The Effect of Layer Orientation on the Tensile Properties of Net Shape Parts Fabricated in Stereolithography

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Abstract

Stereolithographic technologies create parts in thermoset plastic polymeric mixtures of acrylates and epoxies. In order to predict the mechanical behavior of these parts, it is critical to understand the effects that build parameters have on the final properties of the polymer. Using a statistics based approach, the build parameters of layer orientation, layer thickness, and resin class are used as inputs. The response variables, peak stress, elongation at break and Young's modulus (modulus of elasticity), are examined using the methodology specified in ASTM D638-01 with modifications as noted. An initial test in Somos 8120 showed the surprising (and statistically significant) result that load bearing capability in the build direction was greater than in the in-layer direction. Additional tensile tests in Somos 8120 and Vantico SL-5510 were undertaken to verify this result, and determine if this effect is present across different classes of resin. This report details the rationale behind this experiment, presents the results to date, and outlines future efforts.

1.0 Introduction

It is reasonable to assume that material property anisotropies exist due to the nature of layerbased fabrication processes. A sizable body of published work shows material anisotropies in the Fused Deposition Modeling (FDM) [1], and Selective Laser Sintering (SLS) [2][3] technologies; however, there is not a corresponding sizable body of published work with respect to stereolithographic (SLA) processes and photopolymers. Some qualitative discussions of the stereolithography materials in the early 1990's recognized that material anisotropies were likely; however, the differences should not be statistically significant. There does not seem to be a quantitative work in the public record to verify this supposition, though there may be information in the proprietary domain.

As part of a larger effort to characterize SLA materials, a simple screening experiment was performed. The intent was to verify that the expected material anisotropy based on the relationship of build direction to tensile loading direction was small enough (i.e., statistically insignificant) that we could build tensile test coupons in any convenient orientation within the vat.

Stereolithography processes build parts by irradiating a homogeneous photopolymer resin in liquid form. The irradiating energy is provided by an ultra-violet laser. The photoinitiators in the resin system are formulated to react to the specific wavelength of the laser and initiate a polymer chain addition reaction when irradiated. An optic system focuses the laser energy to a discrete spot on the surface of the liquid resin. Laser spot location and motion are controlled

through the mirror and galvanometer set. By vector scanning the laser spot, a line of solidified material is formed. Successive overlapping vector scans create a series of bonded lines to form a layer. A second series of vector scans, moving the laser spot in a series of overlapping scans 90° from the previous direction, helps ensure complete reaction of the material, and formation of a good intra-layer bond.

A 3-dimensional object is created by bonding layers together. Subsequent layers are created by depositing additional resin on the surface of the previous layer, and repeating the hatching and filling processes. The laser energy solidifies the current layer as described above, but some energy is imparted to the previous layer. This energy causes a slight overcure of the previous layer, and causes the inter-layer bond to develop.

The solidified lines that form a single layer are irradiated in an overlapping pattern within a short time frame. This should have the effect of initiating very high levels of cross-linking between the individual scan lines within a layer. High levels of cross-linking would seem to indicate high strength. The inter-layer bonding is initiated by the 'left-over' energy not absorbed by intra-layer cross-link formation. The energy felt at the liquid / solid interface should be more variable than that felt in the liquid. Since the solidified layer has already developed a cross-link network, the number of potential cross-linking sites for inter-layer bond formation should be substantially less than the number available for intra-layer bonding. These two factors should lead to the intuitive conclusion that intra-layer strength is superior to inter-layer strength.

2.0 Experimental Method

Three sets of experiments were performed in this study: (1) a screening experiment in Somos 8120; (2) a more in-depth experiment in Somos 8120; and (3) a validation experiment in Vantico SL-5510. For the first two experiments, the test coupons were built in an SLA-3500 using the standard "Fast" build style. The test coupons for the validation experiment were built in an SLA Viper Si² using the standard "Exact" build style in the low-resolution mode. Both of these SLA machines use Nd:YVO₄ laser systems with output wavelengths of 355 nm, and all of the test coupons were built from the same series of .stl files. In all three experiments, testing was within 24 hours of fabrication.

