MANUFACTURING AND POST-PROCESSING PARAMETERS EFFECT IN THE CURE SHRINKAGE OF STEREOLITHOGRAPHY PARTS BUILT WITH THE RESIN DSM SOMOS 7110

Gean Vitor Salmoria
Universidade Federal de Santa Catarina, Depto. Engenharia Mecânica, CIMJECT-LABMAT
gsalmoria@cimject.ufsc.br

Valter Estevão Beal
Universidade Federal de Santa Catarina, Depto. Engenharia Mecânica, CIMJECT
beal@cimject.ufsc.br

Alfredo Tibúrcio Nunes Pires
Universidade Federal de Santa Catarina, Depto. de Química
pires@qmc.ufsc.br

Carlos Henrique Ahrens
Universidade Federal de Santa Catarina, Depto. Engenharia Mecânica, CIMJECT
ahrens@cimject.ufsc.br

Abstract. *The cure state of an object built by stereolithography depends on several factors such as laser power, building parameters and resin photosensivility. It is normal to find objects built by stereolithography that are in an intermediated degree of cure. They require post-cure processes such as ultraviolet radiation or thermal treatment to improve the thermal, mechanical and chemical properties of stereolithography resins. The degree of cure can be adjusted by geometric factors in the numerical program of the machine or by phisical/chemical resin properties adjustments. This work describes a calorimetric and dilatometric study of parts built by stereolithography with the resin DSM SOMOS 7110 using different line hatch spacing during the manufacture and methods of post-cure such as ultraviolet radiation, microwave irradiation and conventional heating. The results indicated that the use of line hatch spacing of 0.15mm and 0.10mm in the manufacturing with DSM SOMOS 7110 induced to an heterogeneous cure process concerning the internal resin structure and specimens surfaces. Cure shrinkage was remarkable in these green parts. The green parts built using hatch spacing of 0.05mm were in advanced states of cure showing no post-cure shrinkage when the parts were heated. The post-cure processes, especially the thermal treatment, improve the cure state of green specimens built using line hatch spacing of 0.10mm, minimizing the anisotropy inherent to this manufacturing method. This permits a better control over the dimensional behavior of SOMOS 7110 parts.*

Keywords. *Stereolithography, Rapid Prototyping, Shrinkage, Photopolymer.*

1. Introduction

Nowadays, designers and engineers often use modern technologies to evaluate their product design, as Rapid Prototyping technologies, used to build faster and better prototypes. Stereolithography (SL) is a rapid prototype manufacturing process that uses a ultra-violet (UV) laser beam to solidify liquid resins layer-by-layer. These liquid resins are based on monomers and photoinitiators that are activated by the UV radiation of the laser beam. Once a resin is exposed to the UV radiation, a localized chain process starts polymerizing the resin. The resin changes from liquid to solid state generating and sequentially gluing layers to build an object directly from 3D computer data (Jacobs, 1992 & 1996).

Since photo-polymerization degree is a function of several parameters related to the resin composition and build parameters, it is very difficult to obtain a fully cured part after the laser scanning. Usually, green parts (after building in the SL machine) reach conversions about 50 to 80% of cured degree. Post-cured parts (UV chamber, oven and microwaves) may reach up to 90% of cure (Bernhard et al, 1991). For prototypes built with urethane acrylic-based resins it can result in low strength and excessive shrinkage (Fuh et al, 1997 & Wiedemann at al, 1995). Therefore, it is essential to understand the influence of key operating parameters on the curing degree of the photo-curable resin. Otherwise it will not be possible to build SL prototypes with the accuracy and good mechanical properties required by industry.

The polymerization rate of a SL resin is associated to the laser exposure required to achieve cure depth (C_d). The cure depth is defined by the resin parameters: critical laser exposure (E_c), maximum radiation exposure (E_{max}) and deep penetration (D_p), as indicated in Eq. (1).

$$C_d = D_p \cdot \ln \frac{E_{\max}}{E_c} \tag{1}$$

The maximum radiation exposure on the resin (E_{max}) is given by Eq. (2),

$$E_{\max} = \sqrt{\frac{2}{\pi}} \cdot \left(\frac{P_1}{W_0 \cdot V_s} \right) \tag{2}$$

where P_l is the current laser power been applied over the resin, W_o is the beam radius, and V_s is the laser scanning speed.

Theoretically, the laser beam focus is considered as having a fixed "energy" diameter that can polymerize the resin (E_c). This diameter generates a solid bullet-line shape having a penetration (C_d) and thickness (L_w) value. The (C_d) forms and bonds the new layer to the previous layer built. The laser interaction movement and the gaussian energy distribution of the laser are presented in Fig. (1) (Jacobs, 1992 & Fuh et al, 1997).

