STATISTICAL EVALUATION OF ENERGY DENSITY TO OBTAIN POLYAMIDE PARTS MANUFACTURED BY SELECTIVE LASER SINTERING

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Abstract. *Selective laser sintering (SLS) is a rapid manufacturing technology that builds solid objects from particulate materials, layer-by-layer, fusing the particles with laser beam, almost directly from CAD data. The process has many variables that affect directly the mechanical properties of the parts. One of the most important and direct processing parameter is the laser energy density. This work evaluated the effect of the variation of the energy density in the mechanical properties of polymeric material by changing the beam speed and average power. The variables analyzed were stress at 10% of elongation, flexural modulus and density of the samples built with polyamide 2200 (PA2200-EOSINT) using a CO_2 laser (10W). The specimens were build with combined different laser power (2,7; 3,4 and 4,1W) and different scan speed (39,0; 44,5 and 50,0mm/s). To obtain the mechanical properties, the samples were submitted to flexural test. The bulk density was calculated with mass and physical dimensions of specimens. The results analysed by statistical methods indicated that the laser power had more influence over the density and the mechanical properties of the polyamide sample than the scan speed.*

Keywords: *selective laser sintering, polyamide, processing parameters, rapid manufacturing, prototyping*

1. INTRODUCTION

The design and engineering of new products have been using Rapid Prototyping (RP) technologies to perform evaluations during the development cycle. These technologies provide quick and accurate prototypes in different type of materials depending on the process. The development of the technologies and materials had improved and nowadays it is possible to produce parts directly from CAD data without the need of tooling and setup (Hopkinson et al, 2005).

An important and popular technology is Selective Laser Sintering (SLS). SLS can build parts in polymers, ceramics and metals. The process uses a laser that sinters selectively a thin layer of powder spread over a moving platform. Each time that a layer is finished, the platform is lowered and a new layer of powder is spread over the previous layer built. The computer controlled laser scans over the layer sintering again a new layer of the part, attaching it the previously layer built. The process continues until the part is complete. Few post processing steps are required to clean the part depending on the application requirements (Jacobs, 1996).

There are many parameters that are necessary to control in order to successfully obtain SLS parts depending on the type of material under processing. For instance, the laser indicated to sinter metals is different from the laser used to sinter ceramics and polymer as the wavelength must interact differently with the atoms, crystalline structure or polymer chain. In the case of polymers, the CO_2 infrared laser is commonly used in the continuous mode. So, the main variables of the laser system in the process are: the average laser power, the scan speed and spot diameter. These three variables determined the amount of energy directed to the powder over the platform. This energy density can be calculated by Eq. (1), where ρ_e (J/mm^2) is the laser energy density, P (W) is the laser power, v (mm/s) the scan speed and d is the spot diameter (mm) over the power surface (Steen, 1991).

$$\rho_e = P / (v.d) \qquad (1)$$

During the process, the polymer powder must be heated few degrees below its melting temperature (for crystalline polymer such as polyamide, Caulfield et al, 2007). Ideally, the laser should only sinter the unsintered powder of the new layer and bond it to the previous layer built as seen in Fig. 1 (A). According to Hardro et al (1998), if the energy density is low (insufficient laser power or too fast scan speed) it can cause poor bonding between the layers making weak and easy to delaminate parts (Fig. 1 (B)). The energy delivered by the laser must be controlled to only sinter the necessary amount of material. In case of excessive power or too slow speed it might cause the layers to warp (Fig. 1(C)). Warping make deformed parts and in critical cases it blocks the recoating of new layers and crashes the process. Another consequence of not adjusted combination of properties might cause balling presented in Fig. 1(D). Balling can be caused in case of high power and too fast scan speed.

