LAWRENCE
LIVERMORE
NATIONAL
LABORATORY

LLNL-TR-685381

Load Deflection of Dow Corning SE 1700 Simple Cubic Direct Ink Write Materials: Effect of Thickness and Filament Spacing

W. Small, M. A. Pearson, T. R. Metz

March 9, 2016

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

Load deflection of Dow Corning SE 1700 simple cubic direct ink write materials: Effect of thickness and filament spacing

Work done by: Tom Metz (3D printing), Mark Pearson (mechanical testing), Ward Small (data analysis), Lynda Tesillo (data analysis)
Report written by: Ward Small
Date of report: 12/21/2015

SUMMARY

Dow Corning SE 1700 (reinforced polydimethylsiloxane) porous structures were made by direct ink writing (DIW) in a simple cubic (SC) configuration. The filament diameter was 250 µm. Structures consisting of 4, 8, or 12 layers were fabricated with center-to-center filament spacing ("road width" (RW)) of 475, 500, 525, 550, or 575 µm. Three compressive load-unload cycles to 2000 kPa were performed on four separate areas of each sample; three samples of each thickness and filament spacing were tested. Geometry-dependent buckling of the SC structure was evident. At a given strain during the third loading phase, stress varied inversely with porosity. At strains of 25% and higher, the stress varied inversely with the number of layers (i.e., thickness); however, the relationship between stress and number of layers was more complex at lower strains. Intra-and inter-sample variability of the load deflection response was higher for thinner and less porous structures.

MATERIALS AND METHODS

Sample Preparation

Dow Corning SE 1700 clear adhesive is a two-part heat cure reinforced polydimethylsiloxane rubber with a 10:1 by weight mix ratio. After mixing using a Thinky planetary mixer, the resin was vacuum degassed, loaded into a 30 cc syringe, vacuum degassed again, and centrifuged. The trapped air was bled from the syringe and a micronozzle (250 µm inner diameter) was attached to the syringe. The syringe was mounted to the z-stage of a three-axis linear positioning system (Aerotech).

The samples were made using a direct ink write (DIW) 3D printing process [1]. A silicon substrate coated with a teflon mold release agent was mounted on the xy-stage of the positioning system. A positive displacement fluid dispenser (Ultimus IV Model 2800-30, Nordson) was connected to the syringe and programmed to dispense the resin at a constant rate that matched the print speed (15 mm/s). Printing was initiated by executing the tool path program in the A3200 CNC Operator Interface Control software (Aerotech). A multi-layer structure (4, 8, or 12 layers) resembling a simple cubic (SC) configuration was printed (Fig. 1). The nominal filament diameter was 250 µm and the nominal filament center-to-center spacing ("road width" (RW)) was 475, 500, 525, 550, or 575 µm. The finished sample was approximately 72.4 mm square by 0.8, 1.6, or 2.5 mm thick for the 4-, 8-, or 12-layer structures, respectively. The silicon substrate with the completed sample was removed from the positioning system and placed in an oven at 150°C for 1 h under nitrogen purge to cure the resin. The cured sample was detached from the silicon substrate and post-cured at 125°C for 12 h under nitrogen.

Each printed sample was weighed and the thickness was measured at four locations using a digital thickness gauge (Ono Sokki EG-233, spring removed) with an 8 mm diameter flat contact point at a constant force of 40.7 g (7.9 kPa compressive stress).

Load Deflection Testing

Compressive cyclic load deflection was performed at room temperature using an Instron 5967 dual-column load frame with a 28.68 mm diameter fixed lower platen and a 50 mm diameter spherical seat upper platen. No lubricant was used on the polished steel platens. Four areas on each printed sample were tested (three samples for each thickness and filament spacing). Three load-unload cycles up to 2000 kPa compressive stress were performed (test speed = 1.27 mm/min), and the load and crosshead displacement were recorded at 10 Hz. Instrument compliance (~6×10^{-5} mm/N) was measured to correct the crosshead displacement. Stress (engineering) was given by the load divided by the compressed area. Compressive strain (engineering) was calculated using the specimen thickness given by the crosshead displacement at a stress of 2 kPa during the first loading phase; the thickness measured by the digital gauge was not used.

The average porosity P of the printed samples was calculated using the equations

$$P = 1 - \frac{\rho_{prin}}{\rho_{base}}$$

$$\rho_{prin} = \frac{m}{l^2 h}$$

where m is the average mass of the printed samples, l is the side length of the printed samples (assumed identical for all samples), h is the average thickness of the printed samples (given by the Instron crosshead displacement at a load of 2 kPa during the first loading phase), ρ_{base} is the density of the solid SE 1700 base material (1.13 g/cm^3 [2]), and ρ_{prin} is the density of the printed sample.