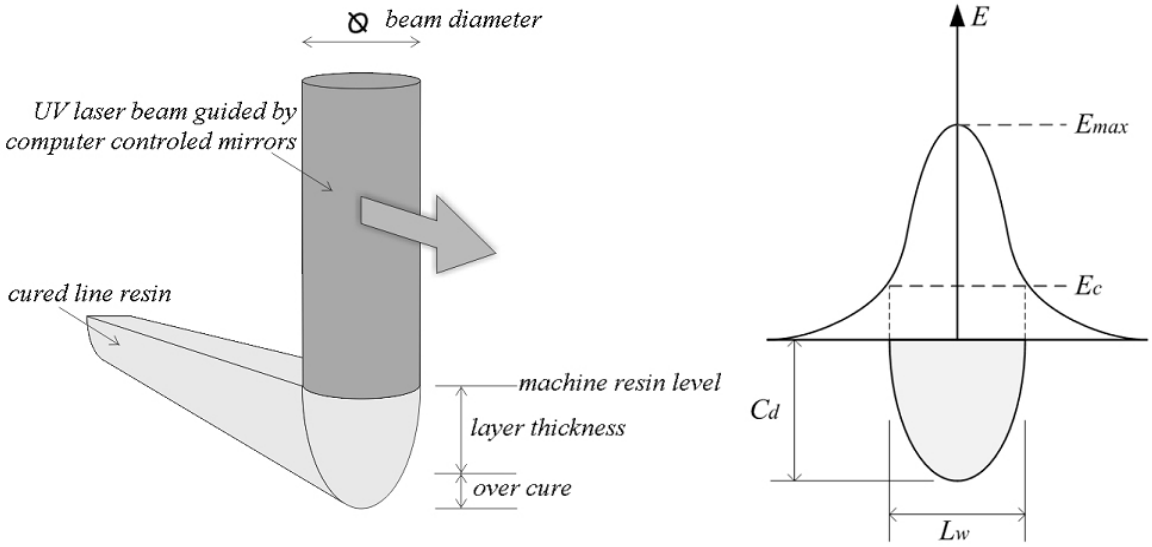

Figure 1. Energy gaussian distribution and the bullet-line shape generated by the laser beam radiation.

Due the parabolic shape of the cured resin, it is well known that regions of uncured resin can exist in the microstructure of the SL parts as presented in Fig. (2). The uncured regions formed between each successive line result in a non-homogeneous structure (Jacobs, 1992 & Fuh et al, 1999). Increasing laser power or slowing scanning speed affects energy density resulting in bullet-lines with greater cure depth. As a result, the resin structure changes to a more cured state with less trapped uncured (liquid or partially cured resin) inside the part. Other alternative is to decrease layer thickness or line hatch spacing (hatch spacing is the distance between each bullet-line that is used to solidify a layer). This procedure induces an over-curing of the previous layer and/or line and also results in higher degrees of polymerization in these regions (Wiedemann at al, 1995).

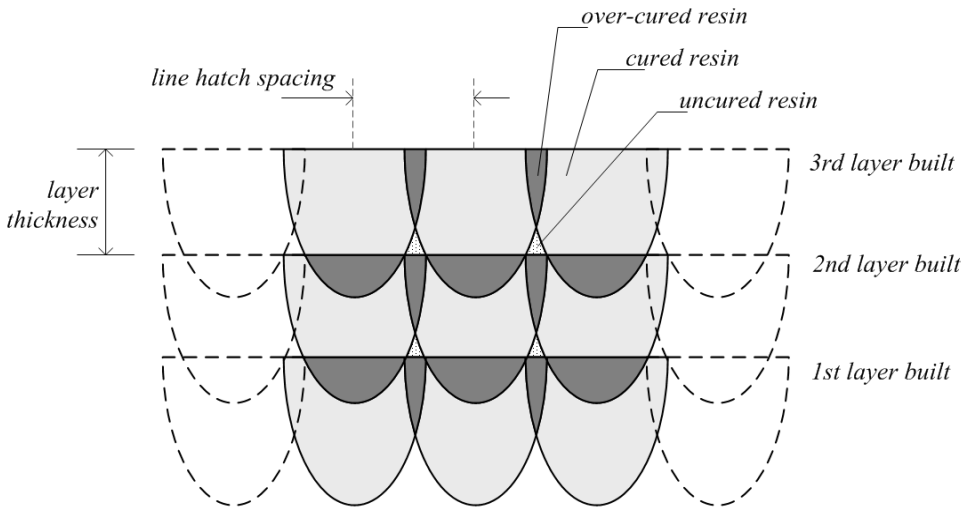

Figure 2. Section of the microstructure of SL objects showing cured and uncured regions.

If the SL resin is not fully-cured the shrinkage increases during post-processing operations such as UV chamber and thermal treatments. This is proportional to the amount of partially uncured resin. The volume reduction of the uncured resin regions is desirable to minimize the post-process shrinkage, preserving the overall accuracy of the SL part.

Due to the polymerization, the shrinkage can reach values up to 8% of the volume. The main part of polymerization shrinkage occurs while the material is still liquid. Only a small part of the shrinkage takes place at higher degree of polymerization, i.e. during post-processing. The presence of different curing degrees in the green-state prototype can cause a disproportional shrinkage resulting in internal stress, the reason for distortion and low strength (Fuh et al, 1999 & Dusel et al, 1996).