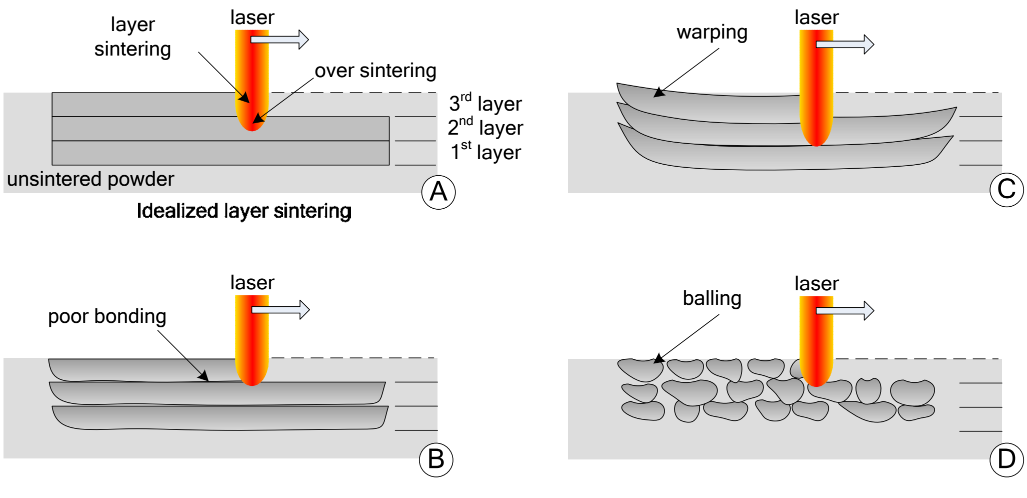

Figure 1. Different effects of the laser energy density variation.

From the point of microstructure analysis, the energy density also affects the bonding between the powder particles. The higher the energy the lower the porosity, higher the shrinkage and higher mechanical properties might be achieved (Fig. 2). Nevertheless, as commented previously, too high energy might cause undesirable results during the part construction. Also, excessive energy might degrade the polymer decreasing its properties.

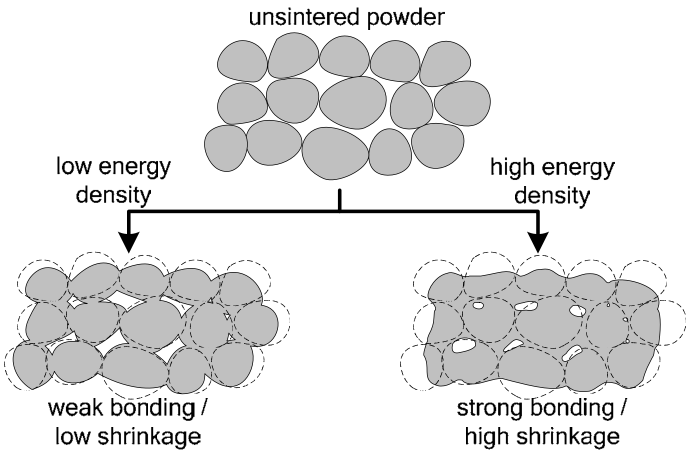

Figure 2. Bonding between the powder particles depending on the energy density.

As it is important for rapid manufacturing to control the mechanical properties, it is necessary to control the processing parameters based on the characteristics of the equipment. In this work, the variation of the laser energy density was the studied. By combining different levels of laser power and scan speed it was evaluated their individual influence over some properties of polyamide PA2200 from EOS. Using design of experiments methodology, experiments were performed to determine the better combination of power and speed for the SLS machine under development. The objectives were to increase density and to analyse the stress at 10% elongation and the elastic modulus considering the power and speed combination.

2. MATERIALS AND METHODS

To perform the experiments, small slabs of the subject material were manufactured with approximate dimensions of 35,0x5,0x1,45mm. The polymer used in this study was fine polyamide PA2200 from EOS. The average grain size was 60µm, obtained by laser diffraction (EOS, 2006). The PA2200 had a melting temperature of 177ºC, obtained by differential scanning calorimetry analysis (DSC).

The specimens were manufactured using a prototype SLS machine with the system elements as presented in Fig. (3). To improve the accuracy and avoid distortion during the building process, infrared lamps and heaters were used to keep the temperature of the unsintered powder and sintered layers around 140ºC. A sliding hopper was used to spread powder over the platform to add new layers. Two mirrors controlled the scanning over the surface of the powder bed, selectively sintering the programmed layer. A dioxide carbon laser unit was used with a nominal power of 10W. The lenses provided a laser beam focus of 250µm and the machine was capable of producing parts with average layer thickness of 200µm.

