IN-SITU PROPERTY MEASUREMENTS ON LASER-DRAWN STRANDS OF SL 5170 EPOXY AND SL 5149 ACRYLATE

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ABSTRACT

Material behavior plays a significant role in the mechanics leading to internal stresses and, potentially, to distortion (curling) of parts as they are built by stereolithography processes that utilize photocuring resins. A study is underway to generate material properties that can be used to develop phenomenological material models of epoxy and acrylate resins. Strand tests are performed in situ in a 3D System's SLA-250 machine; strands are drawn by either single or multiple exposures of the resin to a laser beam. Linear shrinkage, cross-sectional areas, cure shrinkage forces and stress-strain data are presented. Also, the curl in cantilever beam specimens, built with different draw patterns, are compared.

INTRODUCTION

During the stereolithography build process, parts are built layer by layer using a laser to selectively cure slices of photocuring resin. As the laser spot passes over the surface of the resin, the ensuing chemical reaction causes the resin to shrink and stiffen during solidification. When laser paths cross or when new layers are cured on top of existing layers, residual stresses are generated as the cure shrinkage of freshly gelled resin is constrained by the adjoining solid material. These internal stresses can cause significant out-of-plane curling in the compliant material.

A program is underway at Sandia National Laboratories to develop and to evaluate the use of engineering tools for modeling curl and for studying parameters that might improve overall build accuracy in stereolithography parts [1]. These tools include phenomenological material models of the solidifying resins and a 3-D finite element model that incorporates time varying material behavior, laser path dependence, and structural linkage [2]. Methods developed for in situ response of polymer strands drawn during single (or multiple) passes of a He-Cd laser in 3D system's SLA-250 stereolithography apparatus have been reported along with initial experimental results [1]. Additional strand data are presented below for two Ciba Giegy photocurable resins- Cibatool® SL 5170 epoxy and Cibatool® SL 5149 acrylate. The strand specimens were drawn in an SLA-250. The data include linear shrinkage, cross-sectional areas, cure shrinkage forces and initial modulus data of strands following single and multiple laser hits. In addition, out-of-plane curl data for acrylate cantilever beam specimens built with different draw patterns are discussed.

TEST PROCEDURES AND RESULTS

LINEAR SHRINKAGE- Time history linear shrinkage of SL 5170 epoxy and SL 5149 acrylate strands were obtained by using a non-contact video method to measure the change in length of individual strands after gelation following a single laser hit. These data were initially reported in [1]; representative curves for one inch long specimens are shown in Figure 1. Note

that for a single laser exposure, the epoxy has a linear shrinkage of about 1.5% and the acrylate approximately 1.0%. Shrinkage is complete about three minutes after gelation.

AREA MEASUREMENTS- The metallography technique developed to determine the shapes and areas of SL 5170 epoxy and SL 5149 acrylate strands was described in [1]. The same technique was used to measure epoxy and acrylate strand cross sections following single and multiple laser hits; typical binary images are shown in Figure 2. The strands were not recoated with fresh resin between hits in the multiple hit experiments. Curves of strand area as a function of number of laser hits are plotted in Figure 3. Note that the cross sectional area of the epoxy increases at a higher rate than the acrylate area does.

FORCE MEASUREMENTS- Fixtures developed for in situ measurement of forces during cure shrinkage, step displacement loading, and stress relaxation tests of strands are schematically illustrated in Figure 4; more detail is given in [1]. A drawn strand attaches to tapered tabs that are just below the resin surface; one tab is connected to an extension from a miniature force gage and the other tab is connected to a platform whose movement can be controlled by an electronically-driven micrometer. A 1.0 inch strand was used in all force tests. The 150 gram load force gage, calibrated to 20 grams full scale has a resolution of ± 0.02 gram. An LVDT (± 0.05 inch range) is used to monitor strand displacement imposed by the micrometer whose displacement rate and amplitude can be controlled. In all 0.5% step strain tests, strands are strained at a rate of about 0.01 s^{-1} .

In situ force tests are performed to measure (1) cure shrinkage forces developed after single or multiple laser hits on single strands, (2) stress-strain response when a 0.5% step strain is imposed on a strand at different elapsed times after the strand is drawn, and (3) force-time relaxation response after 0.5% step strain. Initial apparent modulus values are based on the initial slope of force-strain curves and the cross sectional area of the strand. These values of modulus are integrated averages over the strand cross section, whose properties are nonhomogeneous due to a cure gradient resulting from attenuation of laser energy in the resin. The cure gradient determines the extent of reaction (degree of cure) at different locations in the strand cross section.