<u>2.1 Screening Experiment in Somos 8120.</u> For the initial screening experiment in DSM 8120, three sets of thirty-six samples were tested. (1) Built flat in the XY plane – Flat group; (2) edgewise in the XY plane – Edge group; and (3) standing along the Z-axis – Standing group. We were not able to gather valid data with respect to the peak stress, elongation at break and modulus of elasticity of the Standing group; however, we were able to gather valid data with respect to ultimate load for all three groups. We chose to use ultimate load as a proxy value for the peak stress in comparing the three groups.

Given the use of the ultimate load as a proxy for peak stress, and the surprising result, we felt it prudent to replicate the experiment, gather valid data for peak stress, elongation at break and Young's modulus, and reexamine the result. Realizing that the Flat and Edge groups were simply variations on a theme, and recalling that maximum shearing stress occurs on a 45° offset plane [4], we redefined the sample orientation set to include in-plane, transverse, and shear loaded samples.

<u>2.2 Second experiment in Somos 8120</u>. For this experiment, we considered three orientations designated as Flat (in-plane), Standing (transverse), and Angled (shear). The three orientations are illustrated in Figure #1.



Figure 1. The three representative sample orientations with respect to the XYZ frame

We arrived at these three orientations by using the following rationale. The primary axes and 45° offsets from the primary axes combine to describe seven (7) unique orientation vectors. If we allow one degree of freedom, a 90° rotation around the vector axis, we create fifteen (15) unique sample orientations. We see that these fifteen sample groups represent five distinct interlayer orientations with respect to the "longitudinal - long transverse - short transverse" frame of the individual samples. Each of these five inter-layer orientations is comprised of three sample groups.

Within these five orientation plane groups, there are three primary groups – "Long-LT" and "Long-ST" orientation planes are both "In-plane" with respect to the loading direction, both "A" and "B" orientation planes are "shear," and the "LT-ST" orientation plane is "transverse." Figure #2 provides a graphical representation of the five distinct inter-layer orientations and their subsequent grouping into the three primary groups denoted. Any single group within a particular primary group (In-plane, transverse, or shear) should provide a reasonable representation for all of the groups within the same primary group. This supposition is borne out by the result of the initial experiment that showed a consistent result within the two in-plane loaded groups (groups Flat and Edge). Based on this evidence and our convenience, we chose the Flat, Standing, and Angled groups for use in subsequent experiments.

Orientation Plane	Groups	Graphic			
LT-ST (Transverse)	STANDING (Z-45) (ZXY)				
Long-LT (In-plane)	FLAT (XY-0) (XYZ)				
Long-ST (In-plane)	(XZY) (YZX) (XY-90)				
"A" (Shear)	Angled (XZ-90) (YZ-90)				
"B" (Shear)	(XZ-0) (Norm-90) (YZ-0)				

Figure 2: Inter-layer bonding plane relative to Long -LT- ST frame

<u>2.3 Validation Experiment in Vantico SL-5510.</u> For the validation experiment in Vantico SL-5510, we fabricated 12 test coupons for each of the same primary groups tested in the second Somos 8120 experiment. Post-processing, curing, and testing were consistent with the standard methods and uniform across each experiment.

<u>2.4 Methods and Apparatus.</u> While exposure to background UV, relative humidity and heat have been shown to affect the properties of parts fabricated in the stereolithography process, we have not sought to control them in this study. The broad spectrum of users neither actively nor consistently controls these factors. One of the purposes of this research is to examine what the user would see outside of the standard conditions. The ASTM standard is designed to provide an "apples-to-apples" comparison of materials – control as many factors as possible and allow the response variable to be driven overwhelmingly by the factor of interest. This is a valid scenario, but it does not necessarily represent what the user in the field will experience.