In the formulations of photo-polymerizable resins, monomers, oligomers, photoinitiators and additives are used in combination to achieve specific desirable properties. The first resins used in stereolithography were the acrylic based type and their properties were poor in many aspects. Studies have been undertaken on photo-polymerization for stereolithography applications aimed to increase the polymerization rate, cure degree and part accuracy (Jacobs, 1996 & Wiedemann at al, 1995).

The stereolithography process has been applied to different types of multifunctional resins to produce highly crosslinked polymer materials (Decker, 1999). Most SL resins still contain the very reactive acrylate group which polymerizes by a radical mechanism (Segurola et al, 1999). Epoxy monomers that polymerize by a cationic mechanism (Decker, 2001) are also employed in some resins. Epoxy and acrylates are desired ingredients in photo-polymerizable resin formulations as they provide chemical resistance, mechanical strength and part accuracy (Jurczak).

This paper analyses two influences over the polymerization degree for the stereolithography resin DSM SOMOS 7110 and their effect in the shrinkage behavior of SL parts. The first analyzed influence is the line hatch spacing parameter related to the building strategy. The second influence that is analyzed is the post-processing techniques that are used to increase the cure degree of the resin. To perform the experiments, small specimens under different hatch spacing and different post-cure treatments were made. The specimens linear dimensional variation under heating cycles, their hardness and thermograms are analyzed and discussed.

2. Methodology

2.1. Specimens Design, Programming and Manufacturing

To observe the shrinkage behavior over stereolithography parts, small specimens were designed in a CAD (computer aided design) system and built in a stereolithography machine model 3D System SLA250/30A. Figure 3 shows the specimens dimensions and the different orientations according to the layer manufacturing plane.

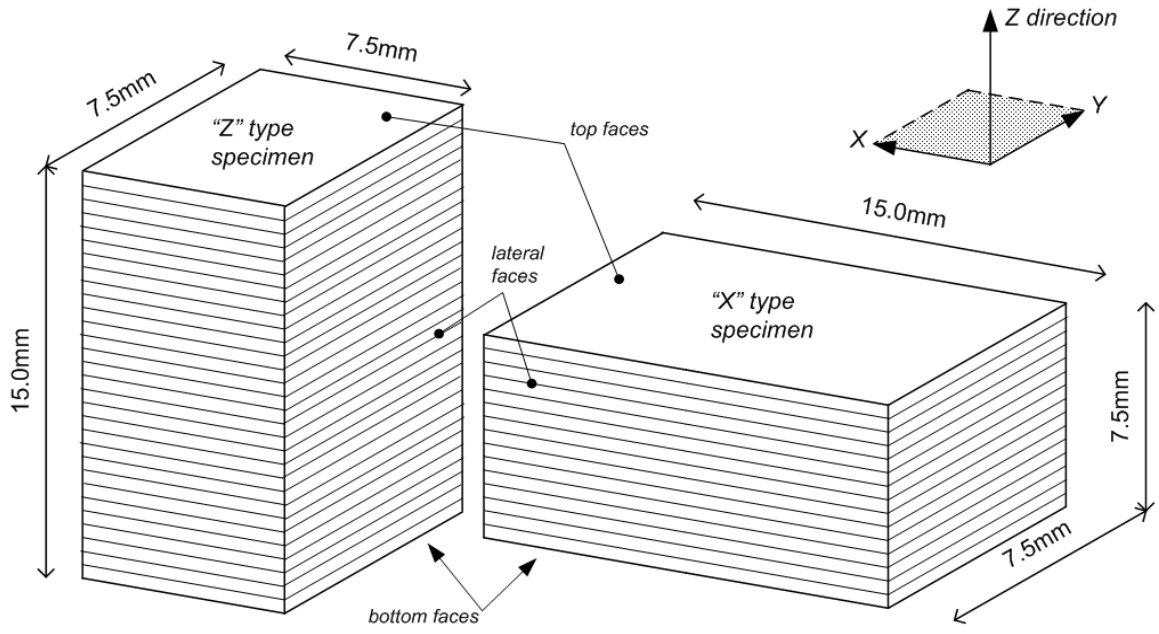

Figure 3. Specimen's dimensions and orientation (note that layers are hypothetically illustrated).

The layer thickness used to build the specimens was 0.150mm as this model of machine does not support layer thickness adjustments. To analyze the influence of the degree of cure over the resin, different line hatch spacing at the CAM (computer aided manufacturing) system were applied. As seen on Fig. (2), three values were used to build the specimens: 0.05mm, 0.10mm and 0.15mm. The line hatch space of 0.10mm is the default value recommended by the resin manufacturer to the machine model and laser type used.

During the manufacturing of the specimens, the average laser power of the machine was 40mW (Helium-Cadmium laser unit, with 325nm of wave lenght), scanning speed of 800mm/s (the numerically controlled machine automatically adjusts the scanning speed under laser power variations), and a laser beam focus diameter of 0.20±0.01mm. The DSM SOMOS 7110 SL resin, used to build the specimens, is an epoxy-based one, composed by epoxies, acrylates, polyols, initiators and additives. This resin presents Ec = 8.2mJ/cm2 and Dp = 0.140mm.