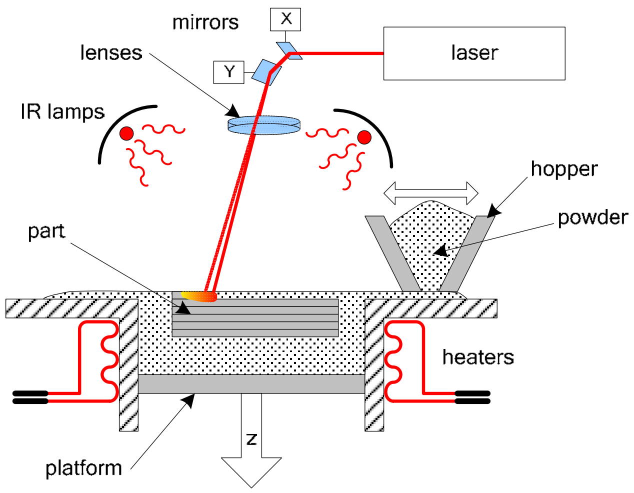

Figure 3. Schematic representation of the prototype SLS machine used in the experiments.

The experiment was designed to find the best combination between laser power of 2,7; 3,4 and 4,1W and scan speed of 39,0; 44,5 and 50,0mm/s. A factorial multi-level design was planned to evaluate the inputs: power and speed; and outputs: density, stress and 10% strain and flexural modulus. The experiment was randomized and a summary of the experiment is shown in Table 1.

Table 1. Statistical summary of the experiment.

Class of Project			Factorial multi-level	
Experimental Factors			2	
Responses			3	
Number of runs			36	
Degrees of freedom			27	
Randomized			yes	
Confidence interval			95%	
Input factors	Laser power	Low	2,7	W
		Mid	3,4	
		High	4,1	
	Scan speed	Low	39,0	mm/s
		Mid	44,5	
		High	50,0	
Output factors	Density			g/cm^3
	Flexural Modulus			MPa
	Stress at 10%			MPa

The volumetric density was calculated based on the measured mass and dimensions of the specimens. To obtain the mechanical properties, it was used a dynamic mechanical analysis equipment (DMA Q800, TA Instruments). The test performed was single cantilever test. The applied force had a rate of 2N/min and all samples were tested at 30^{o}C.

3. RESULTS AND DISCUSSION

The planned experiment produced 36 specimens. In Fig. (4), four specimens are shown and no visible difference in surface finishing, colour or geometry form was noticed.

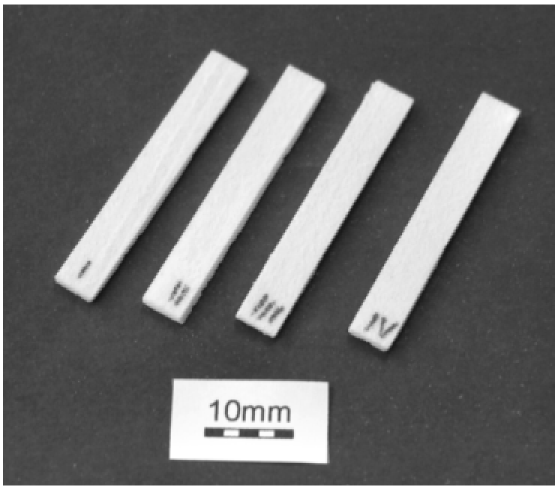

Figure 4. Polyamide specimens obtained during the experiments.

3.1. Analysis of variance (ANOVA)

Table 2, 3 and 4 show the analysis of variance for each output variable measured in the experiment. For each P-Value that was less than 0,05, at 95% of confidence interval, it represented that the source had strong influence over the result.

Table 2. ANOVA for density.

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
A:Laser Power	0,058115	1	0,058115	90,59	0,0000
B:Scan Speed	0,0567454	1	0,0567454	88,45	0,0000
AA	0,0242367	1	0,0242367	37,78	0,0000
AB	0,000150063	1	0,000150063	0,23	0,6322
BB	0,00553001	1	0,00553001	8,62	0,0063
Total error	0,0192464	30	0,000641546		

Table 3. ANOVA for Flexural Modulus.

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
A:Laser Power	38929,8	1	38929,8	72,92	0,0000
B:Scan Speed	41275,9	1	41275,9	77,31	0,0000
AA	27753,7	1	27753,7	51,98	0,0000
AB	1413,76	1	1413,76	2,65	0,1141
BB	183,361	1	183,361	0,34	0,5622
Total error	16017,1	30	533,902		

Table 4. ANOVA for Stress at 10%.

