55.22
18	0.10	25.32	3.00	110	25.87	57.6
19	0.15	25.32	3.50	100	21.62	45.4
20	0.15	25.32	3.25	110	24.9	52.22
21	0.15	25.32	3.25	110	24.7	52.1
22	0.15	25.32	3.00	100	24.97	53
23	0.15	28.48	3.25	120	25.25	54.19
24	0.10	25.32	3.25	100	24.85	52.11
25	0.10	28.48	3.25	110	26.44	64.88
26	0.10	22.16	3.25	110	23.8	48.01
27	0.20	28.48	3.25	110	24.97	48.94
28	0.15	22.16	3.00	110	24.47	47.71
29	0.20	25.32	3.25	120	22.9	35.77

3. Results and discussion

3.1. Statistical analysis and development of models

The statistical analysis and adequacy of developed models are tested using ANOVA (Analysis of Variance) at 95% significance level. The results of linear and quadratic order response surface model fitting for TS and FS in the form of ANOVA are depicted in Table 3. Statistical Design Expert v6.0 software is used to evaluate the effects of process parameters, data analysis, and quadratic model building. Further, the effectiveness of model is validated using co-efficient of determination (R^2) value. In the present work, R^2 value is 96.3% for TS and 86.7 % for FS is closer to 1, which has reasonable agreement and indicates adequacy of developed models. The adequate precision ratio for TS and FS is above 4 specifies adequate model discrimination.

Table 3. The ANOVA table for the fitted models

Source	Sum of square	d.f	Mean square	F	p	R^2	Adj.R^2	Pre.R^2	Adequate precision
TS									
Model	95.325	14	6.809	25.927	<0.0001	0.963	0.926	0.805	21.361
Total	99.002	28							
Residual	3.677	14	0.263						
Lack of fit	3.238	10	0.324	2.955	0.1539				
Pure error	0.438	4	0.109						
FS									
Model	1060.9	14	75.78	6.51	0.0006	0.867	0.734	0.271	9.937
Total	1223.9	28							
Residual	162.97	14	11.64						
Lack of fit	151.76	10	15.18	5.415	0.059				
Pure error	11.211	4	2.803						

The normal probability plot of the residuals of TS and FS are shown in Fig.2 (a-b). It reveals that the residuals are falling on the straight line that signifies the errors are distributed normally.

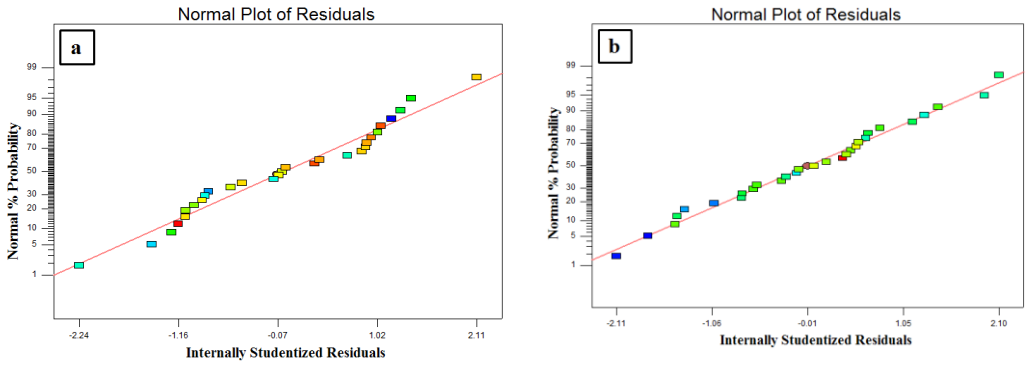

Figure2. Normal probability plot for TS and FS

From the above analysis after excluding the insignificant terms, the second-order quadratic models (i.e., in terms of actual values) for TS and FS are obtained and disclosed in Eqn.1 and 2.

$$\begin{aligned} Tensile\ strength &= 70.459 - 232.489\times A - 10.352\times B + 66.357\times C - 0.032\times D \\ &-2.278\times AB + 110.6\times AC - 0.79\times AD + 3.088\times BC + 0.026\times BD + 0.447\times CD \\ &+11.6\times A^2 - 0.037\times B^2 - 32.556\times C^2 - 0.009\times D^2 \end{aligned} \quad (1)$$

$$\begin{aligned} Flexural\ strength &= -477.873 + 357.254\times A - 9.911\times B + 262.669\times C + 4.1\times D \\ &-32.437\times AB + 350.8\times AC - 2.825\times AD + 1.708\times BC + 0.039\times BD + 1.169\times CD \\ &-1427.97\times A^2 + 0.123\times B^2 - 76.999\times C^2 - 0.039\times D^2 \end{aligned} \quad (2)$$

Where A, B, C and D are layer thickness, heater energy, heater feedrate, and printer feedrate, respectively. Overall, statistical analysis reflected that the experimental values are fitted well within predicted ones and the accuracy of the model is adequate to further explore optimization studies on determining SIS process parameters.