2.2. Post-cure Processes

To analyze the influence of different post-cure processes, three different alternatives were applied to the specimens. The first alternative to cure the specimens was the ultraviolet radiation. So, during 1 hour inside a UV chamber (300W) specimens were placed. Other alternative was to submit specimens under microwave radiation in a Toshiba (700W) microwave oven during 4 minutes. The third alternative to post-cure specimens was by conventional heating. In this procedure, the specimens were submitted to an isothermal treatment at 125^{o}C during 30min. Table (1) resumes all experiments and specimens that were built to perform this investigation.

Table 1. Resume of the manufactured specimens and experiments applied.

Influence	Parameters	Manufacturing orientation	
		"Z" type specimen	"X" type specimen
Line hatch space (UV chamber, 1hour)	0.05mm	1	1
	0.10mm	1	1
	0.15mm	1	1
Post-process (line hatch space 0,10mm)	None (green)	1	1
	UV chamber, 1hour	1	1
	Microwaves, 4 min.	1	1
	Thermal 125^{o}C, 30min	1	1
		Total: 12 specimens	

2.3. Experimental Procedures

To analyze the degree of cure of each different specimen four experiments were performed: scanning electronic microscopy (SEM), differential scanning calorimetry (DSC), dilathometry and hardness measurements.

The SL specimens surfaces were inspected in a scanning electron microscope (SEM) Philips XL30 in order to investigate the morphology of it different faces. To make possible the SEM investigation the surfaces were coated with gold in a Bal-Tec Sputter Coater SCD005.

DSC thermograms of the specimen surfaces were obtained on a Shimadzu DSC50 in dynamic heating mode using Nitrogen flow-rate of 25ml/min. Samples of 10mg were submitted to a heating rate of 10^{o}C/min over a temperature range from 25 to 300^{o}C inside aluminum pans. The DSC analyses were used to determine the residual heat of reaction after curing and post-curing.

The Shore A hardness of the specimen surfaces was determined using a Wultest SD 300 apparatus. The dimensional behavior of the specimens prepared was investigated using a dilatometer model BP Engenharia. The linear variation of dl/l_o was recorded as function of temperature in the widest dimension for all specimens (see Fig. (3)). The analyses were performed from 25 to 150^{o}C (heating rate of 5^{o}C/min) with Nitrogen flow-rate of 25ml/min.

3. Results and Discussion

The micrographs obtained by scanning electronic microscopy of specimens built by stereolithography using the default parameters (line hatch spacing of 0.10mm) showed that each face of SL parts presented different aspects. Figure (4) shows the micrographs of the lateral face of a SL specimen. It is possible to remark the different roughness of the top and bottom surfaces viewing by side angles in these images.

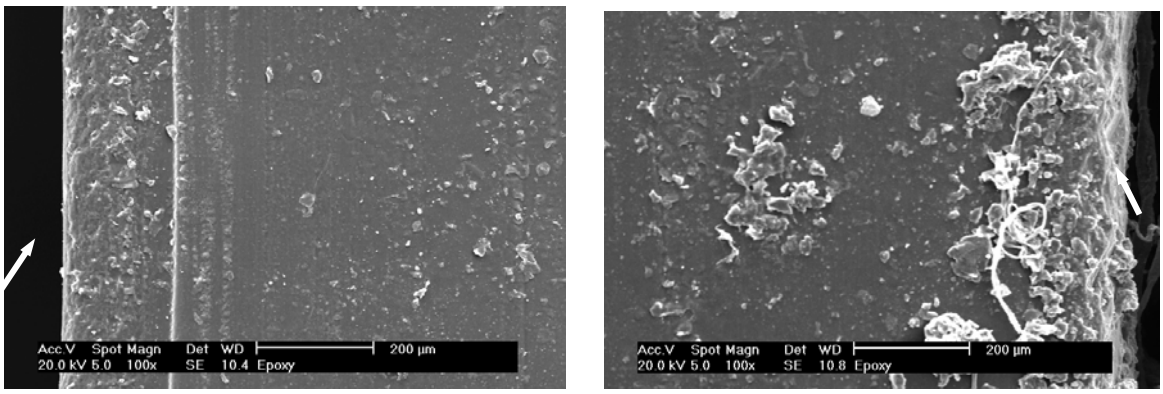

Figure 4. Micrographs of the lateral faces of a SL specimen showing the roughness close to top (left) and to bottom (right) faces (indicated by the arrows).

Micrographs of the top and bottom faces are presented, respectively in Fig. (5). The top face presents a very smooth surface due to the recoating process. The stereolithography is a rapid prototyping manufacturing process which concerns the manufacturing of parts using photo-polymerization layer-by-layer; consequently, the part structure presents characteristics related to its manufacturing distinctiveness. It is difficult to obtain a smooth surface in faces that are orientated to the bottom side of the building platform. The roughness is higher in this surface when compared to the others faces.