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
A:Laser Power	832,728	1	832,728	20,19	0,0001
B:Scan Speed	37,575	1	37,575	0,91	0,3475
AA	275,069	1	275,069	6,67	0,0149
AB	17,1603	1	17,1603	0,42	0,5238
BB	223,767	1	223,767	5,42	0,0268
Total error	1237,46	30	41,2486		

In Table 5, a summary of the significant and predominant factors for each result is presented. The predominant factor was indicated by the strongest effect over the results. It can be seen that laser power had more impact on density and stress at 10%. The beam scan speed was the predominant factor for the flexural modulus.

Table 5. Summary of analysis of variance (ANOVA).

Properties	*Significant factors*	*Predominant factor*
Density	A: Laser Power	Laser Power
	B: Scan speed	
	AA	
	BB	
Flexural modulus	A: Laser Power	Scan speed
	B: Scan speed	
	AA	
Stress at 10%	A: Laser Power	Laser Power
	AA	
	BB	

3.2. Response surfaces

Based on the combination of the laser power and scan speed over the results, it was possible to obtain regression coefficients to represent by an equation the phenomena. The equation was used to plot the estimated response surfaces of the results based on the interaction between laser power and scan speed.

In Fig. (5), the estimated response surface for the volumetric density is presented. The best optimized combination to achieve higher density (0,732g/cm^3) was at lower scan speeds (39,5mm/s) and relative high laser power (3,72W) demonstrated by the peak in the surface in Fig. (5). For this peak, the energy density was 0,37J/mm^2. The speed had less influence in the results than the laser power. In Lu et al (2001), the authors also agree that the laser power and powder bed temperature had more influence over the laser sintering results than the laser scan speed in the manufacturing of full dense parts.

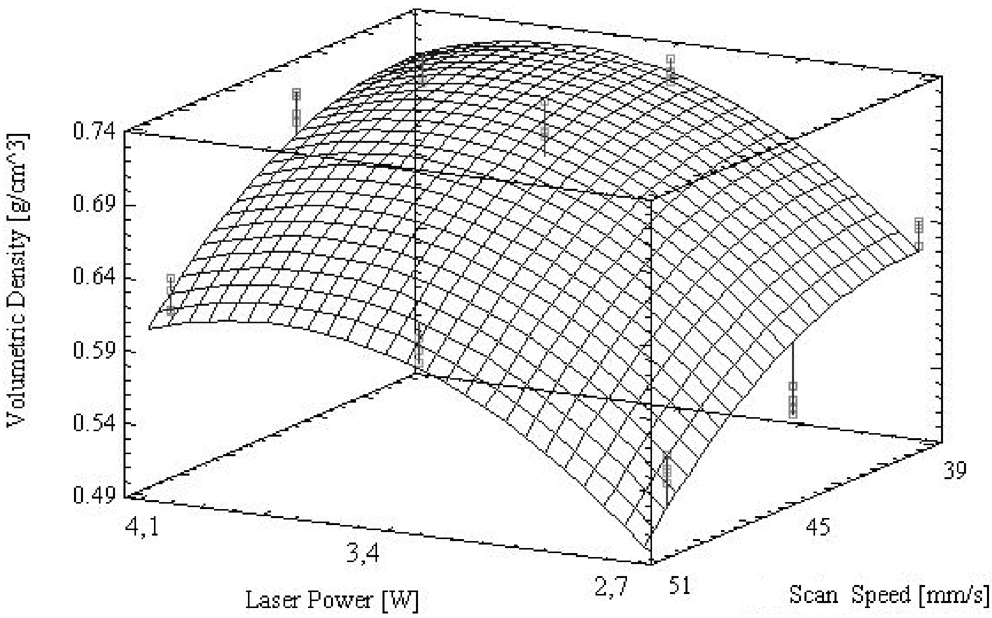

Figure 5. Estimated response surface for volumetric density.

The estimated response surface for the flexural modulus is presented in Fig. (6). In this case, the scan speed had strong influence over the results. It was acquired that a speed of 39,0mm/s and laser power of 3,69W would produce stiffer parts (361,7MPa). For this combination of speed and power the energy density was 0,37J/mm^2. This influence might be related to the time exposure of the material to the heat source that might have affected the kinematics of the laser sintering process. The response surface indicates that at lower speeds the material might become even more rigid. Although lower speeds had caused an increment in the flexural modulus, the laser power showed had a slightly reduction. The estimated response surface showed that to achieve higher flexural modulus a combination of lower scan speed and laser power have to be further studied.