Average sample specifications are summarized in Table 1.

RESULTS

Sample thickness (obtained from the Instron measurement) is plotted as a function of number of layers in Fig. 1 (data shown in Table 1). The linear relationship, which was independent of the filament spacing, suggests that the thickness of a single layer (~0.2 mm) is independent of the number of layers. Approximately 20% overlap between filaments was evident.

Calculated porosity is plotted as a function of center-to-center filament spacing in Fig. 2 (data shown in Table 1). The relationship is approximately linear. Extrapolating the linear fit indicates a porosity of 21% at the minimum printable center-to-center spacing of 250 μm (adjacent parallel filaments touching).

Typical compressive stress vs. strain curves for the three load-unload cycles are shown in Fig.3. The reduction in slope as strain increased was attributed to buckling of the stress columns in the SC structure. The buckling was most severe for the thinnest and most porous samples. Stress was reduced in the second and third loading phases due to the Mullins effect. The stress at 10%, 20%, 25%, 30%, and 40% strain during the third loading phase is plotted as a function of center-to-center filament spacing (and calculated porosity) in Figs. 4-8, respectively (data for 25% strain is shown in Table 1). At a given strain, the stress varied inversely with porosity. At strains of 25% and higher, the stress varied inversely with the number of layers (i.e., thickness); however, the relationship between stress and number of layers was more complex at lower strains. The error bars (standard deviation) indicate that intra-and inter-sample variability of the load deflection response was higher for thinner and less porous structures.

CONCLUSIONS

Thickness varied linearly with number of layers, independent of the filament spacing. Approximately 20% overlap between layers was evident, independent of the number of layers and filament spacing. Porosity varied approximately linearly with filament spacing. Stress vs. strain curves indicated geometry-dependent buckling of the SC structure. At a given compressive strain, the third loading stress varied inversely with porosity (filament spacing). At strains of 25% and higher, the stress varied inversely with the number of layers (i.e., thickness); however, the relationship between stress and number of layers was more complex at lower strains. Intra-and inter-sample variability of the load deflection response was higher for thinner and less porous structures.

ACKNOWLEDGMENTS

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

REFERENCES

1. EB Duoss, TH Weisgraber, K Hearon, C Zhu, W Small IV, TR Metz, JJ Vericella, HD Barth, JD Kuntz, RS Maxwell, CM Spadaccini, TS Wilson. Three-dimensional printing of elastomeric, cellular architectures with negative stiffness. Adv Funct Mater 2014; 24:4905-13.
2. Dow Corning SE 1700 Product Information Sheet, Ref. No. 11-1785-01, 2010. Available online: http://www.dowcorning.com/applications/search/products/details.aspx?prod=01707116&type=PROD

Table 1: Dow Corning SE 1700 DIW Printed Samples: SC configuration, 250 µm filament diameter, 72.4 mm side length

Number of Layers	Center-to-Center Filament Spacing (mm)	n[(a)]	Thickness Using Digital Gauge (mm)	Thickness at 2kPa in Instron (mm)	Calculated Porosity (%)	3rd Load Stress at 25% Strain (kPa)
4	475	12	0.757±0.018	0.784±0.020	43	363±22
	500	12	0.757±0.007	0.785±0.007	45	323±22
	525	12	0.735±0.008	0.755±0.007	48	275±16
	550	12	0.751±0.016	0.773±0.015	50	244±16
	575	12	0.732±0.019	0.759±0.019	52	215±11
8	475	12	1.580±0.026	1.617±0.027	43	316±16
	500	12	1.587±0.016	1.627±0.016	46	257±11
	525	12	1.581±0.008	1.623±0.008	49	227±6
	550	12	1.579±0.004	1.618±0.004	51	197±6
	575	12	1.584±0.010	1.625±0.011	54	175±8
12	475	12	2.413±0.010	2.454±0.009	43	263±6
	500	12	2.468±0.018	2.520±0.019	48	206±10
	525	12	2.453±0.007	2.501±0.008	50	185±5
	550	12	2.423±0.011	2.475±0.009	52	171±4
	575	12	2.399±0.012	2.459±0.014	53	145±2

[(a)] n = number of samples (3) × number of test areas per sample (4)

