Journal of Applied Science and Engineering, Vol. 22, No. 3, pp. 391–402 (2019) *DOI: 10.6180/jase.201909_22(3).0001*

Investigation of Sintering Parameters on Viscoelastic Behaviour of Selective Heat Sintered HDPE Parts

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Abstract

Selective heat sintering (SHS) process aims to produce near net shape components through sintering of specific region of powder particles. Evaluation of viscoelastic properties of SHS parts are of major importance to produce functional parts for diverse applications. The present study focuses on investigation of SHS governing parameters on loss modulus, storage modulus and damping factor of high density polyethylene (HDPE) specimens. SHS system is custom built and experiments are conducted based on four factors three level box-behnken design. The interaction among SHS process variables for examining the viscoelastic properties using response surface analysis is performed. Optimal SHS process variables are obtained using non-desirability statistical approach. Morphological examinations are conducted using scanning electron microscope (SEM) where in pull outs, voids and pores are observed in the sintered surfaces. The results revealed that at high heater energy (26.32 J/mm^2) and low layer thickness (0.1 mm) with high heater feedrate (3.5 mm/sec) and printer feedrate (116.38 mm/min) is beneficial for improving viscoelastic properties of sintered specimens. These analyses provided an insight for the fabrication of near net shape components with sufficient viscoelastic properties.

Key Words: Additive Manufacturing, Sintering, Optimization, Desirability, Viscoelastic Properties

1. Introduction

Additive manufacturing (AM) is considered to be a disruptive technology to manufacture the intricate part profiles for quick realization and functional prototypes for diverse applications pertaining to aerospace, automobile, medical, architectural and toy industries. Non usage of jigs, fixtures, specific tools and moulds made AM as potential replacement of conventional manufacturing process. AM has huge market demands due to cost effectiveness, superior surface quality, high dimensional accuracy and good mechanical strength characteristics. There are more than twenty AM processes are available in the market and most of them utilizes costly laser system. The import of high quality materials (metals and non-metals) for the commercially available AM systems is a burden to the user groups. Hence, it is necessary to implement cost effective heating system and use of indigenously available material resources are of greater impact in mass production. Considering these aforementioned issues a novel powder based AM technique namely selective heat sintering (SHS) is established at University of Southern California [1]. In this process, polymer powder particles are swapped with an aid of roller mechanism from the feed tank to the build chamber. The machine tool path generated from CAD data is used to define the part profile and boundary where in nozzle will deliver the inhibitor. Once the region of separation from desired part body through inhibition is achieved, a low cost heating unit will supply the thermal radiation to the polymer powders. The particles of polymer is heated to below the melting point and they are sintered at specific temperature.

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Thus the layer by layer sintering with inhibition on the parts boundary continues until the final 3D part is manufactured.

The SHS process has many advantages than commercially available AM processes. SHS utilizes variety of polymers including low and high density polyethylene, polyamide, polycarbonate etc. In contrast to FDM, this method does not require costly support structures. The avoidance of high power lasers or electron beams reduces the system cost. In spite of its potential benefits, the uses of SHS in real time applications is currently inadequate because of inferior part quality characteristics such as dimensional accuracy, mechanical strength, viscoelastic properties, surface quality, tribological properties and service life. Enhancing parts strength characteristics has tremendous influence in automobile, armament and bio-medical applications.

The viscoelastic properties of SHS fabricated components are depends on type of polymers and influence process variables. The quality of SHS fabricated parts depends on many controllable factors such as thickness of layer, heat energy, feedrate of heater, printer federate, printer pressure, build tank temperature, enclosure temperature and roller feedrate [2]. The proper control of these factors provides desired part strength. In order to achieve efficient viscoelastic properties of SHS parts, optimal operating conditions are to be determined and deep-rooted mathematical relationship between input parameters and responses needs to be established. Therefore, statistical-mathematical models can be developed to endeavour the correlation among different process variables and response characteristics [3]. Several researchers are used the statistical and optimization techniques includes taguchi technique and response surface methodology to improve the quality and performance features of various AM fabricated parts such as shrinkage characteristics [4,5], part strength [6,7], wear rate [8,9], surface finish [10–12], and viscoelastic properties [13,14]. In the literature, a few studies [15,16] are conducted to explore the influence of SHS sintering parameters on improving the performance and quality features of sintered specimens.

Several researchers have used the statistical and optimization techniques such as taguchi and response surface methodology technique to improve the quality and performance characteristics of various AM fabricated specimens such as shrinkage, mechanical strength, wear rate, and surface finish are function of various governing parameters and can be considerably enhanced with suitable adjustments. However, in concern with SHS process, only few studies [17–20] were performed to optimize the governing parameters to obtain enhanced part quality.

