



# Polymerization Shrinkage Stresses of Different Flowable and Universal Bulk-Fill Composites



Marlen Robles Moreno<sup>1\*</sup>, Yuwein Fan<sup>2</sup> and Dan Nathanson<sup>2</sup>

<sup>1</sup>Department of Restorative Sciences, The Dental College of Georgia at Augusta University, USA

<sup>2</sup>Department of Restorative Sciences and Biomaterials, Boston University, USA

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\*Corresponding author: Marlen Robles-Moreno, DDS, CAGS, MSD, Former Prosthodontic Resident at Boston University, School of Dental Medicine; present Assistant Professor, Department of Restorative Sciences, The Dental College of Georgia at Augusta University. Georgia, USA, 1430 John Wesley Drive GC 4224, Augusta, GA 30912

## Abstract

**Objective:** To analyze and compare the shrinkage stress properties of flowable and universal bulk-fill resin composites over five minutes after photopolymerization.

**Materials and Methods:** Eleven resin composites, seven flowable and four universals, were tested (n=5). Two glass cylinders were affixed end-to-end in a vertical position on an INSTRON universal testing machine, leaving a 1mm gap between them. Each resin was injected until filling the gap and photopolymerized for 20s. The force curve was recorded over 5 minutes after photopolymerization. The stress values were calculated in MPa at 20s, 60s, 180s, and 300s. Statistical analysis using ANOVA and All-Pairs Tukey-Kramer Test (JMP Pro 12.0. SAS, Cary, NC) were done to compare the polymerization shrinkage stresses among the groups at  $\alpha = 0.05$ .

**Results:** All the resins showed a continuous increase in shrinkage stress up to the 300 seconds, following photopolymerization. Three flowable composites Dentex, G-aenial Universal, and Prime-Dent Flow, obtained the highest shrinkage stresses, while another flowable resin SureFil SDR demonstrated the lowest shrinkage stress from all the materials tested. In general, universal composites demonstrated lower shrinkage stress values than the flowable composites.

**Conclusions:** In general, flowable resin composites presented higher shrinkage stresses compared to universal composites.

## Clinical Significance

Bulk-fill resin composites are widely used clinically, yet little information regarding the polymerization shrinkage stresses produced by these materials is available.

**Keywords:** Resin composites; Polymerization; Shrinkage; Stress; Bulk-fill

## Introduction

It is clinically important to understand how polymerization shrinkage stress affects the interface between resin restorations and tooth structure. These have the potential to damage both enamel and resin composite in the form of cracks, and produce marginal gaps, which can lead to postoperative sensitivity and secondary caries [1,2]. In some cases, fracture lines attributable to polymerization stresses through the enamel cusps have been observed by clinicians following photopolymerization of resins in posterior teeth [3].

Shrinkage is a characteristic of the polymerization of resins, which once constrained by the walls of a cavity preparation will result into shrinkage stress. The amount of material and the material's formulation play important roles in stress development, as well as, in the transmission of these stresses to the tooth structures [4,5]. Any stress that approaches or exceeds the local adhe-

sive force or any structural strength, resulting in gap formation, is considered a high shrinkage stress [6].

In recent years, many manufacturers have introduced "bulk-fill" resin composites that are said to have more light-sensitive photoinitiation systems. There are two types of bulk-fill composites available, low viscosity (flowable) and high viscosity (universal). Studies have shown that low viscosity bulk-fill composites have shrinkage values like regular flowable composites [7-9].

The amount of polymerization stress transferred to the resin/tooth interface is not always proportional to the shrinkage and is influenced by the composition of the resin. While an increase in filler content results in decreased shrinkage, flowable materials with a lower filler content may plastically deform and transfer less stress to margins than stiffer resin composites.

Much of the effort among manufacturers to decrease the shrinkage stresses produced by resin restorations has been based on changing the chemical structure of resin monomers as well as the type and percentage of their fillers [10]. Recent studies of experimental composites have demonstrated that higher shrinkage values and higher polymerization stresses are observed when resin composite viscosity is reduced with increasing ratios of TEGMA/BisGMA [11].