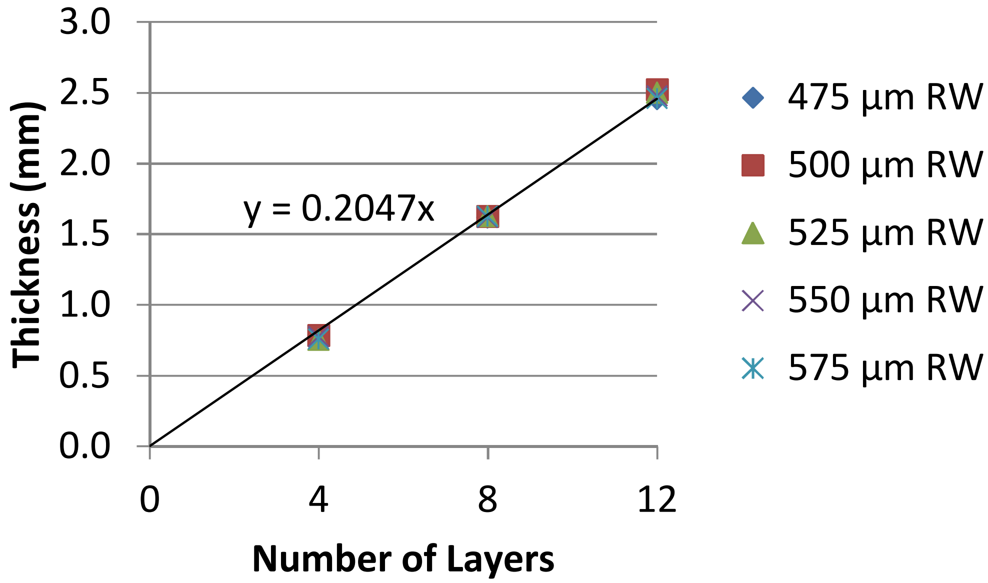


Fig. 1. Sample thickness vs. number of layers for the SE 1700 DIW SC samples with 4, 8, or 12 layers and center-to-center filament spacing of 475, 500, 525, 550, or 575 µm. Data points represent the average thickness and errors bars (too small to visualize in this plot) represent standard deviation (n=12).

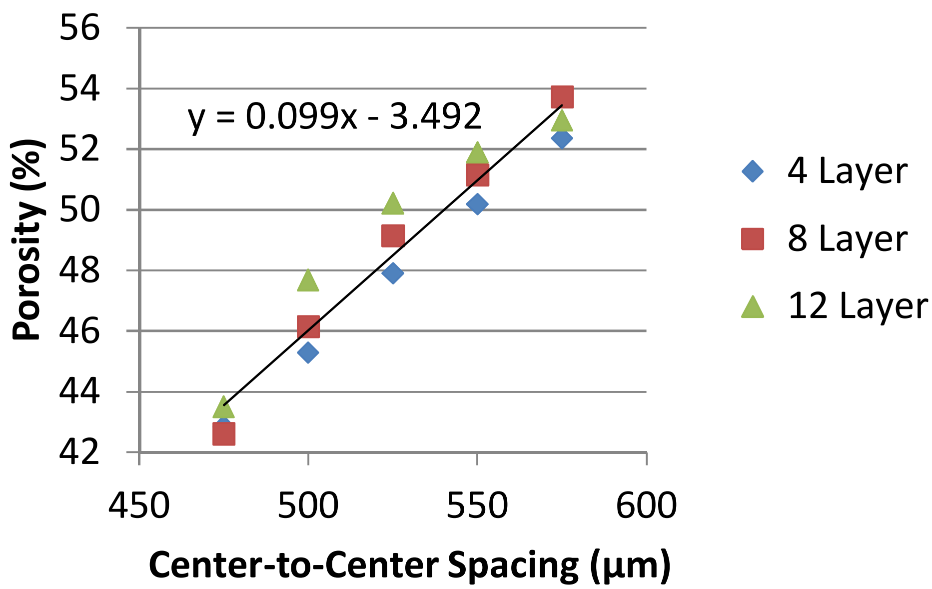


Fig. 2. Calculated porosity vs. center-to-center filament spacing for the SE 1700 DIW SC samples with 4, 8, or 12 layers and center-to-center filament spacing of 475, 500, 525, 550, or 575 µm. Extrapolating the linear fit indicates 21% porosity at the minimum printable center-to-center spacing of 250 µm (adjacent parallel filaments touching).