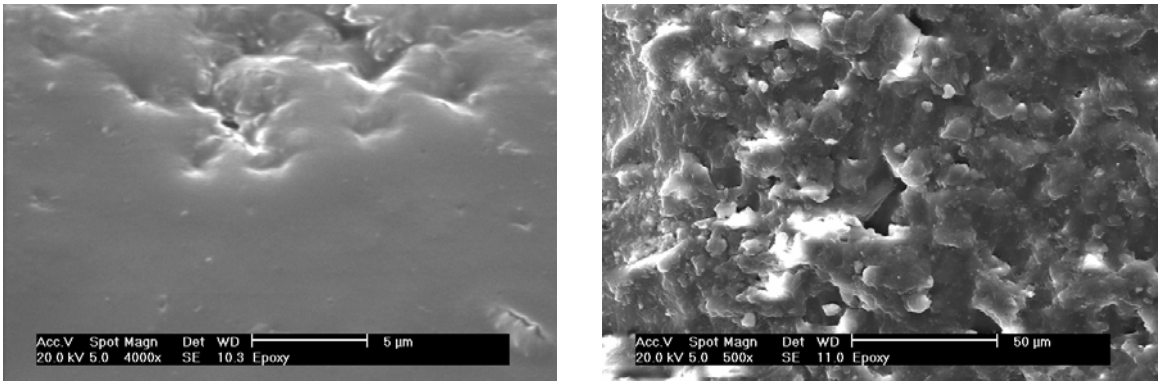

Figure 5. Micrographs of the top face (left) and the bottom face (right) of SL specimens.

The use of DSC analysis in the study of the degree of cure of SL resin has been described as a useful technique by several authors (Fuh et al, 1999 & Colton et al, 1999). DSC thermograms for different faces of a green specimen are shown in the Fig. (6). The exothermic peaks at 95^{o}C and 180^{o}C revels the energy releasing by the uncured resin regions, i.e. the cure enthalpy. However a single DSC exothermic peak related to one overall cure reaction is most often observed in thermoset curing. There are other examples of the two DSC peaks revealing two cure reactions for thermosets materials (Wingard, 2000).

For the green specimens built using the default parameters (0.10mm of line hatch spacing), the faces show different states of cure. The top face presents a more advanced degree of cure releasing less energy in the exothermic process (ΔH_c= 10J/g). The lateral face thermogram (B) is in an intermediate state of cure when compared to the top (C) and to the bottom faces (A) curves, as shown in Fig. (6) and Tab. (2). The bottom face presented the greatest energy release (ΔH_c= 40J/g), indicating a less cured state. The energy released and hardness of each surface for the investigated specimens are presented in Tab. (2).

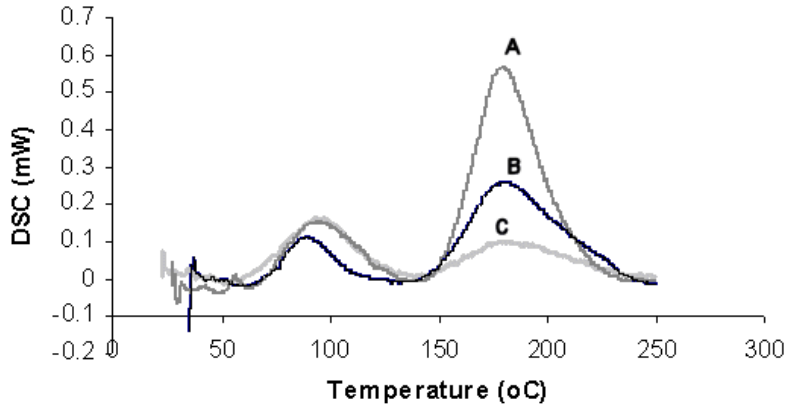

Figure 6. DSC thermograms of the bottom (A), lateral (B) and top (C) faces of the SL green specimens built with the default parameters.

Table 2. Values of cure enthalpy and Shore D hardness for the specimens built using different line hatch spacing

Specimens	Top face (C)	Lateral face (B)	Bottom face (A)
	Enthalpy (Hardness)	Enthalpy (Hardness)	Enthalpy (Hardness)
Hatch space **0.05mm**	2.72 J/g (84)	21.74 J/g (81)	32.58 J/g (80)
Hatch space **0.10mm**	4.72 J/g (83)	42.40 J/g (79)	57.25 J/g (77)
Hatch space **0.15mm**	15.83 J/g (82)	54.31 J/g (77)	74.82 J/g (74)

(*) all average values.

The enthalpy related to cure of the uncured resin and the hardness of the faces of the specimens presented in Tab. (2) shows that the use of line hatch spacing of 0.05mm improves the state of cure and the hardness in all specimens faces compared to the specimens manufactured with 0.15 and 0.10mm of line hatch spacing.

The influence of the post-cure method in the energy release by the uncured resin specimens built using the default parameter were availed by the DSC. Also the hardness of the specimens were measured. The results presented in Tab. (3) demonstrated that the post-cure process by thermal treatment at 125^{o}C during 30 minutes was the most efficient method to improve the state of cure and the hardness over all faces.

Table 3. Values of cure enthalpy and Shore D hardness for the specimens post-cured using different methods.