Polymerization shrinkage of resin composites is most commonly measured with the sample unconstrained [12-14], while polymerization shrinkage stresses are usually measured via uniaxial testing in a universal testing machine [15,16], like the method used in this study. In this testing design, the resin composite is shaped into a cylinder between two flat surfaces, one of which is fixed and the other of is attached to the load cell. When the resin composite starts the polymerization, the contraction force pulls the two bonded surfaces together, and the signal of the load cell is monitored continuously by a computer program. Then, the shrinkage stress is calculated by dividing the contraction force by the area of the bonding substrates [17,18].

**Table 1:** Resin composites used in this study.

Material	Manufacturer, City, State, Country of Manufactory & Lot Number	Shade	Monomer By Weight%	Fillers Content By Weight%
Filtek Supreme Ultra-Flow Bulk-Fill Nano composite	3M ESPE, ST Paul, MN. USA Lot N734412	A2	UDMA 525% BisGMA 510% TEGDMA 510%	Silane treated ceramic 50-60% Silane treated silica 5-10%
NovaPro Flow (Nanocomposite with nanofibers)	NANOVA Columbia, MO. USA Lot FC091715B	A2	EBADMA 5-25% UDMA 5-25% Triethyleneglycol Dimethacrylate 5-25%	Hydrophobic Amorphous Fumed Silica 1-10%. Barium, Barosili-cate-glass 40-80% Hydroxyapatite 1-10%
SureFil SDR Flow (Nano-filled)	DENTSPLY Milford, DE. USA Lot 1512091	Universal	Modified UDMA EBPADMA TEGMA	-Barium strontium alumino-fluoro-silicate glasses 68%
G-aenial Universal Flo (Bulk-fill)	GC AMERICA Costa Meza, CA. USA Lot1508072	A2	UDMA, 10-20% BISEMA 5-10% Dimethacrylate 5-10	63.2% (Trade Secret)
Herculite Ultra Flow (Bulk-fill)	KERR Orange, CA. USA Lot 5401009	A2	BisEMA 10-20% Dimethanol dimethacrylate 1-10%	Silicon Dioxide 1-5% ytterbium trifluoride 5-10%
Dentex Flow (Nanocomposite)	SINO-DENTEX Changchun City, Jilin. CHINA Lot 14070303	A1	BisGMA"	Barium Glass Nano Silica 34%
Prime Dent Flowable	PRIME DENTAL, Chicago, IL. USA Lot QF295	A3	BisGMA 20-40%"	(not specified on MSDS by the company)
Filtek Supreme Ultra Universal (Bulk fill)	3M ESPE ST Paul, MN. USA Lot N679748	A2	BiseMA6, 5-15% UDMA 5-10% BisGMA 1-10% EDMAB <0.5%	Silane Treated Ceramic 65-75% Silane Treated Silica 5-15 Diphenyliononium Hexafluorophosphate <0.5% Zirconia/silica clusters
Beautifil II Universal (Nanofilled)	SHOFU DENTAL CORP, Higashiya-ma-Ku JAPAN Lot091471	A2	BisGMA 7.5% TriethylenglycolDimethacrylate <5%	Aluminofluoro-borosilicate Glass 70%
NovaPro Fill Universal (Nano-filled composite)	NANOVA Columbia. MO. USA Lot UC321	A2	UDMA 1-10% EBADMA 5-25%	Hydrophobic Amorphous Fumed Silica 1-10% Barium, Barosili-cate-glass 40-80% Hydroxyapatite 1-10%

Despite efforts to document the chemical and mechanical properties of resin composites, there is little information regarding the polymerization shrinkage stress produced by the recent class of bulk-fill resin composite materials. Therefore, the purpose of this study is to analyze and compare the different polymerization shrinkage stress properties and characteristics of some recently-introduced commercial flowable and universal bulk-fill resin composites.

**Materials and Methods**

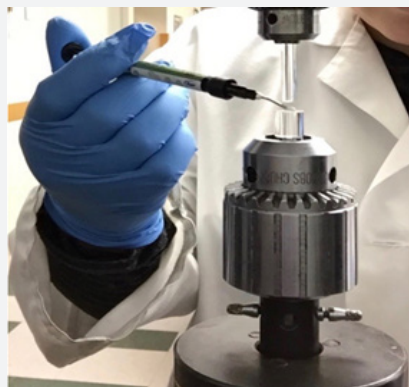
The shrinkage stress properties of eleven resin composites were examined. Seven flowable resins: Filtek Supreme Ultra Flow, NOVAPRO Flow, SureFil SDR, G-aenial Universal Flo, Herculite Ultra Flow, Dentex Flow and Prime Dent Flowable. And four universal resin composites: Filtek Supreme Ultra, NOVAPRO Fill, Beautifil II, and finally, Herculite Ultra. Five specimens from each group (n=5) were tested for polymerization shrinkage.