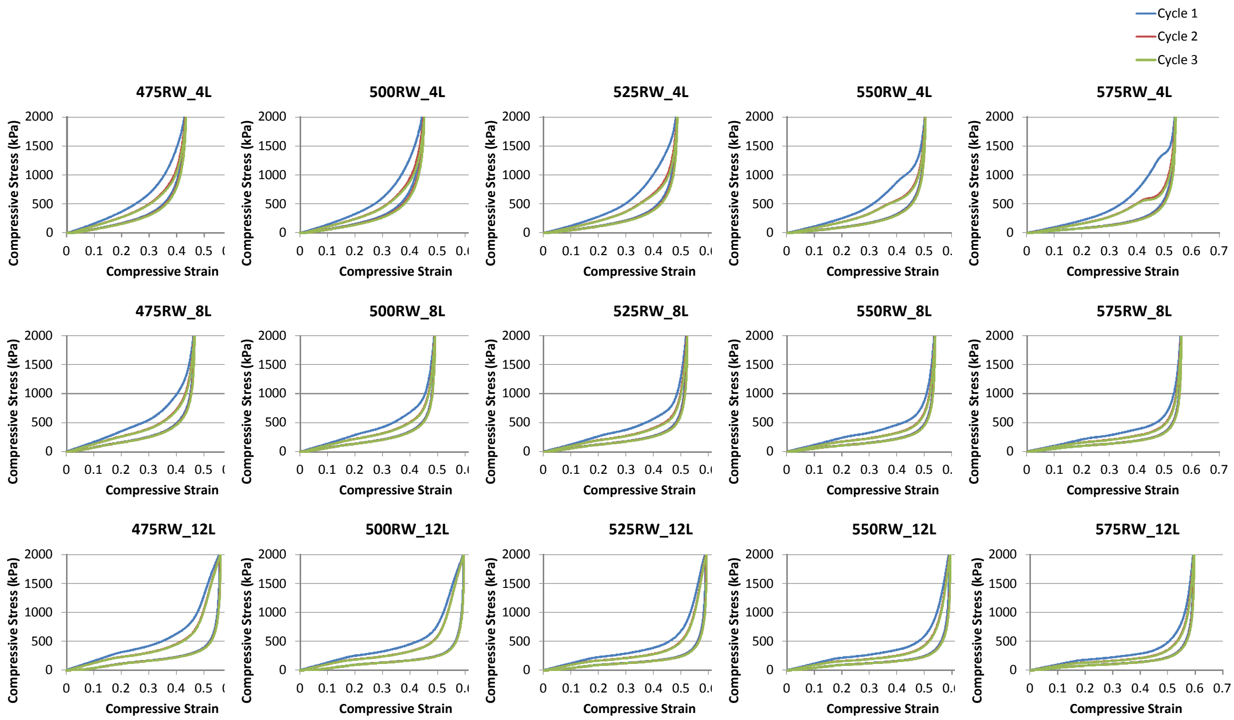
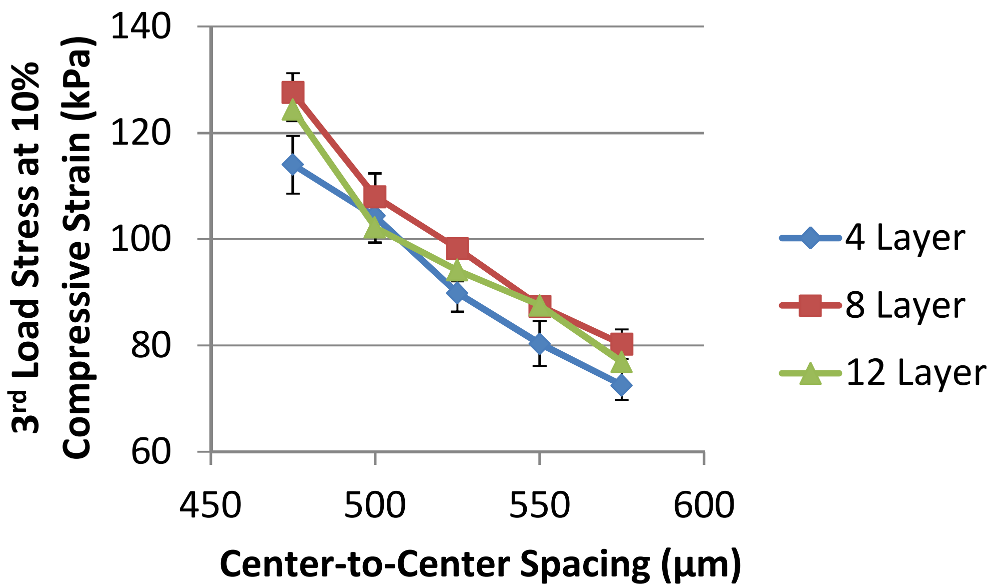


Fig. 3. Typical compressive stress vs. strain (engineering values) for the SE 1700 DIW SC samples with 4, 8, or 12 layers and center-to-center filament spacing of 475, 500, 525, 550, or 575 µm. Data from one test area on a single sample is shown. The onset of buckling (sudden decrease in slope) occurred at ~40% strain in the 4-layer specimens with 500 and 575 µm filament spacing, but was not evident in the less porous specimens. Buckling began at ~20% strain in the 8- and 12-layer specimens for all filament spacings.