To the best of author's knowledge, the impact of SHS governing parameters on evaluating the viscoelastic properties are not dealt by any researcher. Therefore, this present work deals the influence of various SHS governing parameters such as applied heat energy, thickness of powder layer, feedrate of heater and inhibition printer on viscoelastic properties including loss modulus, storage modulus and damping parameter of sintered HDPE parts. RSM based BBD was utilized to design and conduct the experiments. The influence of process variables on the selected responses are assessed through analysis of variance (ANOVA) technique. Further, desirability statistical approach is incorporated to attain optimal SHS governing parameters to enhance the viscoelastic properties.

2. Experimental Details

2.1 SHS system development and fabrication of Specimen

The test specimens for this study have been manufactured by using high density polyethylene (HDPE) powders through the developed SHS system (Figure 1) based on the geometry and dimensions specified in the ASTM D7028 standard. Specimen fabrication process was began by preparing the 3D CAD model using CATIA V5R20 modeling software. The CAD model was then exported to slic3r program as .STL file format. Then after, the model was sliced into finite number of thin sections, and G-codes for each sliced sections have been generated in order to control the SHS machine path during part fabrication. By using the generated G-codes, the developed SHS system does the following key operations among others in fabricating the desired specimens.

1. First, spread uniformly thin powder layer on the build tray.
2. Next, deposit inhibition liquid to the region of the pow-

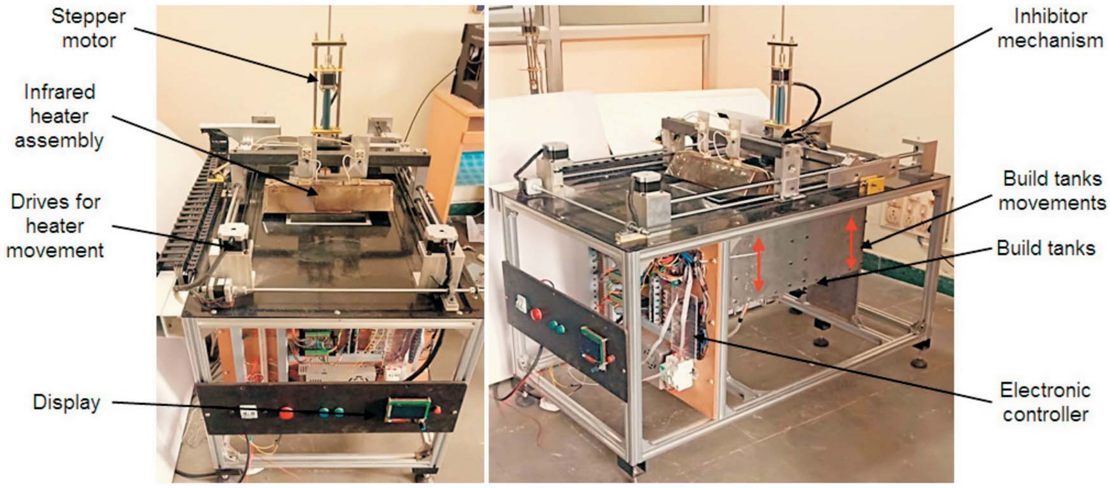

Figure 1. Selective heat sintering system.

der layer that define part boundary profile.
3. Finally, sinter the entire powder layer.
4. Repeat the above mentioned procedures until the entire part is printed.
5. Post processing of cleaning of inhibitor from the built part completes the SHS process.

The most influencing SHS governing variables were identified through conducting exhaustive preliminary experiments. From the experimental studies, the thickness of layer, feed rate of heater, inhibition printer rate and heater energy are identified as the most influencing parameters. Their numerical values and ranges are presented in Table 1. The following system parameters were consider as constant throughout the experiments. Build tank temperature of 90 °C, printer nozzle diameter of 0.4 mm and nozzle stand-off distance of 5 mm.

DMA Seiko SII Exstar 6100 DMS instrument is utilized to conduct experiments for dynamic mechanic analysis. The loss modulus (LM) storage modulus (SM) and damping parameter (DP) are determined for the SHS specimens of 50 × 10 × 3.5 mm. The range of heating is varied between 30 °C to 150 °C at a rate of 2 °C/min with different frequencies 0.2, 0.5, 1, 2, 5 and 10 Hz respectively. The measurement of viscoelastic properties given in Table 2 is the average value which is measured under similar processing conditions.

2.2 Experimental Design

Response surface methodology (RSM) is employed for modeling and optimizing the governing parameters [21]. The box-behnken design (BBD) is a RSM based approach which is utilized to minimize the experimental runs and quadratic model is established and also the interactions between the SHS governing parameters are studied. The second-order polynomial equation formed from RSM was utilized to manifest the behaviour of the SHS process is as shown in Eqn. (1). In this investigation, the viscoelastic properties of sintered specimens are modelled in accounting *HE*, *LT*, *HF* and *PF*.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_i \sum_j \beta_{ij} X_i X_j + \varepsilon \qquad (1)$$

In the present investigation, a second-order quadratic model is developed using RSM for correlating process variables and the responses. Additionally, ANOVA is exploited to justify the consequence of developed quadratic models.