The composition, manufacturer, place of manufacture, and the LOT number for all included resin composites tested in this study are presented on Table 1.

Herculite Ultra Universal (Bulk fill)	KERR Orange, CA. USA Lot 5302358	B1	2,2bis (acryloyloxymethyl) butyl acrylate 3 trimethoxysilylpropyl methacrylate 1-5%	Glass-Oxide Chemicals 30-60% -Silanamine, 1,1,1-trimethyl-N-(trimethylsilyl)-, hydrolysis products w/ silica 10-30%
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EBADMA: Ethoxylated BisPhenol A Dimethacrylate, UDMA: urethane dimethacrylate, Bis-GMA: bisphenol A glycidyl methacrylate, Bis-EMA: ethoxylated bisphenol A glycol dimethacrylate, EDMAB: ethyl-4-dimethylaminobenzoate. BISEMA6: diether Dimethacrylate. (Information disclosed by manufacturers on MSDS).

A universal mechanical tester Instron Model 5566A (Instron Corp., Norwood, Massachusetts, USA) was used to analyze the polymerization shrinkage stress.



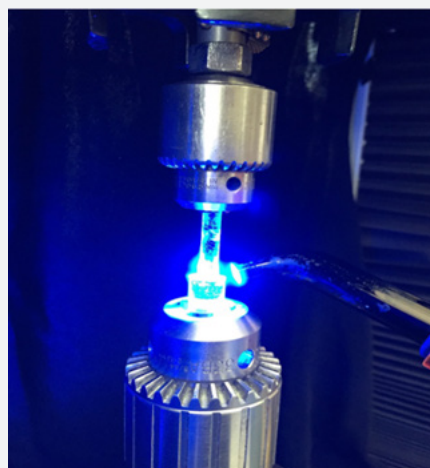
**Figure 1:** The resin composite was injected on the flat surface of the lower glass cylinder, until filling the 1mm space.



**Figure 2:** The gap was adjusted at 1mm using the crosshead controller and the excess of composite was cleaned off.

The load of contraction force was recorded from 100N load cell. One fused silica cylinder with 10mm in diameter and one glass cylinder 5mm in diameter, had one of their flat ends treated with 100 grit sand paper to obtain a rough surface necessary for resin composite bonding. The surfaces were then rinsed and air dried, the two rods were fixed in a vertical position with two chucks attached to the universal testing machine. The working load cell was mounted at the top. A bonding agent (Optibond Solo Plus, Kerr Restoratives Orange, CA.) was applied on the surface of the lower glass cylinder, and light cured for 10 seconds with a light curing diode (10 Wats). (Blue phase 16i, Ivoclar, HIP mode, ~1200mW/cm<sup>2</sup>). The upper and lower glass cylinders were placed into close contact together, this contact was controlled by

a minimal load recorded by software from the Instron computer. The distance between the upper and lower cylinders was set to 1.5-1.8mm. The resin composite was then injected on the flat surface of the lower glass cylinder, until filling the 1mm space (Figure 1). The 1mm space was adjusted using the crosshead controller. The excess of composite was cleaned off (Figure 2).



**Figure 3:** The tip of the curing light was placed perpendicular to the junction at a distance of 2mm.

After a 30 seconds baseline was acquired, the resin was photo initiated for 20 seconds (Figure 3). The tip of the curing light was placed perpendicular to the junction at 2mm, approximately. A radiometer, Demetron Model 100 (Demetron Research Corp. Danbury, CT) was used to monitor the adequacy of the curing light throughout the entire experiment. Finally, the peak load vs. time curve was recorded by the attached 100N load cell in Bluehill 3 software until five minutes (300s) after the photo-initiation and calculated into polymerization shrinkage stress.

Five specimens for each resin were analyzed following the same method described (n=5). The shrinkage stress values of each specimen were calculated in MPa from the division of contraction force recorded at 20s, 60s, 180s, and 300s. The entire experiment was conducted at room temperature of 73° F.