3.2. Effect of process parameters on Tensile strength

The effect of layer thickness and heater energy on tensile strength is shown in Fig. 3a. It can be seen from the plot that tensile strength is maximum at higher heater energy, whereas it slightly decreases with increase in layer thickness. It is consistent with the fact that increase in heat energy improves the degree of sintering and flow of polymeric particles and a well-defined structure is formed. Hence,

tensile strength of the specimen increased with an increase in heater energy. At a low level of layer thickness, the sufficient heat energy can be transferred to the powder particles resulted in better fusion of HDPE particles and achieved a more compact structure. When layer thickness increases, distortion effect dominates the bonding effect and decreases tensile strength of specimen. The interaction effect of heater feedrate and layer thickness on tensile strength is depicted in Fig.3b. It is observed that with an increase in layer thickness causes decrease in tensile strength from 25.39 to 22.15 MPa.

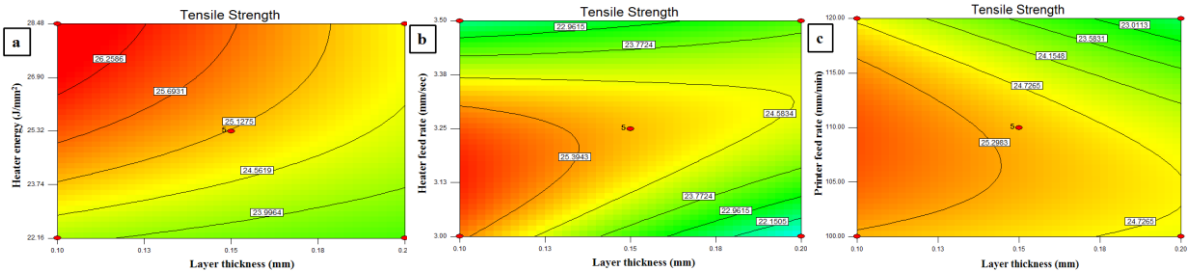

Figure3. 2D surface graphs for Tensile strength (a) effect of layer thickness and heater energy, (b) effect of layer thickness and heater feedrate, and (c) effect of layer thickness and printer feedrate

However, increase in heater feedrate improves the TS up to 3.28 mm/sec and then decreases. It can be justified with the fact that at maximum heater feedrate, the energy absorbed by powder particles is not uniform due to shorter heat time which causes improper sintering. Therefore, poor sintering decreases the tensile strength of sintered specimens. Fig. 3c demonstrates the effect of printer feedrate and layer thickness on tensile strength. It is inferred from simulation analysis that, tensile strength increases up to maximum value of 25.29 MPa and then decreases with increase in printer feedrate from 100 to 120 mm/min.

3.3.Effect of process parameters on Flexural strength

The response surface plots in Fig.4 (a-c) describe the effect of input process parameters on flexural strength. It can be understood that the effects of process parameters on evaluating flexural strength have similar trend as in the case of tensile strength of the sintered specimens. Fig. 4a reflects the effect of heater energy and layer thickness on flexural strength. It is observed that, flexural strength increases from 48.574 MPa to 60.94 MPa with increase in heater energy from 22.16 to 28.48 J/mm^2, whereas slightly decreases with the increase in layer thickness ranging from 0.1 to 0.2 mm. Similarly, the effect of heater feedrate and layer thickness on flexural strength has shown in Fig.4b. It can be seen that, flexural strength slightly increases with increase in heater feedrate from 3 to 3.25 mm/sec and layer thickness up to 0.14 mm and then decreases with the increases in these two parameters. Finally, the effect of printer feedrate and layer thickness depicted in Fig.4c indicates that, at low layer thickness of 0.1 mm and middle range of printer feedrate (110 mm/min) attained maximum flexural strength.