Table 1. SHS governing parameters and levels

Symbols	Parameters	Units	Coded values		
			-1 (low)	0 (medium)	1 (high)
A	Heater energy (*HE*)	J/mm^2	22.16	25.32	28.48
B	Layer thickness (*LT*)	mm	0.1	0.15	0.2
C	Heater feedrate (*HF*)	mm/sec	3	3.25	3.5
D	Printer feedrate (*PF*)	mm/min	100	110	120

Table 2. Experimentally measured viscoelastic properties

Run	Heater energy (J/mm^2)	Layer thickness (mm)	Heater feedrate (mm/sec)	Printer feedrate (mm/min)	Loss modulus $\times 10^8$ (Pa)	Storage modulus $\times 10^9$ (Pa)	Damping parameter (Tan δ)
1	28.48	0.15	3.5	110	6.441	7.735	0.1235
2	25.32	0.2	3	110	2.736	4.322	0.0845
3	22.16	0.15	3.5	110	5.282	8.487	0.1045
4	25.32	0.15	3.25	110	3.097	8.977	0.0925
5	28.48	0.15	3.25	100	4.294	10.85	0.0975
6	22.16	0.15	3.25	120	4.522	9.173	0.0945
7	25.32	0.15	3.5	120	6.194	8.452	0.1145
8	25.32	0.15	3	120	3.648	7.421	0.1095
9	25.32	0.2	3.5	110	4.579	5.162	0.0755
10	25.32	0.15	3.25	110	3.135	9.249	0.0918
11	25.32	0.2	3.25	100	2.641	5.442	0.0635
12	25.32	0.1	3.25	100	3.648	9.595	0.1195
13	28.48	0.15	3	110	4.104	7.927	0.0965
14	25.32	0.1	3.25	120	3.876	9.594	0.1098
15	25.32	0.1	3.5	110	5.301	9.537	0.1420
16	28.48	0.1	3.25	110	4.351	8.686	0.1155
17	25.32	0.15	3.25	110	3.382	9.905	0.0947
18	22.16	0.15	3	110	4.731	6.615	0.0955
19	25.32	0.15	3.5	100	4.294	8.715	0.1187
20	25.32	0.15	3.25	110	3.363	8.995	0.0981
21	25.32	0.15	3.25	110	3.249	9.415	0.0915
22	25.32	0.15	3	100	4.579	8.715	0.1036
23	25.32	0.2	3.25	120	2.394	5.285	0.0834
24	22.16	0.15	3.25	100	3.401	5.967	0.0875
25	22.16	0.2	3.25	110	2.565	3.955	0.0750
26	22.16	0.1	3.25	110	4.066	8.662	0.1019
27	28.48	0.2	3.25	110	3.344	5.477	0.0705
28	25.32	0.1	3	110	4.940	9.222	0.0995
29	28.48	0.15	3.25	120	4.161	6.457	0.1024

3. Result and Discussions

3.1 Development of Mathematical Models and Empirical Analysis

The second-order mathematical models are developed to statistically analyse the relative importance of SHS governing parameters, the regression models are developed. The competency of established models is substantiated through analysis of variance (ANOVA) with 95% confidence interval using Design Expert 7 software. Tables 3−5 shows the ANOVA outcomes for the viscoelastic properties such as loss modulus, storage modulus and damping parameter. It is perceived from the tables that $p < 0.05$, which illustrates that the developed models are at 95% assurance level. Also, the factor of determination (R^2) for the loss modulus, storage modulus and damping parameter are also found to be 0.9806, 0.9765 and 0.9828, respectively. It illustrates that the established models reasonably fit with the real data. The adequate precision (AP) values obtained for selected responses are well above 4, that signposts the acceptable model perception. The model F values denote that the models are substantial. From the established models, certain variables can be observed at as inconsequential terms due to their "Prob. > F" value presence more than 0.05. The backward elimination approach was utilized to remove the inconsequential terms from the developed models. The remaining terms are retained for further estimation. The final regression models are given by,

$$Loss\ modulus(Pa) = +407.168 - 3.806 \times A - 80.978 \times B - 183.741 \times C - 1.012 \times D + 0.565 \times A \times C - 9.92 \times 10^{-3} \times A \times D + 29.64 \times B \times C + 0.283 \times C \times D + 0.061 \times A^2 - 95.19 \times B^2 + 20.968 \times C^2 + 1.634 \times 10^{-3} \times D^2 \quad (2)$$

$$Storage\ modulus(Pa) = -379.442 + 13.137 \times A + 82.832 \times B + 82.906 \times C + 1.497 \times D + 2.381 \times A \times B - 0.653 \times A \times C - 0.061 \times A \times D - 0.0917 \times A^2 - 619.523 \times B^2 - 10.011 \times C^2 \quad (3)$$

Table 3. ANOVA for response surface reduced quadratic model (Loss modulus)