Statistical analysis of the data was performed using the JMP Pro 12.0.1. SAS, Cary, NC software. One-way ANOVA and All-Pairs Tukey-Kramer Test were used to compare the polymerization shrinkage stress differences among the groups with  $\alpha = 0.05$ .

### Results

All the resins evaluated in this study showed a continuous increase in shrinkage stress over the 300 seconds, following photopolymerization. Three flowable resin composites Dentex Flow, G-aenial Universal Flow, and Prime-Dent Flow, demonstrated sig-

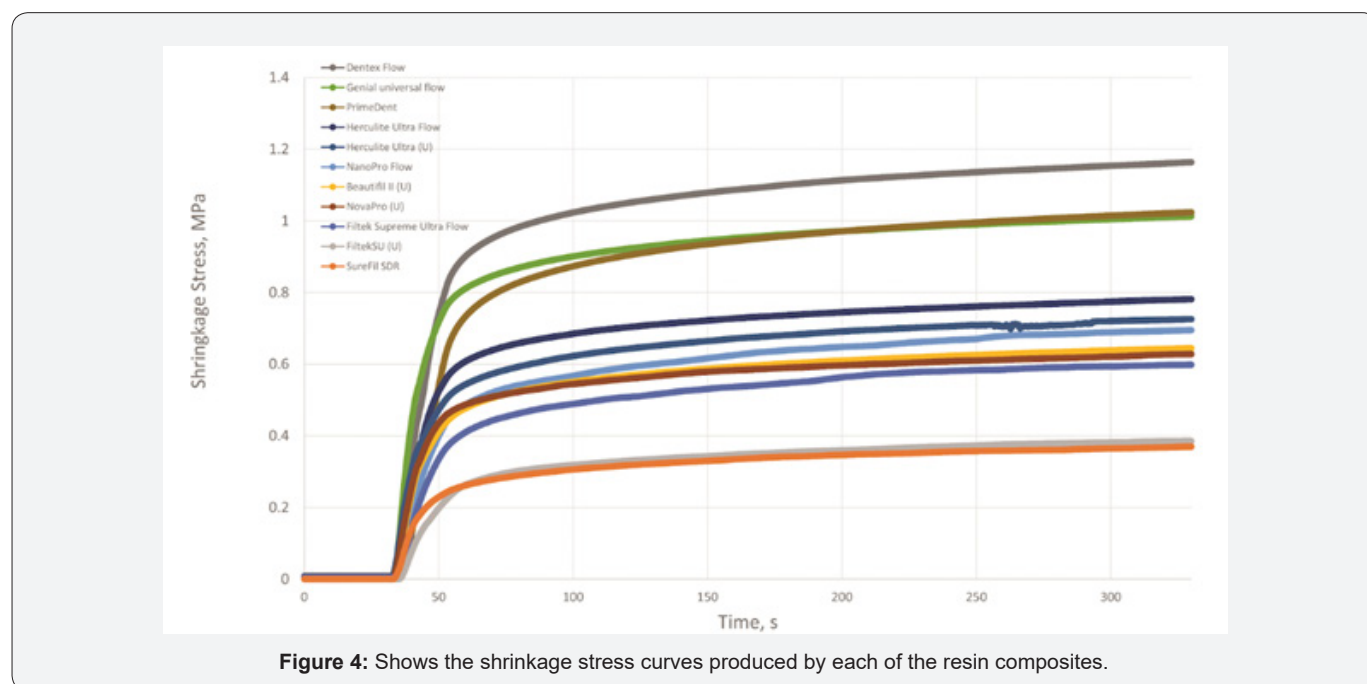
nificantly higher levels of shrinkage stress than the other materials, while another flowable resin composite (SureFil SDR-flow) demonstrated the lowest polymerization shrinkage stress from all

the materials tested in this study. In general, universal resin composites demonstrated lower polymerization stress values than the flowable resin composites.

**Table 2:** Mean shrinkage stress and MPa's at 5 minutes after photo-polymerization.

Composite Material	Mean Shrinkage Stress, MPa	Significance *			
Dentex Flow	1.12±0.09	A			
Genial Universal Flow	0.90±0.06		B		
Prime Dent Flow	0.85±0.07		B		
Herculite Ultra Flow	0.66±0.08			C	
Novapro Flow	0.66±0.02			C	
Herculite Ultra Universal	0.65±0.05			C	
Beautiful II Universal	0.57±0.05			C	
Novapro Universal	0.55±0.03			C	
Filtek Sup. Flow	0.55±0.03			C	
Filtek Sup. Ultra	0.37±0.02				D
Sure Fil SDR	0.33±0.03				D

\*Levels not connected by same letter are significantly different.