Specimens (line hatch space of 0.10mm)	Top face (C)	Lateral face (B)	Bottom face (A)
	Enthalpy (Hardness)	Enthalpy (Hardness)	Enthalpy (Hardness)
Green	4.72 J/g (83)	42.40 J/g (79)	57,25 J/g (77)
Ultraviolet post-cured	3.68 J/g (83)	38.39 J/g (79)	52.02 J/g (77)
Microwave post-cured	2.69 J/g (84)	32.47 J/g (80)	41.42 J/g (79)
Thermal post-cured at 125^{o}C	2.63 J/g (84)	12.23 J/g (82)	16.81 J/g (82)

(*) all average values.

For stereolithography objects, the applied manufacturing strategy is the same for X and Y axis as presented previously in Fig. (3). On the other hand, it is completely different in Z axis where layers are added one-by-one. So, to analyze the shrinkage as a function of the temperature the specimens behavior was observed in X and Z axis.

Figure (7) plots the linear dimensional variation of a green specimen built with line hatch spacing of 0.10mm. It had shown an initial expansion up to 55^{o}C.

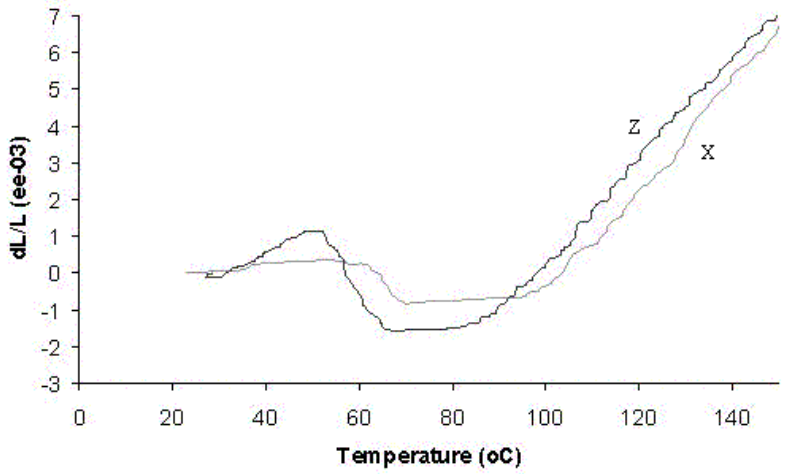

Figure 7. Linear dimensional behavior in X and Z type specimens built using line hatch spacing of 0.10mm without any post-processing treatment (green).

The specimens built with large line hatch spacing (0.15mm) presented shrinkage from 55°C to 95°C in both directions, X and Z. This shrinkage is related to the cure process of uncured resin, and it was more significant in the Z type specimens than in the X type. At temperatures above 100°C, the SL specimens present expansion. When the specimen is heated there is a competition between cure shrinkage and thermal expansion. At low temperatures, the shrinkage starts with the cure reaction. However, close to 100°C the thermal expansion overpasses the dimensional effect of the cure shrinkage (Fig. (8)).

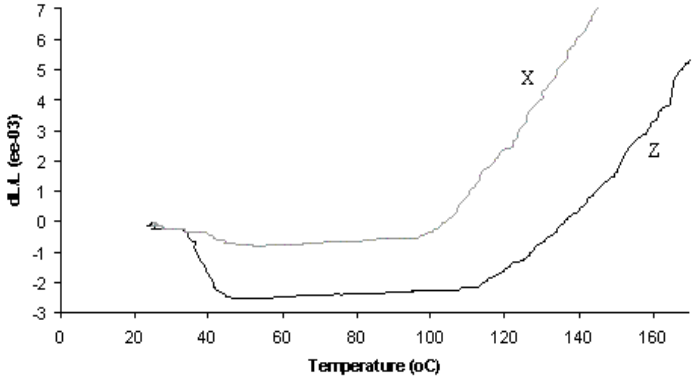

Figure 8. Linear dimensional behavior in X and Z type specimens built using line hatch spacing of 0.15mm.

In general, it is difficult to correlate shrinkage and the cure state to SL resin because the inability to produce samples of uniform microstructure. But the results obtained to the specimens built using a line hatch spacing of 0.10mm suggest that the dimensional behavior of the SL specimens presents a relationship to the degree of cure to the different faces, i.e. the layer-by-layer structure.

The specimens built using 0.15mm in the line hatch spacing presented a similar dimensional behavior as function of temperature to both X and Z type. The cure shrinkage was more pronounced in the Z type where it attended almost 0.3% at 50°C. Higher values of line hatch spacing increase the amount of partially cured resin in each fabricated layer. This fact increased the cure shrinkage of the specimens during the post-curing process, thus resulting on prototypes with poor dimensional accuracy.

The linear dimensional behavior as function of temperature for specimens built using 0.05mm is indicated in Fig. (9). These specimens show a different behavior. The Z type presented a dimensional behavior similar to the others specimens studied and already presented in this work. The cure shrinkage is remarked in the specimens up to 95°C, when it starts to expand. On the other hand, the X type presented a linear dimensional behavior with almost no evidence of shrinkage cure. The specimens showed an expansion behavior from 25 to 85°C with a coefficient of thermal expansion (CTE) average of 30×10^{-6} K^{-1}, and above 85°C it presented a significant expansion with a CTE average of 130×10^{-6} K^{-1}.