Source	Sum of squares	DOF	Mean square	F value	Prob > F	
Model	28.2926	12	2.3577	67.245	< 0.0001	Significant
A – Heater energy	0.3774	1	0.3774	10.763	0.0047	
B – Layer thickness	5.2312	1	5.2312	149.200	< 0.0001	
C – Heater feedrate	4.5056	1	4.5056	128.504	< 0.0001	
D – Printer feedrate	0.3130	1	0.3130	8.927	0.0087	
AC	0.7974	1	0.7974	22.744	0.0002	
AD	0.3931	1	0.3931	11.213	0.0041	
BC	0.5491	1	0.5491	15.661	0.0011	
CD	2.0036	1	2.0036	57.147	< 0.0001	
A^2	2.4506	1	2.4506	69.893	< 0.0001	
B^2	0.3673	1	0.3673	10.477	0.0052	
C^2	11.14	1	11.14	317.74	< 0.0001	
D^2	0.1732	1	0.1732	4.940	0.0410	
Residual	0.5610	16	0.0351			
Lack of fit	0.4943	12	0.0412	2.470	0.1984	Not significant
Pure error	0.0667	4	0.0167			
Cor. total	28.8535	28				
R^2	0.9806	Adj. R^2	0.9660	AP	32.3693	

Table 4. ANOVA for response surface reduced quadratic model (Storage modulus)

Source	Sum of squares	DOF	Mean square	F value	Prob > F	
Model	94.114	10	9.411	74.763	< 0.0001	Significant
A – Heater energy	1.519	1	1.519	12.070	0.0027	
B – Layer thickness	54.773	1	54.773	435.112	< 0.0001	
C – Heater feedrate	1.246	1	1.246	9.902	0.0056	
D – Printer feedrate	0.703	1	0.703	5.587	0.0295	
AB	0.566	1	0.566	4.498	0.0481	
AC	1.066	1	1.066	8.469	0.0093	
AD	14.421	1	14.421	114.558	< 0.0001	
A^2	5.652	1	5.652	44.900	< 0.0001	
B^2	16.137	1	16.137	128.194	< 0.0001	
C^2	2.634	1	2.634	20.921	0.0002	
Residual	2.266	18	0.126			
Lack of fit	1.686	14	0.120	0.831	0.6473	Not significant
Pure error	0.580	4	0.145			
Cor. total	96.380	28				
R^2	0.9765	Adj. R^2	0.9634	AP	31.6409	

Table 5. ANOVA for response surface reduced quadratic model (Damping parameter)

Source	Sum of squares	DOF	Mean square	F value	Prob > F	
Model	0.00812	11	0.00074	88.297	< 0.0001	Significant
A – Heater energy	0.00023	1	0.00023	26.955	< 0.0001	
B – Layer thickness	0.00478	1	0.00478	571.796	< 0.0001	
C – Heater feedrate	0.00066	1	0.00066	78.961	< 0.0001	
D – Printer feedrate	0.00005	1	0.00005	5.505	0.0313	
AB	0.00005	1	0.00005	5.861	0.027	
AC	0.00008	1	0.00008	9.159	0.0076	
BC	0.00066	1	0.00066	79.317	< 0.0001	
BD	0.00023	1	0.00023	26.915	< 0.0001	
B^2	0.00011	1	0.00011	12.562	0.0025	
C^2	0.00107	1	0.00107	127.992	< 0.0001	
D^2	0.00014	1	0.00014	16.653	0.0008	
Residual	0.00014	17	0.00001			
Lack of fit	0.00011	13	0.00001	1.072	0.525	Not significant
Pure error	0.00003	4	0.00001			
Cor. total	0.00826	28				
R^2	0.9828	Adj. R^2	0.9717	AP	41.792	

$$Damping\ parameter(\tan\delta) = +2.763 - 0.0133\times A + 2.333 \times B - 1.267\times C - 0.012\times D - 0.0221\times A\times B + 5.537\times 10^{-3} \times A\times C - 1.03\times B\times C + 0.015\times B\times D - 1.58\times B^2 + 0.201 \times C^2 + 4.548\times 10^{-5}\times D^2 \quad (4)$$

From the ANOVA tables, it is observed that the loss modulus and damping parameter has significantly affected by *LT* and *HF*, whereas storage modulus is substantially influenced by *LT*. Moreover, the normal probability plots shown in Figure 2(a–c) indicated that the blunders are dispersed normally which implies that the proposed models are legitimately accurate and acceptable. Hence, the established second-order mathematical equations are realistically satisfactory in place of the SHS process.

3.2 Influence of Sintering Parameters on Selected Responses

In this investigation, the consequence of diverse SHS governing parameters such as heat energy and feedrate, layer thickness, and printer feedrate on the viscoelastic behaviour of sintered specimens are investigated. From Figure 3(a–h), it is observed that the designated governing parameters are partaking a considerable effect on viscoelastic properties such as *LM*, *SM* and *DP*.