(a)



(b)

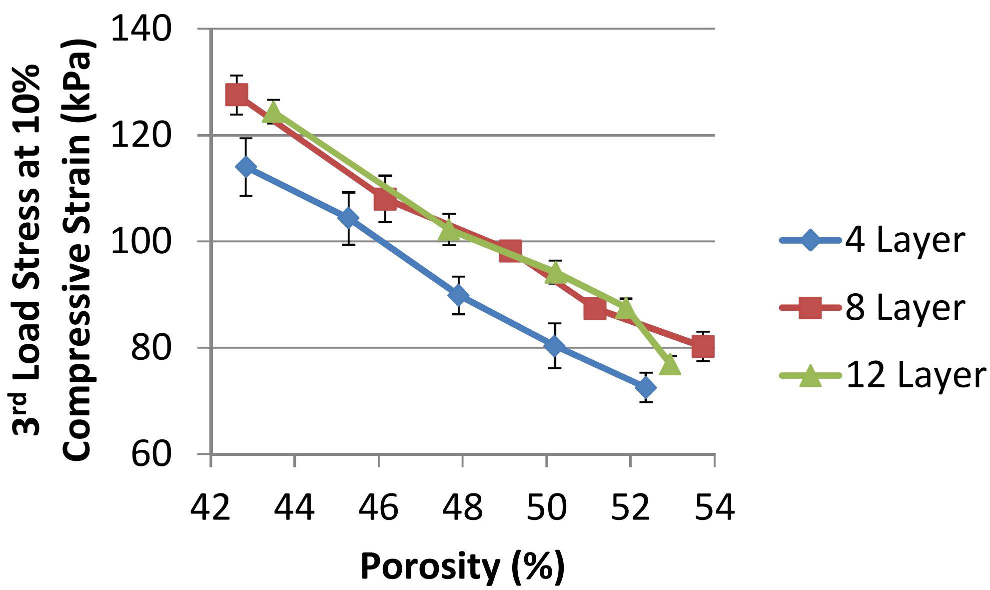
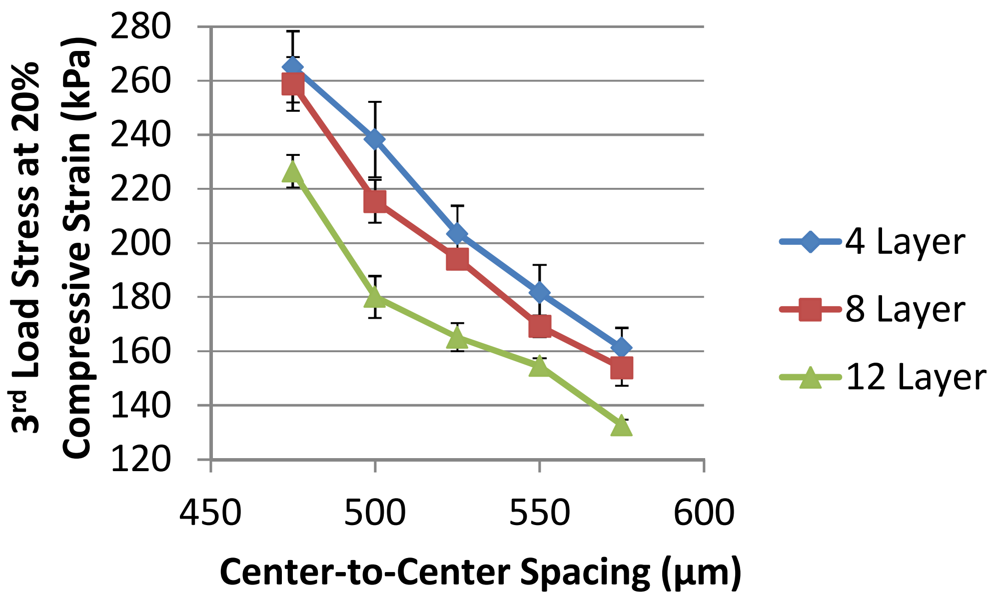


Fig. 4. Third loading stress at 10% strain (engineering values) as a function of (a) center-to-center filament spacing and (b) calculated porosity for the SE 1700 DIW SC samples. Data points represent the average stress and errors bars represent standard deviation (n=12).