<u>SL 5149 acrylate data</u>- A typical force versus time response of an SL 5149 acrylate strand which was hit with 4 laser exposures prior to applying 0.50% step strain is shown in Figure 5. This curve illustrates that no shrinkage forces are detected after the 1st laser hit, but that cure shrinkage forces are measured following additional laser hits. The actual linear shrinkage associated with multiple hits can not be measured because the strands are attached at both ends and cannot shorten. The stress-strain responses during the 0.5% step strain provide initial modulus values.

Figure 6 clearly shows that the acrylate continues to shrink for at least 20 laser exposures. The magnitude of each additional cure shrinkage force increment decreases as the number of laser hits increases. A small region of Figure 6 is expanded in Figure 7 to show in greater detail the shape of the force-time response during and following a laser hit. It is of interest to note that immediately following a laser hit, the force initially decreases, possibly due to thermal expansion of the strand from laser heating. The strand then undergoes additional cure shrinkage producing a force that initially increases rapidly before leveling off.

Initial modulus of acrylate strands as a function of the number of laser hits is shown in Figure 8. The modulus calculations account for the strand area associated with the number of laser hits. The initial modulus increases from about 1000 psi for a single hit to near 110,000 psi for a strand exposed to 28 laser hits. It was reported in [1] that the initial modulus of acrylate strands following a single laser hit did not increase with increasing time interval between the laser exposure to application of the step strain. Acrylate strands continue to shrink and to increase in modulus (degree of cure) following multiple laser hits, for at least 28 hits. Recall that the strand is not recoated with fresh resin between laser exposures.

<u>SL 5170 epoxy data</u> - No shrinkage forces were detected for SL 5170 epoxy strands in tests in which specimens were subjected to as many as 8 laser hits. However, forces were measured when strands were subjected to a 0.5% step strain. Strands were strained at different elapsed times following the laser draw. Results from tests on strands of a single laser hit with elapsed times up to 240 minutes show that both initial stiffness and peak force increase with longer elapsed times [1]. However, in tests on strands after multiple laser hits, initial modulus does not appear to be influenced by the number of hits but by the elapsed time from the 1st hit to the step strain. Typical stress-strain responses for multiple hit strands are plotted in Figure 9. The stress-strain curves for 1, 2, and 3 laser hits are about identical (all with an elapsed time of 5 minutes). Strands with more hits and longer elapsed times had stiffer responses. Moduli data from these curves are compared with the single strike data as a function of elapsed time in Figure 10. These data seem to illustrate that the initial modulus SL 5170 epoxy is dependent on elapsed time and not the number of laser hits as is the SL 5149 acrylate.

CURL MEASUREMENTS - SL 5149 acrylate cantilever beam artifacts of the dimensions shown in Figure 11 were built and the resulting out-of-plane curl was measured. Two different laser beam vector paths were used to build the specimens. In one build style, the direction of the laser was always in the 1-(length-wise) direction of the cantilever. In the other build style, the laser path utilized vectors in the 2- (transverse) direction of the cantilever. A 0.010 inch resin recoat was applied for each layer; the center section of the specimen is 8 layers thick and the cantilever beam is 6 layers thick. The other variable is the number of laser hits each layer receives (either 1 or 2). There was no resin recoat between the 1st and 2nd hits of those artifacts which had a 2nd laser hit on each layer. In all cases, there was a three minute delay time between the laser draw of the individual layers and between the 1st and 2nd hit on the same layer.

Four cantilever beams of each build style (all with 1 laser hit on each layer) were built-- two of the four were postcured in a UV oven for 30 minutes. A side view of each specimen was photographed using an optical microscope. Its image was digitized and image analysis software was used to measure the out-of-plane (3-direction) curl or distortion. Figure 12 shows typical results for artifacts drawn with 1-direction and 2-direction vectors, each with a single laser exposure for each layer. The photographs show the cantilever beams and the graph gives the vertical curl as a function of the position along the bottom of the beam from the beam support to the beam tip. The photographs clearly show the individual layers and curl. The rectangular shapes within each layer in (a) results from the transverse direction draw pattern. The curves in Figure 12(c) show that the bottom edge curl is about 0.016 inch at the tip of the 0.15 inch long beam for all specimens. Neither the draw style nor the post curing step influenced the magnitude or distribution of curl in the acrylate beams.