Similarly, the ASTM D638 standard prescribes fabrication of the test coupons by either die cutting or machining from flat material (e.g., sheet), or by molding. Neither of these matches the SLA process particularly well – generally, SLA parts are made to net shape and used. There may be some surface finishing or treatment, but mostly the surfaces are used as fabricated. By fabricating the test coupons to net shape, we leave the edge effects extant. So, we have chosen to examine those factors (layer orientation, layer thickness, and resin selection) that the user actively controls. For the purposes of this experiment, we do conform to the specifications found in the ASTM standard (coupon form and dimensions, strain rates, etc) not discussed above.

We have discussed layer orientation in the section above, the other parameters of interest are:

Layer thickness (Quantitative) – This is a discrete parameter that can be set at one of three values - .002 inches, .004 inches, or .006 inches.

Resin Class (Qualitative) – This refers to the general class of materials represented by each of the subject resins.

Layer thickness is a user selectable parameter based on the build style chosen. Advanced users have the capability to create and tailor custom build styles. Like vat orientations, the potential number of different styles that can be created is unlimited. For the purposes of approximating typical user results, we limit our build styles and, consequently, layer thickness to those provided by the resin manufacturers.

Resin Classes - There are three classes of material in the stereolithography process: polyethylene-like / polypropylene-like (PE/PP); general purpose; and ABS-like. Each has a different combination of rigidity and durability properties. Parts made from PE/PP - like resins have low rigidity and high durability while GP resins yield high rigidity and low durability. ABS-like materials are highly rigid and highly durable.

The chemical composition of the resins within classes is similar, so a commonly used resin should provide a good representation of its class; however, there are differences in chemistry between the classes. Differences in the cross-linking reaction are a potential cause for differences in the material properties. By using a representative resin from each class, we seek to gain data that will help us define this phenomenon as being process related, chemistry related, or an interaction of process and chemistry. For our purposes we will use these resins to represent the three classes.

General purpose (Epoxy Acrylate):	Vantico SL-5510
Polyethylene-like:	DSM Somos 8120
ABS-like:	Vantico SL-7560

The particular formulations and compositions of the resin systems are proprietary. A review of the constituents of the resin systems shows, even without a precise delineation of constituents, that the materials are dissimilar enough in nature to allow us to declare them to be different [5], [6]. This is borne out by an examination of the experimental data values. The differences in the measured loads, stresses and elongation are large enough to see by observation without need for comparison of the mean values by statistical methods.

After fabricating the test coupons and post-processing them according to standard practice, we subjected the coupons to tensile testing in an Instron Tensile Tester model 4466. The standard manufacturer provided software (Instron Series IX Automated Material Tester – version 8.11.00) was used for reporting of the pertinent data.



Figure 3: Typical test coupons before and after



Figure 4: Instron tensile testing set-up

<u>2.5 Statistical methods</u>. The tensile testing data was subjected to Analysis of Variance (ANOVA) for a fixed effects model per standard and accepted methodologies [7]. As a short review - ANOVA allows us to partition the total variability of the data into its component parts. In our experiment, these components are: (1) the variability between groups due to differences in treatment, and (2) variability within a particular group due to random error. Dividing the components by the degrees of freedom, we have an estimated sample variance of each component. The estimate of the sample variance between treatment groups is denoted MS_t , and the estimate of the sample variance within groups due to random error is denoted MS_e .

Applying Cochran's theorem, we compare the two components. If the null hypothesis, (that the treatments are equal) is true, then the estimated sample variances should both be independently distributed Chi-square random variables. The ratio of the two components is distributed as an "F" statistic. Standard "F" test tables give a reject value based on the level of confidence, the degrees of freedom of the treatments, and the degrees of freedom of the random errors.