(a)



(b)

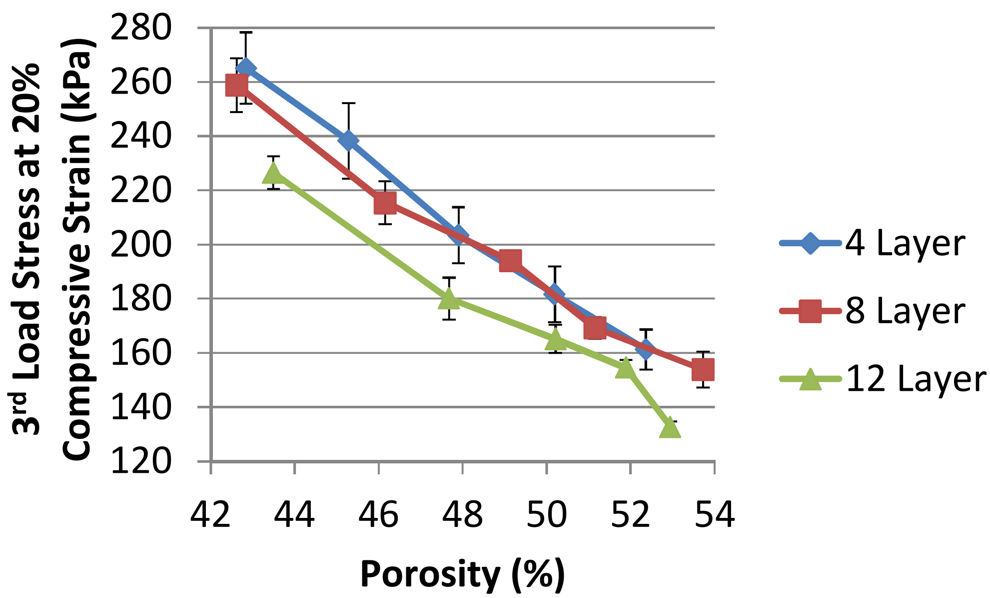
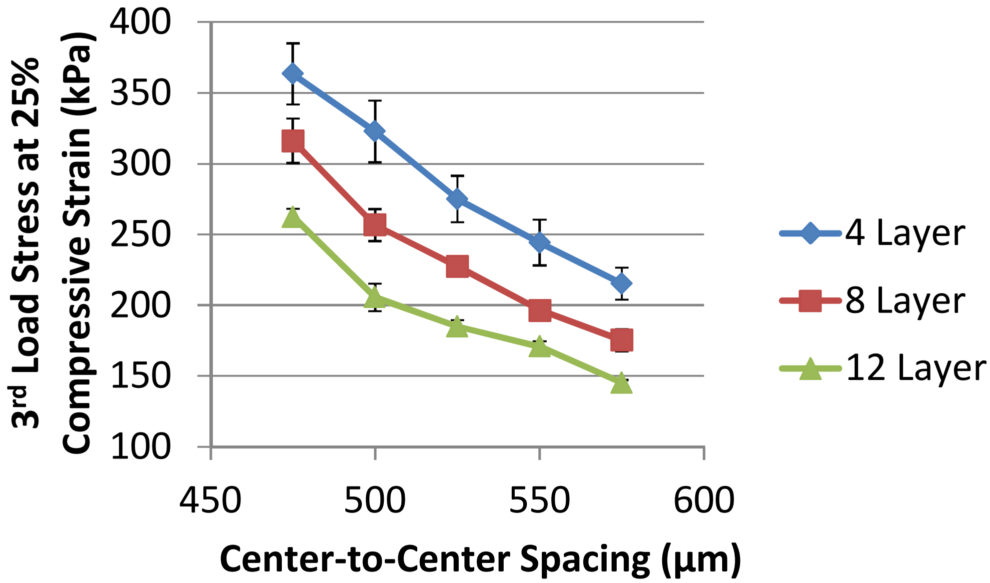


Fig. 5. Third loading stress at 20% strain (engineering values) as a function of (a) center-to-center filament spacing and (b) calculated porosity for the SE 1700 DIW SC samples. Data points represent the average stress and errors bars represent standard deviation (n=12).

(a)



(b)