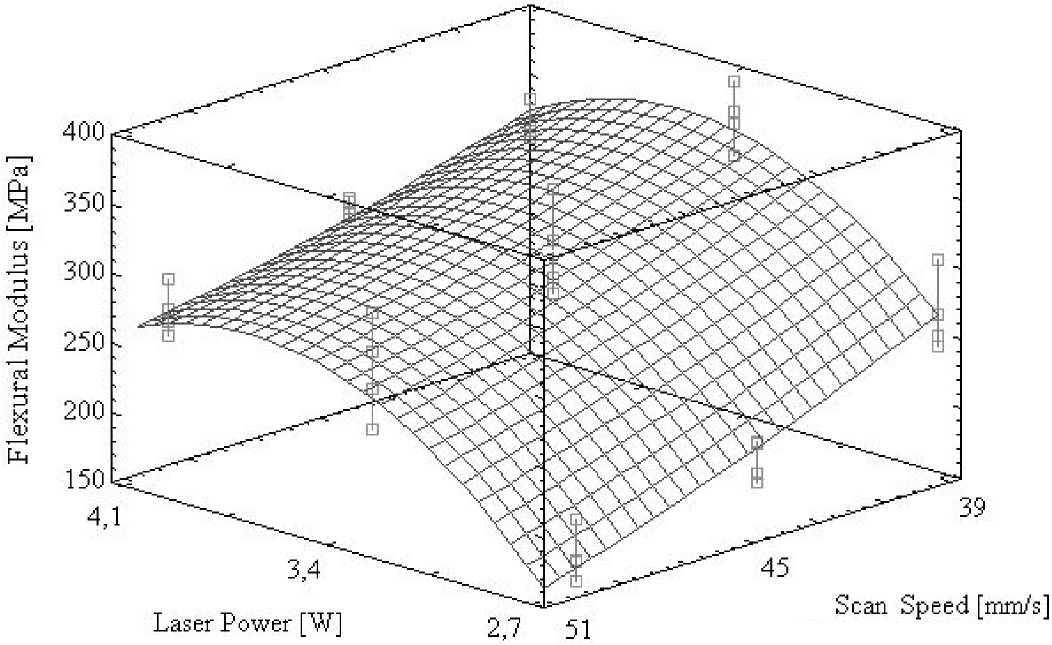

Figure 6. Estimated response surface for flexural modulus.

The stress measure at 10% strain of the material had similar behaviour to the density results. The estimated response surface built based on the measured stress is shown in Fig. (7). The optimum value of stress, 50,2MPa, was obtained by a combination of scan speed of 44,0mm/s and laser power of 3,78W. The energy density to the maximum stress obtained was 0,34J/mm^2.