Our test statistic is derived by taking the ratio MS_t / MS_e . Any value of this test statistic greater than the standard table value allows us to reject the null hypothesis. If we cannot reject the null hypothesis, then performing a contrast gives us no additional information. We have chosen a 1% level of confidence - meaning that the probability of falsely rejecting the null hypothesis as a result of this comparison to the standard table values is less than 1%.

Since we are testing more than two distinct groups within each material property and class, a rejection of the null hypothesis would require further analysis contrasting each possible pair of treatment groups. This gives us additional information regarding the sources of the differences between the individual groups, and relationships between the groups.

3.0 Experimental Results

<u>3.1 Screening Experiment</u>. For the initial screening experiment, an analysis of variance of the ultimate load data associated to Figure #5 showed that we were able to reject the initial hypothesis - that build direction anisotropies were *not* significant in load carrying capability - with a much less than 1% chance of falsely rejecting. The test statistic value needed for the 1% chance of false rejection was 4.98 (or greater). Our analysis of experimental data yielded a test statistic of 57.81 - an order of magnitude greater than the "reject" value.

Contrasts of the Flat, Edge and Standing data groups showed a statistically significant difference between the Flat and Standing groups, and between the Edge and Standing groups. The Flat and Edge groups did not demonstrate a statistically significant difference. These contrasts make sense.



Build Orientation Effects

Figure 5: Load carrying capability data for the initial experiment

The difference in means between the Flat and Edge groups is less than 5%, and all of the data values of the Edge group fit between the high and low extremes of the Flat group. On further examination, one can see that both the Flat and Edge groups experience in-plane loading during tensile testing. This is not where one would anticipate the anisotropy to be evident. Rather this is an expected result of comparing two variations of in-plane loading samples.

The difference in mean of the Standing group with respect to the means of the Flat group and the Edge group is +25% and +20%. It is not surprising to see differences between the mean of the transverse loaded Standing group and the means of the in-plane loaded Flat and Edge groups. This is where one would expect to see evidence of an anisotropy; however, it is surprising that the mean of the Standing group exceeds the means of the Flat and Edge groups by fairly large, and statistically significant, percentages.

<u>3.2 The Second Experiment in DSM 8120</u> material yielded a similar result with additional information. In this experiment, we examined three hypotheses based on the loading direction versus build layer orientation relationship, namely this relationship would not cause statistically significant anisotropies in Peak Stress, Young's Modulus, and Elongation at Break for test coupons aged less than 24 hours. Also, when an anisotropy was identified, a contrast of the data groups was undertaken. A summary of the test data and contrast results is provided in Figure #6.

				Different	Different	Different
	_			from	from	from
Parameter	Group	Mean	Std Dev	Flat	Standing	Angled
Peak Stress (psi)	Flat	2098.35	63.15		Yes	Yes
	Standing	2495.53	131.47	Yes		Yes
	Angled	2238.14	43.20	Yes	Yes	
Young's modulus (ksi)	Flat	10.32	0.39		Yes	Yes
	Standing	12.90	0.52	Yes		Yes
	Angled	11.13	0.41	Yes	Yes	
Elongation (%)	Flat	10.19	0.78		No	Yes
	Standing	9.05	1.57	No		No
	Angled	8.18	0.66	Yes	No	

Figure 6: Summary of 8120 data for the second experiment

Peak Stress - The analysis of variance for the peak stress data showed that we were able to reject the initial hypothesis - that build direction anisotropies were *not* significant in peak stress - with a much less than 1% chance of falsely rejecting. The test statistic value needed for the 1% chance of false rejection was 5.39 (or greater). Our analysis yielded a test statistic of 24.96 - an order of magnitude greater than the "reject" value.

A statistical contrast of the two peak stress data groups with the least difference in means, the Flat and Angled groups, showed statistically significant differences between those groups. Contrast bears out a statistically significant difference between the Flat and Standing groups as well. The Angled and Standing groups were contrasted as well. These two also proved to have statistically significant differences. In all of the contrast cases, the test statistic values were an order of magnitude greater than the required rejection value, and each of the groups was different from the others with respect to the Peak Stress property.