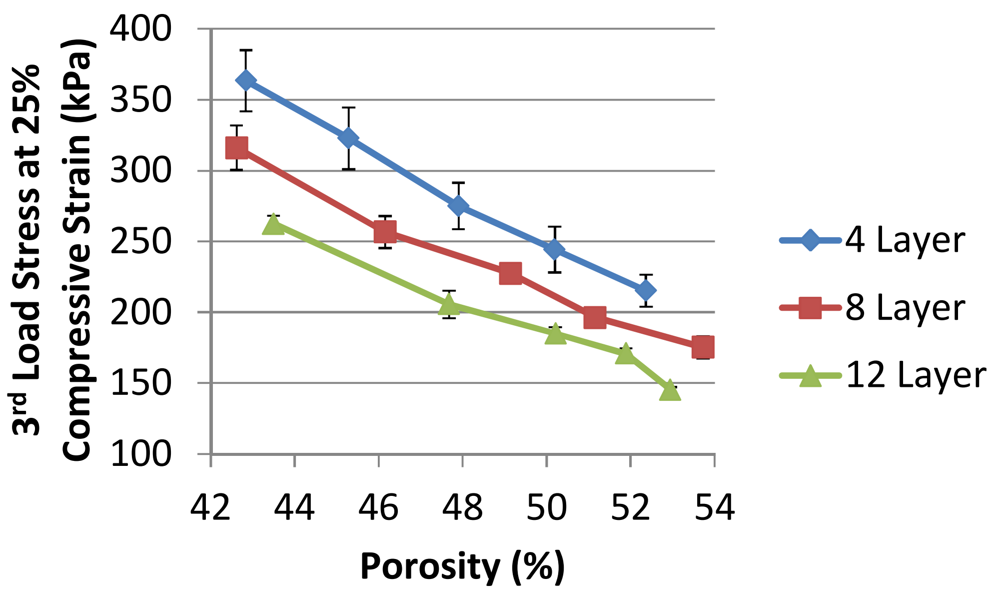
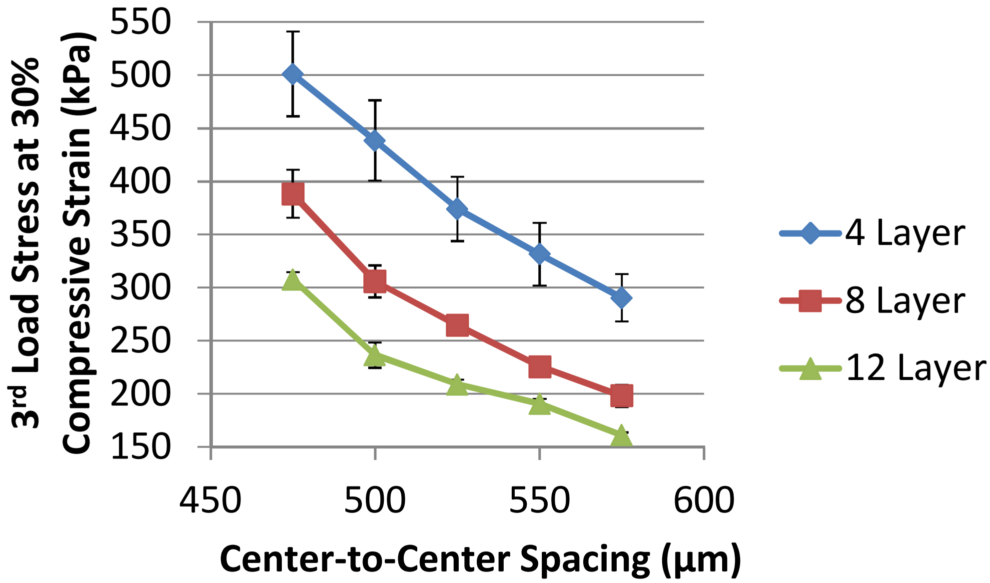


Fig. 6. Third loading stress at 25% strain (engineering values) as a function of (a) center-to-center filament spacing and (b) calculated porosity for the SE 1700 DIW SC samples. Data points represent the average stress and errors bars represent standard deviation (n=12).

(a)



(b)