3.2.1 Impact of Heater Energy

Figure 3(a, b, d, e and f) signifies the linear influence of heat energy on the viscoelastic behaviour of sintered specimens. In accordance with the melting point of HDPE, applied heat energy is varied from 22.16 J/mm^2 to 28.48 J/mm^2. The viscoelastic behaviours such as LM, SM and DP are increased with the increase in applied heat energy from low to high levels. It can be attributed due to the enhanced penetration of heat energy on the width and depth of powder layers during sintering process. The higher heat energy escalates the area of curing and simultaneously declines the amount of crystallinity [22]. The reduction of crystallinity leads dense structure with no obvious void formation. Therefore, the high density parts with enhanced viscoelastic properties were formulated at higher heat energy heater energy regardless of other governing parameters.

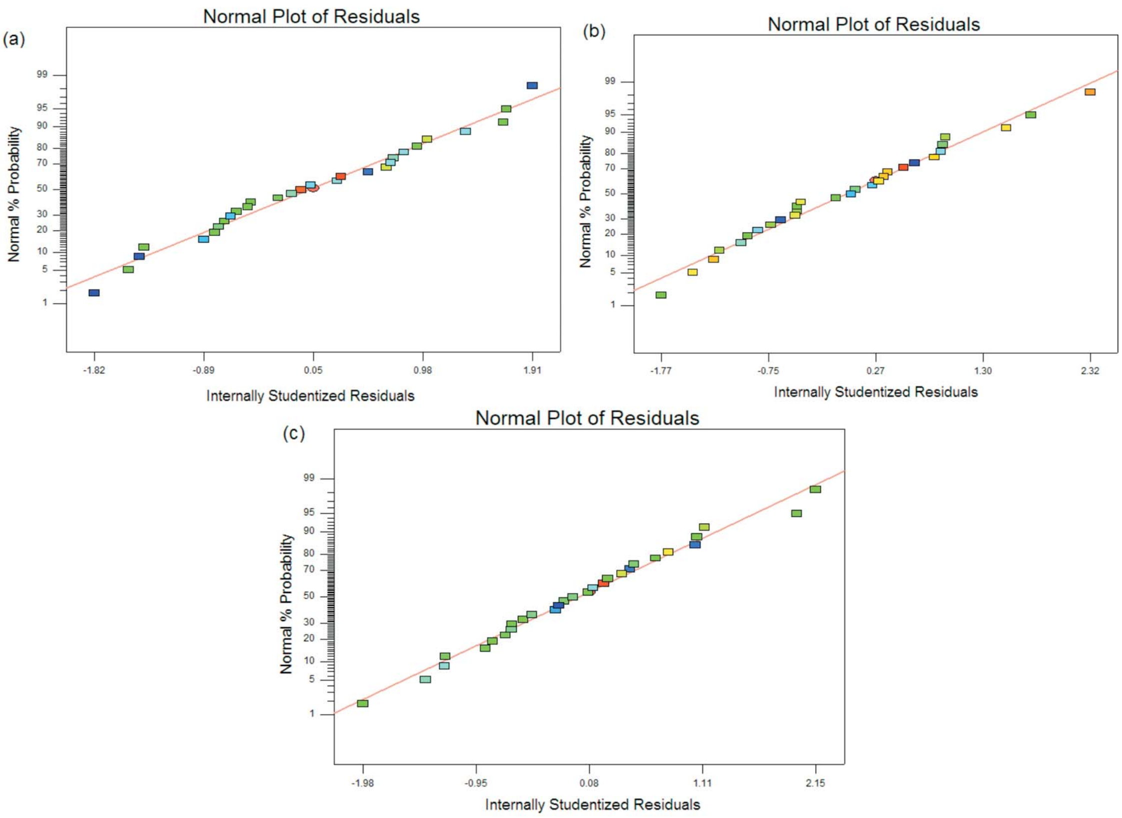

Figure 2. Normal probability plots for selected responses.