The fact that the draw direction did not affect curl in SL 5149 acrylate is probably because the draw time of a single layer is on the order of a few seconds, a very short time relative to the 2-3 minutes necessary for full shrinkage to occur (see Figure 1). Alternate draw styles and time delays that may cause cantilever beams to have different amounts of curl are planned for future tests. If those results are successful, cantilever specimens will also be built with the SL 5170 epoxy.

SUMMARY

In situ property measurements were made on single strands of SL 5170 epoxy and SL 5149 acrylate photocurable resins. In a single laser exposure, epoxy strands shrank approximately 1.5% and the acrylate strands about 1.0%. The cross sectional area of strands increased with additional laser hits (no recoating with liquid resin between hits). The increase in area with laser hits was greater for the epoxy than for the acrylate. No cure shrinkage forces were measured after the first laser hit in either epoxy or acrylate, and none was measured after multiple laser hits in the epoxy. This suggests that the epoxy either does not continue to shrink after the 1st hit or that shrinkage forces cannot overcome strand stiffness. On the other hand, the acrylate develops additional cure shrinkage forces for 20 or more laser hits. In force relaxation tests, a strand is drawn and then a 0.5% step strain is applied after different number of laser hits and/or elapsed time between the first hit and the step strain. The acrylate initial modulus is a function of the number of laser hits, but not the elapsed time. In contrast, epoxy modulus is a function of elapsed time, but not the number of laser hits.

Procedures were developed to measure out-of-plane distortion (curl) of cantilever beam specimens. Beams (depth of 0.060 inch and length of 0.15 inch) drawn with two different build styles had curl of about 0.016 inch at the tip. Neither the draw style nor a post cure step influenced the magnitude or distribution of curl in the acrylate beams. The draw time was so short that essentially no shrinkage occured during the drawing of a single layer by either of the two draw patterns used.

REFERENCES

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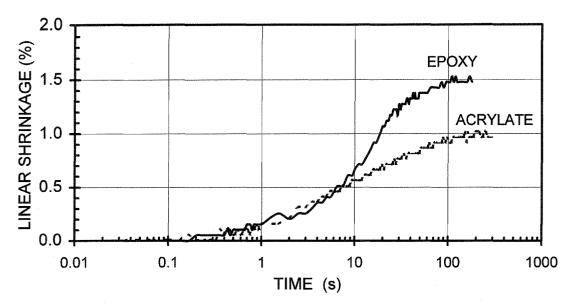


Figure 1. Linear shrinkage of SL 5170 epoxy and SL 5149 acrylate strands following a single laser exposure.

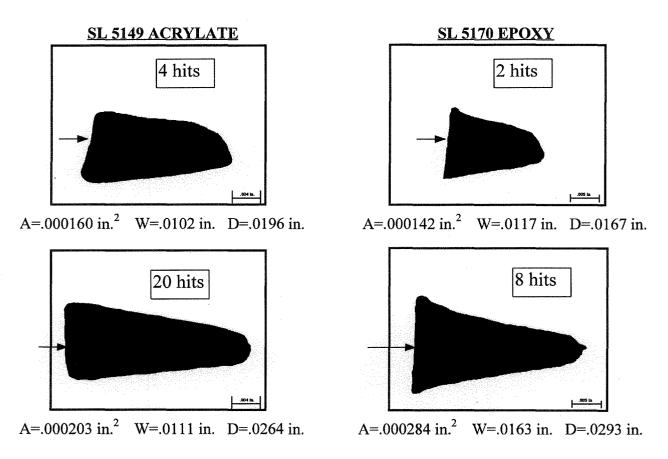
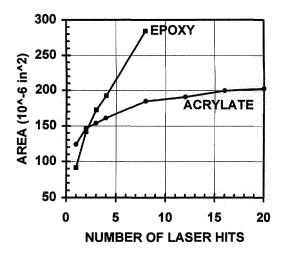


Figure 2. Cross sections of SL 5149 acrylate and SL 5170 epoxy strands for different number of laser hits. Arrow indicates direction of laser hit. Strands were not recoated between hits. A, W and D denote strand area, width (at widest point) and depth, respectively.



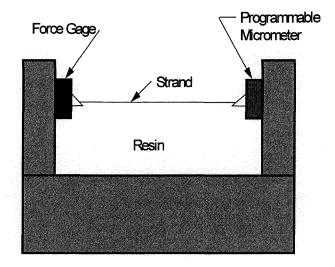


Figure 3. Strand cross sectional area as a function of number of laser hits.

Figure 4. Schematic of experimental setup for in situ measurement of forces.