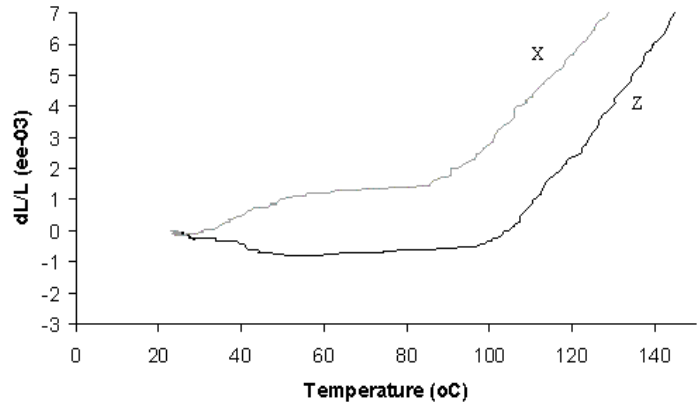

Figure 9: Linear dimensional behavior in X and Z type specimens built using line hatch spacing of 0.05mm.

However, it was also found that using lower line hatch spacing, shrinkage encountered in the X direction could be made much lower than shrinkage in the Z direction as illustrated in Fig. (10). The increase in the Z type shrinkage may be caused by the further polymerization of the higher amounts of liquid and partially cured polymer retained in the lower portions of each layer of the specimens. The use of short line hatch spacing resulted in lower polymerization in Z direction of each layer comparing to X direction.

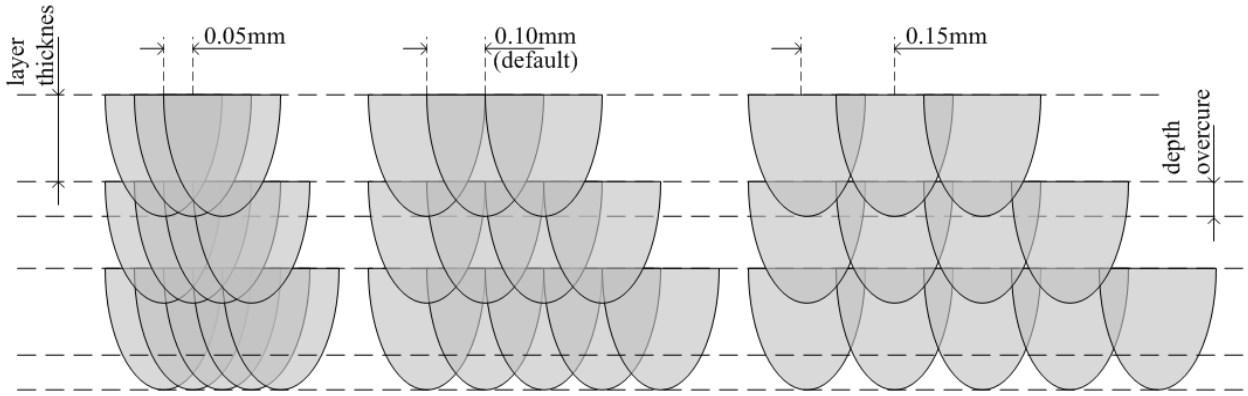

Figure 10. Different line hatch spacing values applied to the specimens and over-cure effects.

The investigation about the effect of the post-cure process over the linear dimensional behavior of SL specimens built was conduced using default parameters, i.e. 0.10mm as line hatch spacing. The linear dimensional behavior in the Z type specimen as function of temperature for the ultraviolet post-cured showed a cure shrinkage between 40°C and 95°C (Fig. (11)). The X type demonstrated a constant dimension resulted from the competitive relationship between cure shrinkage and thermal expansion up to 105°C.

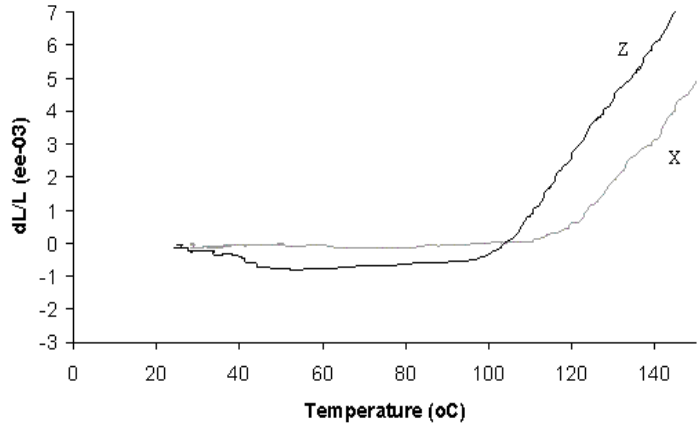

Figure 11. Linear dimensional behavior in X and Z type specimens post-cured by ultraviolet radiation.

Comparing the green and ultraviolet post-cured specimens graphs, the post-cured specimens had shown less shrinkage compared to the green specimens. These results agree with the DSC and Shore D hardness measurements. It indicates that green specimens are not in a full-cured state. Similar results were obtained to microwave post-cured specimens.