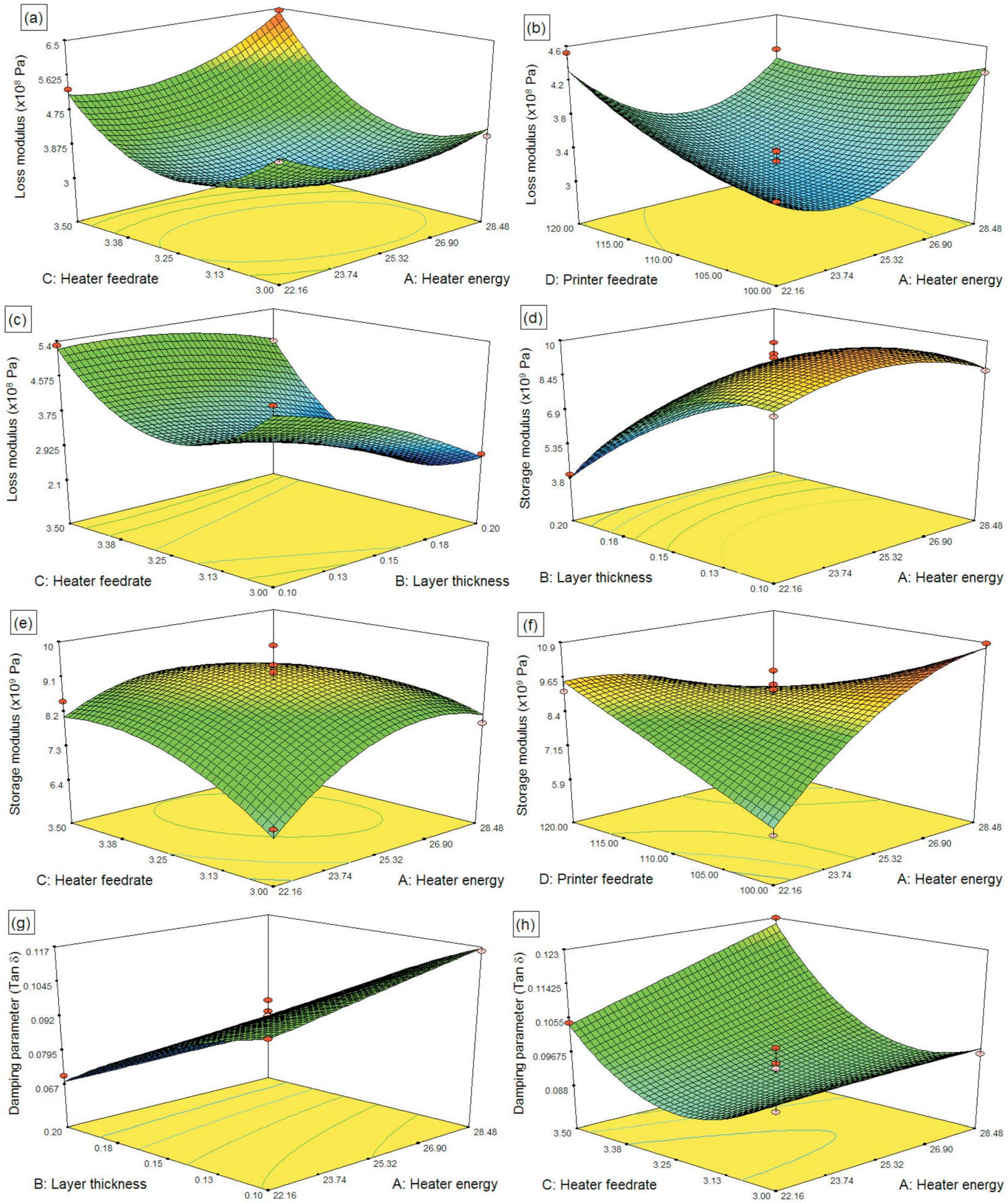

Figure 3. Influence of sintering parameters on viscoelastic properties.

3.2.2 Impact of Layer Thickness

Layer thickness is one of the most significant parameter which influences the viscoelastic properties of SHS parts. As the thickness of powder layer increases from 0.1 mm to 0.2 mm, viscoelastic properties are decreasing as observed in Figure 3(c, d and g). Sintering of powder particles with low thickness of layer leads to uniform penetration which leads to accurate sintering. From the response surface plots, the improved viscoelastic properties were observed at low thickness of layer, whereas the higher thickness of powder layer leads improper diffusion with increased porosity. Hence, the viscoelastic properties decreased with the increase in thickness of powder layer.

3.2.3 Impact of Heater Feedrate

The influence of heater feedrate on the viscoelastic behaviour of fabricated specimen is presented in Figure 3(a, c, e and h). It is observed from these figures that an increase of heater feed rate from 3 mm/sec to 3.5 mm/sec has improved the viscoelastic properties. As heater feedrate increases with a high heat energy or low thickness of layer, sufficient heat energy is transmitted from the heater to powder surface. Hence, the sintering time decreases and dense parts produced, which leads to improved viscoelastic properties. On the contrary, low heater feed rate may ancestries over melting of polymer powder and thus shrinks the viscoelastic behaviours such as *LM*, *SM* and *DP*.

3.2.4 Impact of Printer Feedrate

The substantial impact of printer feedrate on viscoelastic properties of fabricated specimens can be seen in Figure 3(b and f). The viscoelastic properties is found to be increase significantly with the increase in feedrate of inhibitor printer from a low level (80 mm/min) to a high level (120 mm/min). This could be attributed due to the increased inhibition spell and the inhibitor deposition takes place outside of the desired profile during low printer feedrate. This excess inhibitor penetration causes ineffective sintering on powder surface and hence the viscoelastic properties decreases. Conversely, increasing of printer feedrate provides accurate inhibition on sintering region, resulting increased loss modulus, storage modulus and damping parameters.