Young's modulus - An analysis of variance for the Young's modulus data showed that we were able to reject the initial hypothesis - that build direction anisotropies were *not* significant in

Young's modulus - with a much less than 1% chance of falsely rejecting. The test statistic value needed for rejection was 5.39 (or greater). Our analysis of the experimental data yielded a test statistic of 38.78 – again, an order of magnitude greater than the "reject" value.

Contrasting the groups Flat and Angled, we see a statistically significant difference in the Young's modulus. Subsequent contrast of the remaining two possible comparisons shows that they are different as well with respect to the Young's modulus property.

Elongation at Break – The initial analysis of variance of the entire data set of three groups shows that we can reject the hypothesis - that build direction anisotropies were *not* significant in Elongation at Break – with a less than 1% chance of falsely rejecting that premise. The test statistic value needed for the 1% chance of false rejection was 5.39 (or greater). Our analysis of experimental data yielded a test statistic of 10.45.

In this case, the Flat and Angled groups showed the largest difference in the group means. The least difference in means was between the Standing and Angled groups. The test statistic for the Standing and Angled contrast was 3.09 versus a minimum reject value of 7.95 – we cannot reject the hypothesis based on differences in these two groups. A similar result occurs with the Standing and Flat groups – the test statistic is 5.14 versus a reject value of 7.95 or greater. Only in the contrast of the groups with the largest difference in means, groups Flat and Angled, do we have a test statistic sufficiently large (46.66) to reject the null hypothesis.

<u>3.3 Validation Experiment in Vantico SL-5510</u> - As noted in the Experimental Method section, the suspected material property anisotropies may be related to the chemical composition of a particular class rather than being process related, or evident across all classes. A similar series of tests were run using net-shaped coupons fabricated from Vantico SL-5510 resin. A similar result in this material may give an indication that the effect is process related.

			[Different	Different	Different
				from	from	from
Parameter	Group	Mean	Std Dev	Flat	Standing	Angled
Peak Stress (psi)	Flat	5762.32	152.26		Yes	Yes
	Standing	7755.67	972.35	Yes		No
	Angled	7837.70	838.50	Yes	No	
Young's modulus (Ksi)	Flat	51.23	3.43		No	Yes
	Standing	55.76	9.70	No		No
	Angled	59.33	8.26	Yes	No	
Elongation(%)	Flat	33.90	3.70		Yes	Yes
	Standing	21.70	6.40	Yes		No
	Angled	22.30	7.40	Yes	No	

Figure 7: Summary of test data and contrast results in Vantico SL- 5510 resin

Peak Stress - The analysis of variance for the peak stress data showed that we were able to reject the initial hypothesis - that build direction anisotropies were *not* significant in peak stress - with a much less than 1% chance of falsely rejecting. The test statistic value needed for the 1% chance of false rejection was 5.39 (or greater). Our analysis of yielded a test statistic three orders of magnitude greater than the "reject" value.

A statistical contrast of the two peak stress data groups with the least difference in means, the Standing and Angled groups, showed no statistically significant differences between those groups. The test statistic for these two groups was .05 versus a reject value of 7.95 or greater, and the hypothesis that the two treatment means are different cannot be rejected. Contrasting the two remaining sets leads to the rejection of the hypotheses in both cases. The Standing and Flat contrast and the Angled and Flat contrast reject the hypothesis with test statistics of 49.22 and 71.17, respectively.

Young's modulus - Similarly, an analysis of variance for the Young's modulus data showed that we were able to reject the initial hypothesis - that build direction anisotropies were *not* significant in Young's modulus - with a much less than 1% chance of falsely rejecting. The test statistic value needed for the 1% chance of false rejection was 5.39 (or greater). Again, the analysis yielded a test statistic three orders of magnitude greater than the "reject" value.