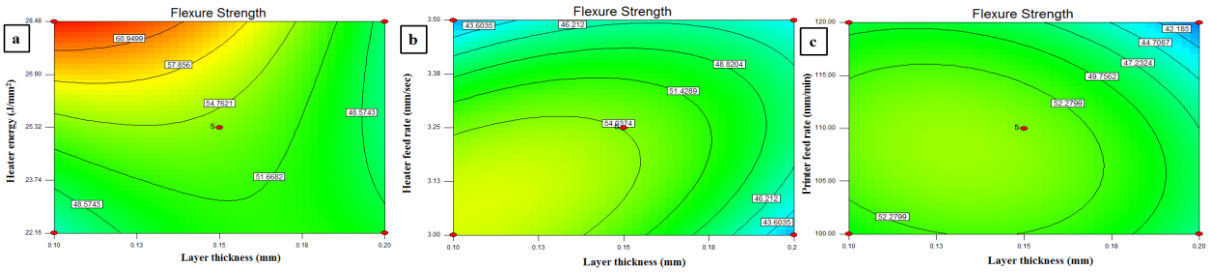

Figure4. 2D surface graphs for Flexural strength (a) effect of layer thickness and heater energy, (b) effect of layer thickness and heater feedrate, and (c) effect of layer thickness and printer feedrate

3.4. Optimization of SIS process parameters

Due to the presence of large number of input parameters in SIS and intricate stochastic process mechanism has tremendous effect in selection of optimal SIS process parameters to achieve improved

mechanical properties. The present study emphasise a multi-objective problem to simultaneously optimize the process variables using desirability approach which is proposed by Derringer and Suich [8]. It is a unique optimization technique used for optimizing multiple quality characteristic problems in industries [9]. In a multi response domain, a measure of how the solution has satisfied the combined goals for all responses must be assured [10]. In this work, based on composite desirability optimization technique, the two responses i.e., tensile and flexural strengths have been optimized simultaneously using developed mathematical models as described in Eqn.1 and 2. The ramp desirability graph shown in Fig.5 describes the optimum points obtained through numerical optimization procedure. It reveals that, earlier predicted maximum tensile strength of 26.44 MPa and flexural strength of 64.33 MPa can be attained under the SIS process conditions such as layer thickness of 0.11 mm, heater energy of 28.48 J/mm^2, heater feedrate of 3.21 mm/sec, and printer feedrate of 112.53 mm/min.

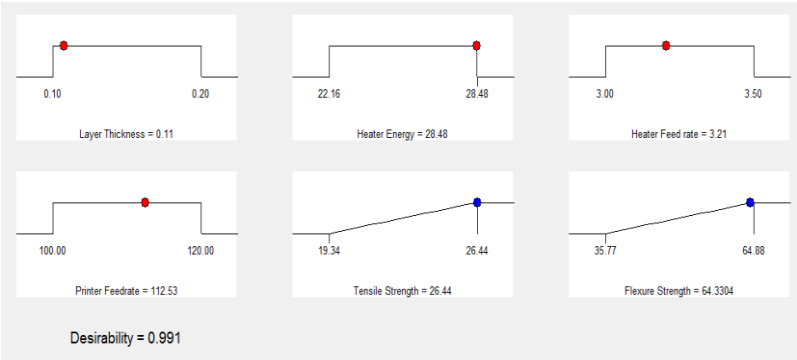

Figure5. Desirability ramp function for numerical optimization

4. Conclusions

HDPE specimens have been fabricated through SIS process. The mechanical properties including tensile strength and flexural strength are investigated using RSM with box-behnken design. Empirical models are developed to predict the mechanical properties of the sintered specimens. The following inferences are deduced from this study.

- Mathematical models of TS and FS are formulated to correlate the dominating input parameters, including layer thickness, heater energy, heater feedrate, and printer feedrate.
- The maximum tensile strength of 26.44 MPa and flexural strength of 64.33 MPa could be attained through considering optimal SIS process parameters layer thickness of 0.11 mm, heater energy of 28.48 J/mm^2, heater feedrate of 3.21 mm/sec, and printer feedrate of 112.53 mm/min.
- Influence of heater energy followed by layer thickness and printer feedrate has predominant effect on mechanical strength. However, heater feedrate has insignificant effect on parts strength.
- The higher heater energy of 28.48 J/mm^2 offered higher mechanical strength. On contrary, TS and FS decreased with increase in layer thickness from 0.1 to 0.2 mm.

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