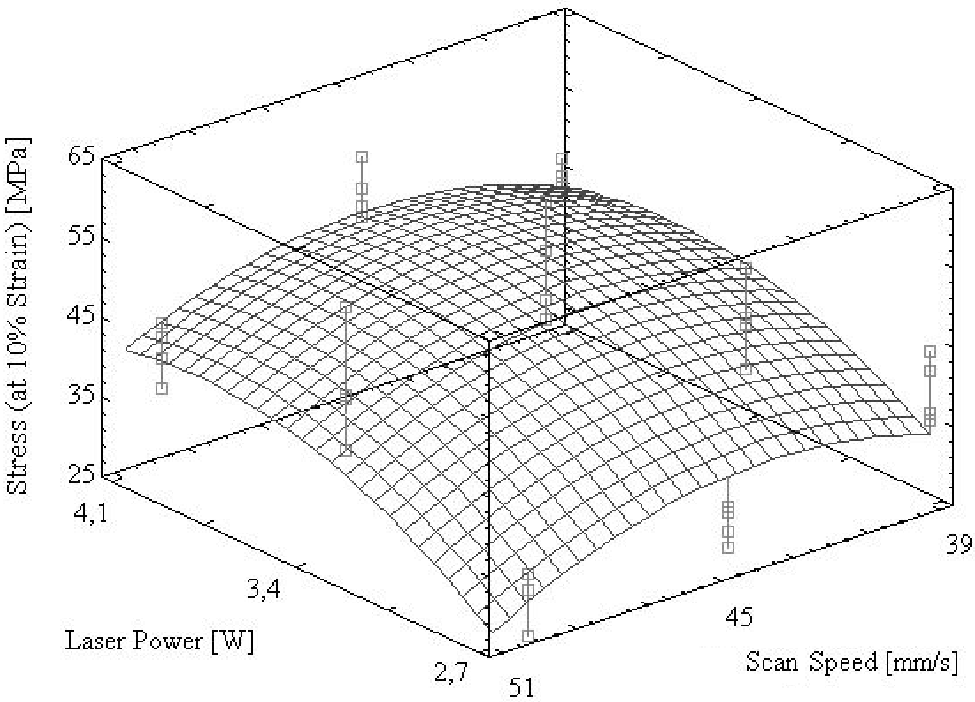

Figure 7. Estimated response surface for stress at 10% strain.

3.3. Multiple response optimizations

The use of SLS parts can be spread in different applications. By doing the multiple response optimizations, it is possible to tailor the material of the part. The multiple response optimization is simple the overlapping of the curves from the estimated response surfaces obtained for each previous presented result.

In most usual applications for rapid manufacturing or prototyping, it is required dense parts with maximum flexural modulus and stress. This case is shown in Fig. (8), where the peak of the curve is the best combination of scan speed and laser power to obtain dense, stiff and strong parts. The scale of desirability is a number, from 0 to 1, that represents how far or close is the optimization from the desired combination of the three response variables.

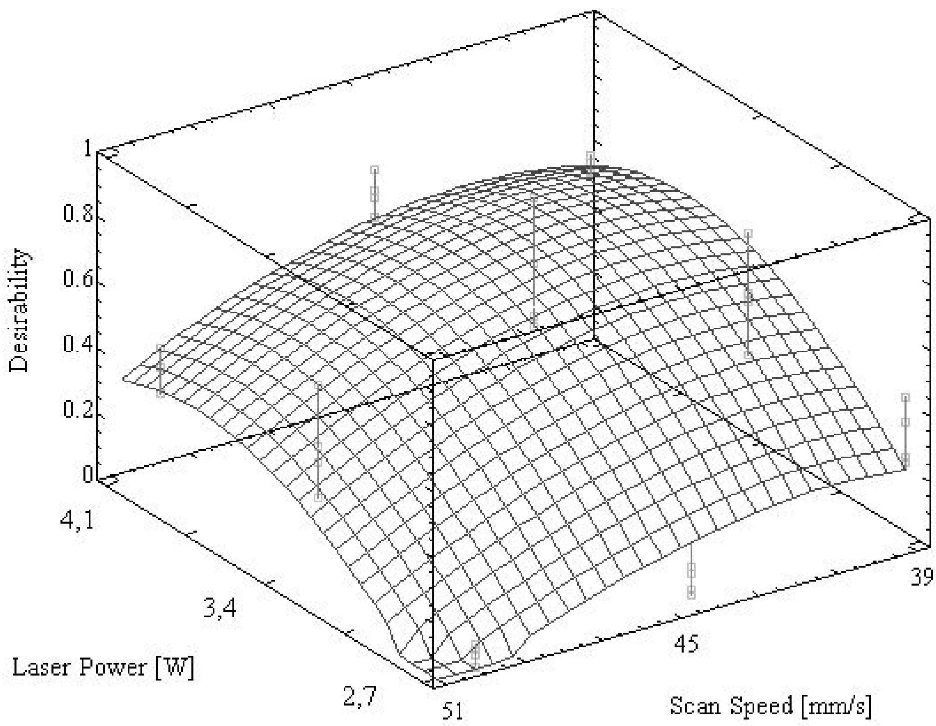

Figure 8. Estimated desirability surface for strong, stiff and dense material.

Nevertheless, other applications might not require dense parts. In manner of fact, applications like tissue engineering and time controlled drug delivery capsules can require both dense and porous materials in the same part (Mironov et al, 2003; Wu et al, 1996). For this kind of applications, the optimization is represented by the estimated response surface shown in Fig (9). The complex shape was obtained as it was more difficult to obtain maximized stress and flexural modulus with low density. As the low density was obtained by higher amount of pores inside the material it is difficult to maximize the other two properties as the internal porous structure reflects in the section area of the samples.