Group contrast results are summarized in Figure #7. Even though the ANOVA of the full data set rejects the hypothesis with a test statistic much larger than the required reject value, we see that two of the contrasts do not show a statistically significant difference between treatment means of the groups under consideration. Neither the Angled and Standing contrast nor the Flat and Standing contrast is capable of rejecting the hypothesis. In these two contrasts, the test statistics were .94 and 2.33, respectively while the reject value was 7.95 or greater.

Elongation at Break – The initial analysis of variance of the entire data set of three groups shows that we can reject the hypothesis - that build direction anisotropies were *not* significant in Elongation at Break – with a less than 1% chance of falsely rejecting that premise. The test statistic value needed for the 1% chance of false rejection was 5.39 (or greater). Our analysis of experimental data yielded a test statistic of 15.75 – nearly a factor of 3 greater than the "reject" value.

In this case, both the Flat and Angled group contrast and the Flat and Standing group contrast generated test statistics sufficiently large to reject the hypothesis. The test statistics generated were 24.06 and 32.96, respectively, versus reject statistic of 7.95 or greater. The test statistic for the Standing and Angled contrast was .04 versus a minimum reject value of 7.95 – we cannot reject the hypothesis.

Summary of results – Statistically significant material anisotropies related to build orientation were shown for the Peak Stress, Young's modulus and Elongation at Break properties of net shaped test coupons fabricated using the Stereolithography process. This anisotropic effect was seen in two chemically different classes of material.

4.0 Conclusions

The initial screening experiment yielded two results: First, that there is a statistically significant anisotropy in the ultimate load carrying capability as a result of the relationship between build orientation and loading direction; and secondly, that the intuitive solution that the in-plane (Flat) property would be superior to the transverse plane (Standing) property was found not to hold in this case. The first result may be expected, but the second result is somewhat of a surprise.

The second experiment confirmed the finding of the first experiment and amplified the finding by yielding data on the Peak Stress, Young's modulus, and Elongation at Break properties. The in-plane loaded (Flat) coupons showed the lowest Peak Stress and Young's modulus and exhibited the greatest Elongation at Break. The transverse loaded (Standing) coupons showed the highest Peak Stress and Young's modulus while the least Elongation at break was found in the shear loaded (Angled) orientation.

The third experiment set had a similar finding – statistically significant anisotropies in Peak Stress, Young's modulus, and Elongation at Break – in a different class of material. Again the in-plane loaded (Flat) coupons showed the lowest Peak Stress and Young's modulus and exhibited the greatest Elongation at Break; however, in this trial, the transverse loaded (Standing) and shear loaded (Angled) coupons switched rankings. The shear loaded (Angled) coupons showed the highest Peak Stress and Young's modulus while the least Elongation at break was found in the transverse (Standing) loaded orientation.

The similar results with regard to the in-plane loaded coupons (Flat) across all three trials may be an indication of a process driven effect. The switching of ranks of the transverse loaded (Standing) and shear loaded (Angled) coupons in the results of the second and third experiments may be an indication of some process-chemistry interaction. The only two orientations that were found to be statistically different in every contrast, regardless of material, were the shear loaded (Angled) and in-plane loaded (Flat) orientations.

These results support the existence of a process driven effect with respect to tensile properties as a function of build orientation. Also, the data suggests that there may be a secondary process-chemistry interaction.

5.0 Future work

The next series of experiments will seek to verify the process driven effect by testing the third class of material. As noted, we will be using Vantico 7560 to represent the ABS-like class of materials. Other materials run on equipment other than our own, my yield additional information. In addition, to gain further information on the process driven effect, we will be testing coupons built in 7110 on a HeCd laser system. Given the proprietary nature of the materials, verification of a process-chemistry interaction could be quite difficult to verify.

Also, we will be checking this effect at various stages of material aging to determine if it is consistent across time, or simply transient.

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