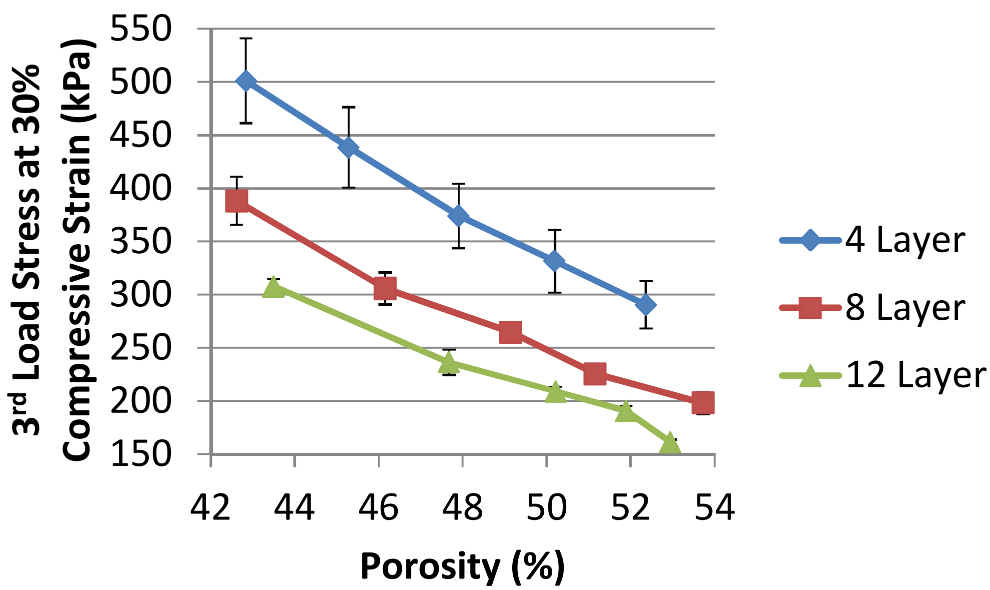
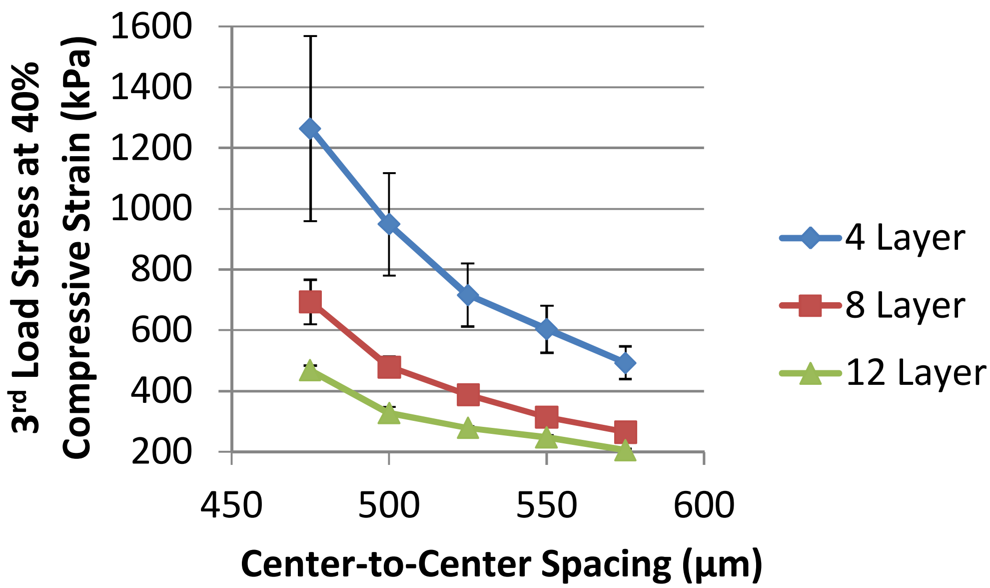


Fig. 7. Third loading stress at 30% strain (engineering values) as a function of (a) center-to-center filament spacing and (b) calculated porosity for the SE 1700 DIW SC samples. Data points represent the average stress and errors bars represent standard deviation (n=12).

(a)



(b)

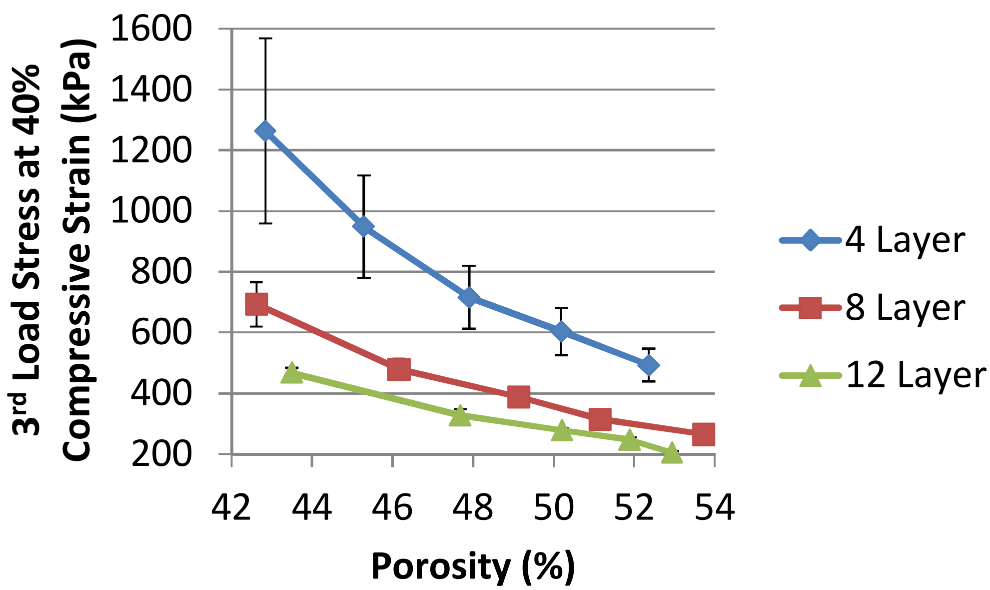


Fig. 8. Third loading stress at 40% strain (engineering values) as a function of (a) center-to-center filament spacing and (b) calculated porosity for the SE 1700 DIW SC samples. Data points represent the average stress and errors bars represent standard deviation (n=12).