In Fig. (12), it can be observed that thermal post-cured specimens exhibit lower amounts of shrinkage than the ultraviolet post-cured specimens. The linear dimensional behavior of the thermally post-cured specimens presented no cure shrinkage for the X and Z type specimens. There is less difference in the linear coefficient of thermal expansion for parallel and perpendicular fabrication directions. The coefficients of thermal expansion were $16x10^{-6}$ K^{-1} from 25°C to 85°C, and $11x10^{-6}$ K^{-1} to temperatures above 95°C for the X type. For the Z type the coefficients of thermal expansion were $26x10^{-6}$ K^{-1} from 25 to 85°C, and $162x10^{-6}$ K^{-1} to temperatures above 95°C. It was observed that improvements in shrinkage and accuracy of the overall final prototype can be obtained by thermal post-cure.

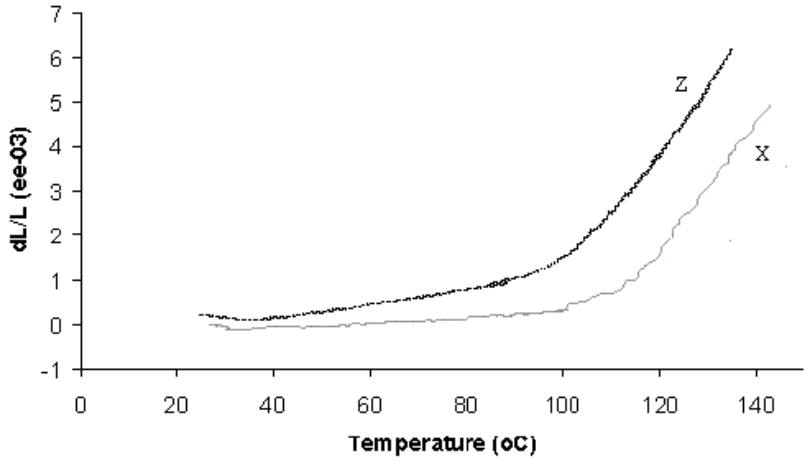

Figure 12. Linear dimensional behavior in X and Z type specimens post-cured by thermal treatment.

It was observed that shrinkage phenomenon depends on the direction of construction, i.e., the part orientation. The over cure necessary to bond layers generates solid planes with higher polymerization degrees as seen in Fig. (13). The last surface of objects built by stereolithography is extra-cured by the default resins parameters. It receives extra amount of energy and shows an expansion as other regions of the object shrink.

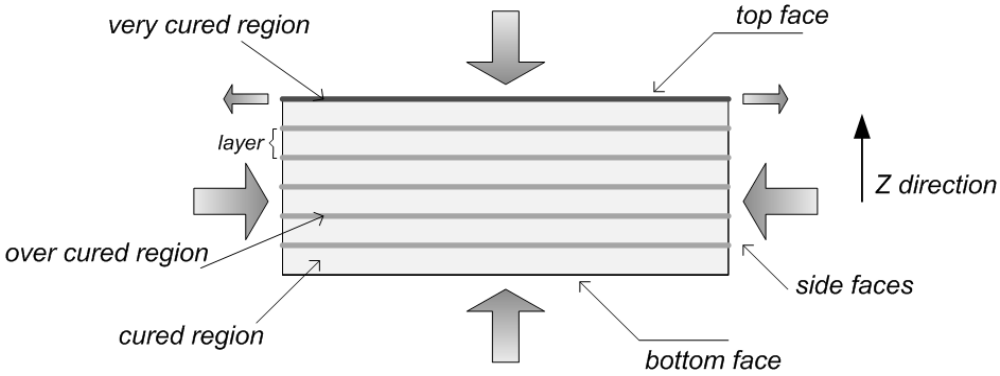

Figure 13. Shrinkage forces over a hypothetical specimen.

4. Conclusions

The difference in the state of cure in the specimens based on heat release obtained by DSC gives an indication of the heterogeneity of SL parts. It was found that uncured and partially cured resins trapped within the part resulted in anisotropy on the structure and properties such as hardness and linear dimensional behavior.

The variation of linear cure shrinkage is proportional to the cure state of the overall specimen. The SL specimens built with the DSM SOMOS 7110 resin presented low cure shrinkage after manufacturing (<0.3%). This can avoid internal stress and it is function of cure shrinkage and Young's modulus.

The use of lower line hatch spacing will deliver a more compactly cured structure, leaving little space for partially-cured and uncured polymer to be retained. This also lowers the amount of post-curing induced shrinkage.

Dimensional accuracy and strength of the rapid prototypes created using the stereolithography method have always been a major problem. The results obtained in this paper, shows that the cure shrinkage and the anisotropy can be minimized by the post-cure process of SL parts. When it is applicable, the thermal post-cure can be used to improve the

cure state minimizing the internal stress. It is important to notice that the treatment temperature is normally above the glass transition of the resin.

The improvement of laser-assisted processing of photosensitive polymers depends primarily on further progress in the scientific understanding and control of the basic phenomena relating high-speed curing and structure-properties.

5. Acknowledgments

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