In Table 6, the summary of the multiple response optimizations is presented. In the case of stiff, strong and dense parts, the desirability achieved was 0,69. On the other hand, the desirability for strong and stiff but porous parts was 0,43. Comparing both cases, the scan speed was the factor that had higher degree of changes. Care must be taken to not analyze the multiple response optimizations isolated from the ANOVA of each response.

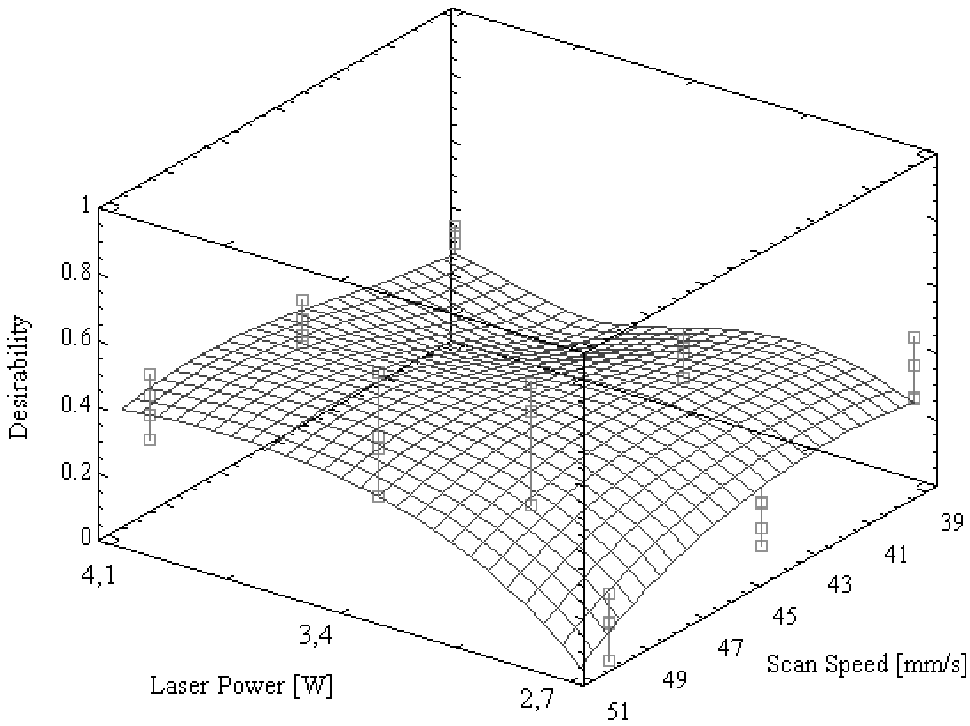

Figure 9. Estimated desirability surface for minimum density (porous material) with maximum stress and flexural modulus properties.

Table 6. Summary of the multiple response optimization.

	Target: • Maximum Density • Maximum Flexural Modulus • Maximum Stress		*Target:* • Minimum Density • Maximum Flexural Modulus • Maximum Stress
Factor	*Value*		*Value*
Laser Power	3,71W		3,82W
Scan speed	41,9mm/s		48,9mm/s
Energy density (*)	0,35J/mm^2		0,31J/mm^2
Response	*Optimum Value*		*Optimum Value*
Density	0,728g/cm³		0,656g/cm³
Flexural Modulus	337,1MPa		289,7MPa
Stress at 10%	49,3MPa		46,2MPa
Desirability	0,69		0,43

(*) calculated.

4. CONCLUSIONS

The objective of this work was to study the effect of the laser power and scan speed over the material properties of the polyamide PA2200. It was identified that laser power had great influence over in the results to increase density, flexural modulus and stress at 10% strain obtained by the single cantilever test. Nevertheless, the scan speed also had great influence and the combination of speed and power must be taken with care to obtain the desired results.

In the case of density and flexural modulus, despite the fact that the energy density was the same, the results were different. The flexural modulus results required lower speed and power to achieve higher stiffness in the material. This indicates that the kinematics of the laser sintering affected the process. The time and intensity of the exposure affected the grow rate of the necks between the polyamide particles. Further microstructure analysis must be investigated in order to study the pore formation and the bonding between the layers.

It is also important to notice that the results obtained in the machine used in the experiments might be different from the results obtained on commercial systems. The apparatus used in this work was an experimental machine and still need improvements such as controlled atmosphere chamber and precise heat control.

5. ACKNOWLEDGEMENTS

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