3.3 Microstructure of Sintered Specimen under Various Sintering Conditions

The SEM micrograph of the sintered specimens built under various sintering conditions are shown in Figure 4(a–d). At low heater energy of 22.16 J/mm^2, the polymer particles are marginally fused together at points of contact and the distinct particles can still be notorious as

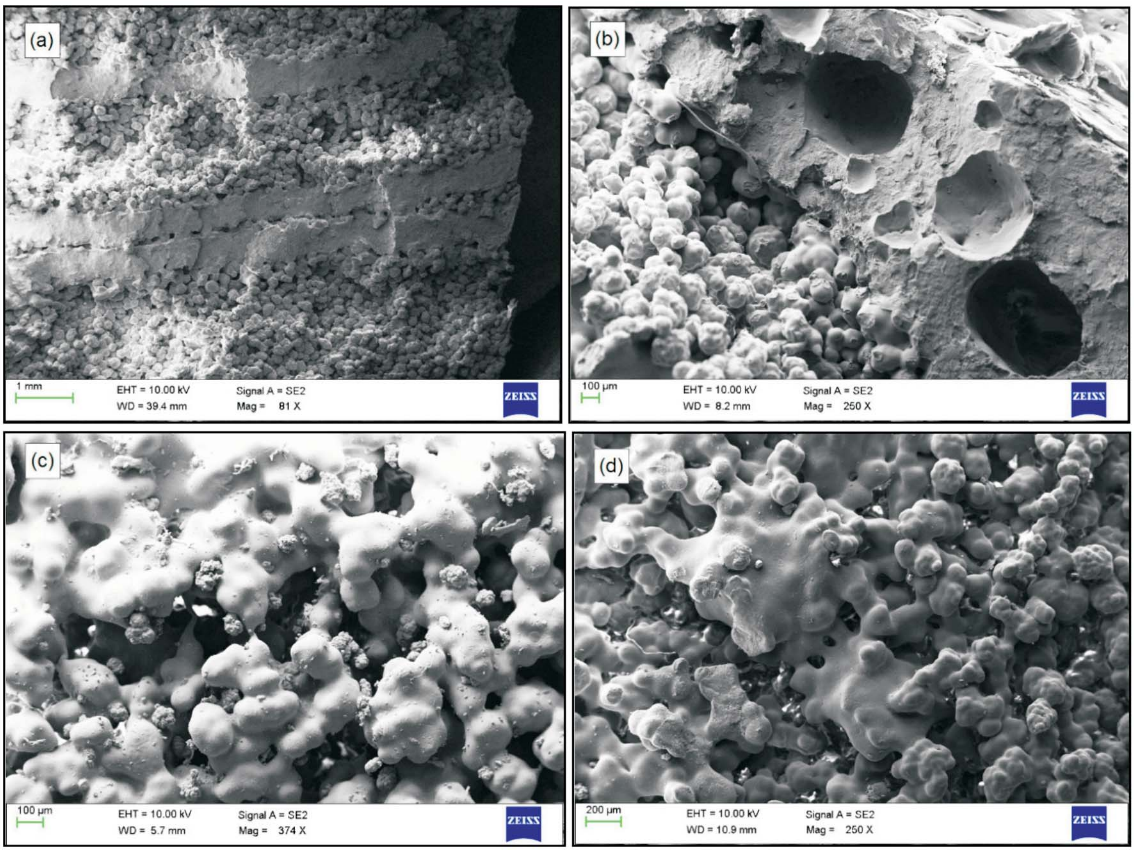

Figure 4. SEM micrographs of fabricated specimens at various sintering conditions.

shown in Figure 4a. This could be a reason for reducing the specimen strength and viscoelastic properties.

A partially sintered specimens at medium heater energy (25.48 J/mm^2) is presented in Figure 4b. Some necks are found protruding from the sintered surface, which are most likely instigated by unsintered powder particles beneath the surface. However, at HE = 28.48 J/mm^2, the microstructure of the sintered HDPE changed drastically, and distinct surface structure is formed (Figure 4c & 4d). This specifies that the flow of particle is expedited through enhanced fusion of the HDPE particles coalesce together under the driving force of surface tension. However, some protrusions are apparent in the sintered surface. It is due to unsintered polymer particles underneath the surface or insufficient heat energy input and time for the molten material to coalescence. They are likely caused by the high heater energy escalations, better fusion of polymer particles and facilitated a well-defined dense surface.

4. Multi Response Optimization

Multi-response optimization is carried out to identify the optimal SHS governing parameters and also maximize the viscoelastic properties in sintered specimen using desirability approach [23]. In this strategy, the goal used for the selected responses such as loss and storage modulus and also damping parameter are 'maximize' and for the factors are 'within range'. Weighing factors assigned (0.1 to 10) for the response variables to improve the contour of desirability function results. Process variables are from least to most (i.e., 1 and 5) are also varied to obtain suitable best solutions. An optimal condition is reached at the desirability of 0.854 for improving viscoelastic properties such as loss modulus of 6.05 × 10^8 Pa, storage modulus of 8.703 × 10^9 Pa and damping parameter of 0.142. The corresponding SHS governing parameters are: heater energy of 26.32 J/mm^2, layer thickness of 0.1 mm, feedrate of heater 3.5 mm/sec, and feedrate of printer 116.38 mm/min, respectively as shown in desirability ramp function (Figure 5). In the desirability ramp function, the reflection of parameter setting is denoted through dot and the amount of desirability is designates as height of the dot [24].