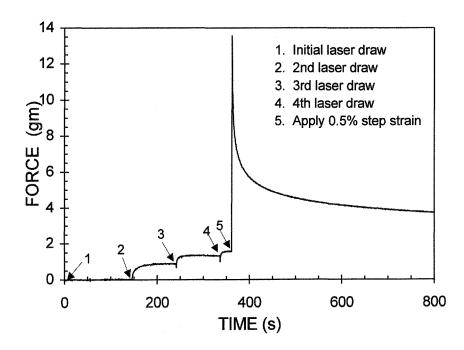


Figure 5. Forces developed in SL 5149 acrylate due to multiple hits of the laser and a 0.5% step strain to a 1.0 inch strand. Force relaxation occurs following the step strain.

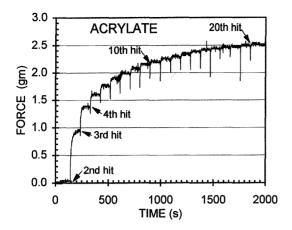


Figure 6. Cure shrinkage forces in a 1.0 inch acrylate strand due to multiple hits of the laser.

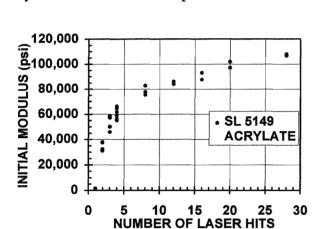


Figure 8. Initial modulus of SL 5149 acrylate as function of number of laser hits.

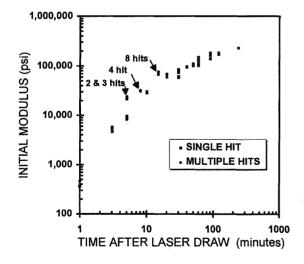


Figure 10. Initial modulus of epoxy strands as a function of elapsed time from the 1st laser hit.

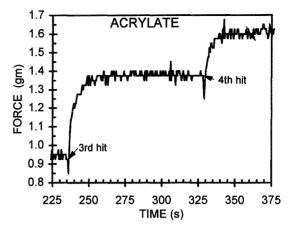


Figure 7. Expanded view of cure shrinkage forces in the Figure 6 acrylate strand.

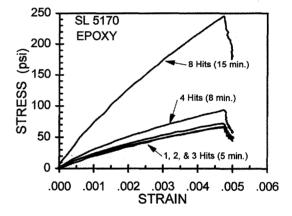
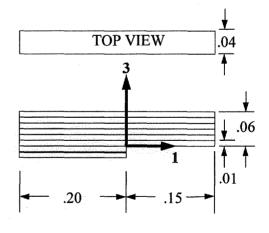
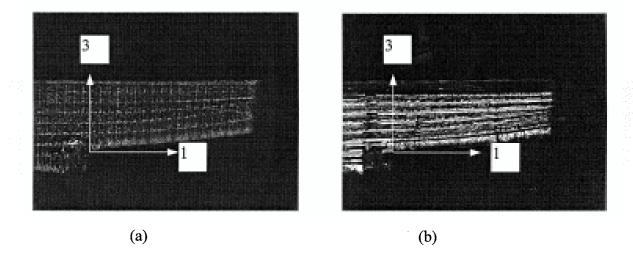


Figure 9. Stress vs. strain response of epoxy strands for different number of laser hits.



CANTILEVER SECTION HAS 6 LAYERS

Figure 11. Dimensions (inches) of 6-layer distortion (curl) specimen.



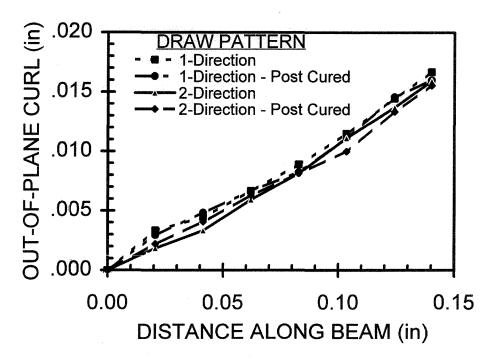


Figure 12. Out-of plane curl of six -layer acrylate cantilever beams (about 0.010 inch thick layers) for two draw patterns and cure conditions: (a) side view of a beam built with a 2-direction (transverse) draw pattern, (b) a beam built with a 1-direction pattern, and (c) comparisons of bottom surface curl versus distance along the beam for specimens of the two build patterns, without and with postcure.

(c)