The effectiveness of proposed approach has been validated by performing confirmation experiments. The confirmation experiments were conducted by using the optimal governing parameters obtained through desirability optimization approach. The comparison of experimental and predicted viscoelastic properties are presented in Table 6. The confirmation trial results indicate good agreement among the desirability predicted and experimental response values with an average error of 1.81% for loss modulus, 2.17% for storage modulus and 4.69% for damping parameter. Hence, the proposed approach can be used to estimate the viscoelastic properties of SHS fabricated parts.

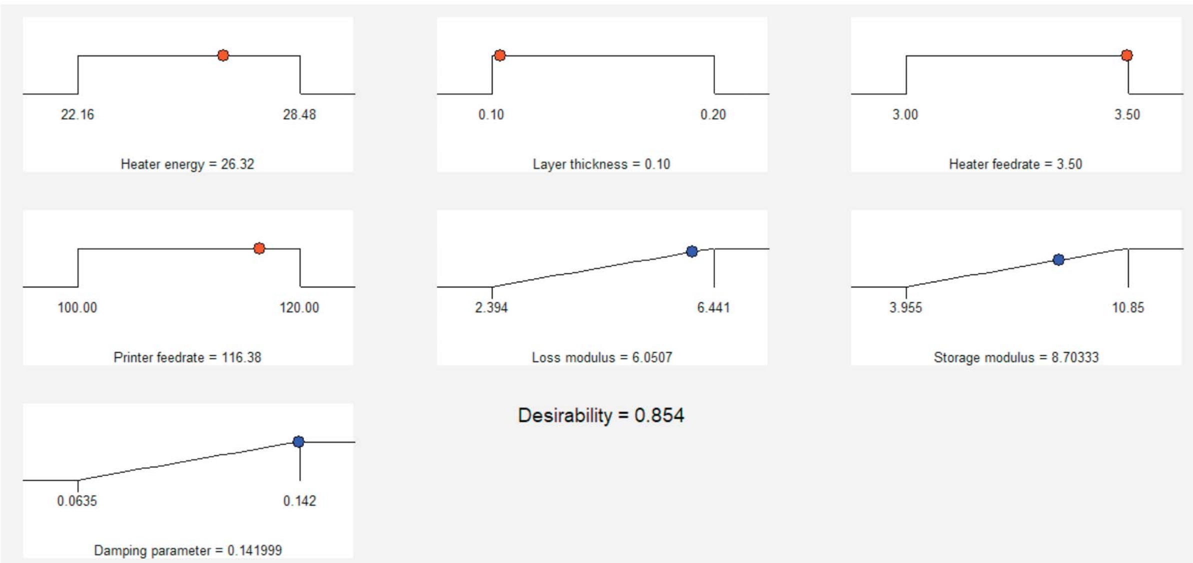

Figure 5. Desirability ramp function for multi-response optimization.

Table 6. Comparison of viscoelastic properties predicted by conformation experiment and desirability approach

Parameters				Responses	Predicted	Exp.	Error %
A (J/mm^2)	B (mm)	C (mm/sec)	D (mm/min)	Loss modulus	6.05	6.162	1.81
26.32	0.1	3.50	116.38	Storage modulus	8.703	8.514	2.17
				Damping parameter	0.142	0.149	4.69

5. Conclusions

The viscoelastic properties of HDPE parts fabricated using developed SHS system with respect to various governing parameters are examined. The conclusion drawn from the results obtained are summarized as follows:

- Based upon Box-behnken design of RSM, twenty nine experiments are conducted and corresponding loss modulus, storage modulus and damping factor are determined.
- The analysis of variance results indicated that layer thickness and heater feedrate are found to be more influencing parameters for selected responses. The proposed regression models for loss modulus, storage modulus and damping parameter are found to be adequate with experimental result within the 95% of confidence level to predict the responses accurately within the limits of governing parameters considered.
- Improved viscoelastic properties of sintered parts has been obtained at the following optimal parameters: heater energy of 26.32 J/mm^2, layer thickness of 0.1 mm, heater feedrate of 3.5 mm/sec and printer feedrate of 116.38 mm/min.
- The confirmation experimental results are found to be in conformance with the predicted optimal parameters with an average error of 1.81% for loss modulus, 2.17% for storage modulus and 4.69% for damping parameter, respectively. Therefore, the proposed approach can be efficiently applied to estimate the viscoelastic behaviour of SHS parts.

Acknowledgement

The authors gratefully acknowledge the financial assistance received from ARMREB, Defence Research and Development Organization (DRDO), Government of India to carry out this research work under grant no. (ARMREB/MAA/2015/167).

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Manuscript Received: Jan. 14, 2019
Accepted: May 16, 2019