**Figure 4:** Shows the shrinkage stress curves produced by each of the resin composites.

The mean shrinkage stress values and MPa's for all the resin composites at different times are presented in Table 2 and Figures 4 & 5. Table 2 shows the mean polymerization shrinkage stresses, and four overlapping Tukey groups categorizing the shrinkage stresses, for each resin composite until five minutes after polymerization, which was the peak stress in each case. Figure 4 shows the polymerization shrinkage stress curves produced by each of the materials over the entire five minutes. Figure 5 shows the shrinkage stresses of the resins at different times in bar graph form, with the materials grouped into flowable and universal resin composites.

Pooled data demonstrated that the flowable composites group presented higher polymerization shrinkage stress values in gen-

eral. However, SureFil SDR flow had the lowest values among both universal and flowable resin composites groups. The universal resin with the lowest shrinkage stress values was Filtek Supreme Ultra.

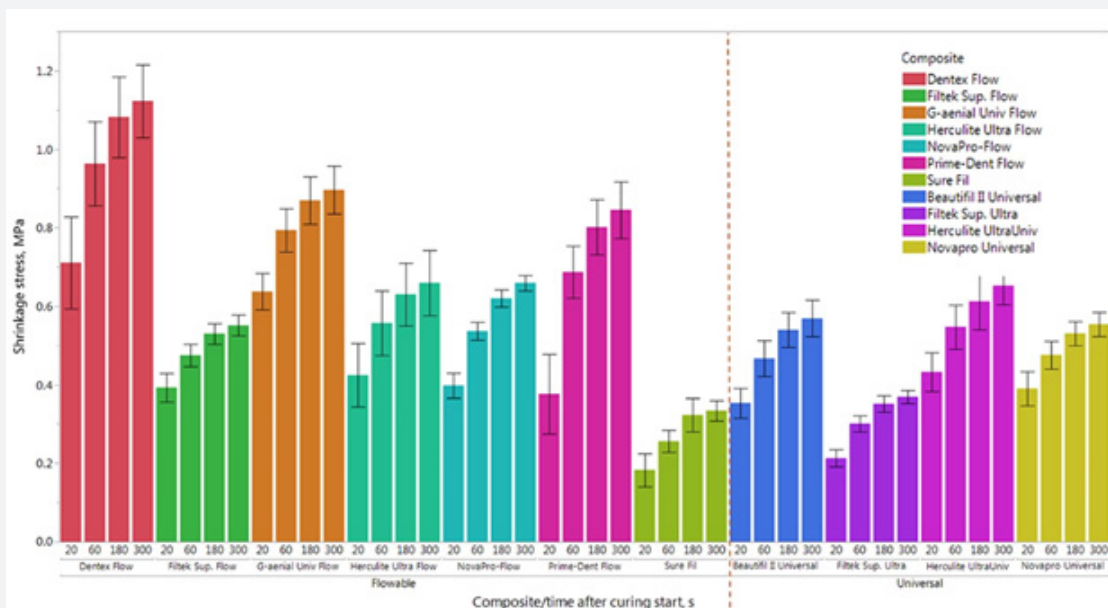
### Discussion

Malhotra et al. have concluded that it is important to minimize the polymerization stress in order to improve the survival and success rate of resin restorations [19]. For this reason, effective methods of investigating the polymerization shrinkage stress properties of resin composite materials are necessary to predict their clinical behavior. The use of a universal testing machine with either extensometers that register movement caused by polymerization stresses or load cells which measure the forces produced

has been widely used in various studies before [5,16,19-24]. One limitation of these techniques is that they record stress along one axis, whereas stresses occur along three axes [25,26].

In this study, all the resins had differing levels of polymerization shrinkage stress. Moreover, the highest shrinkage stress values among all tested materials was observed with Dentex Flow (Sino-Dentex), this could be a result of filler content as low as 34% w/w and use of monomers such as Bis-GMA, which produces a stiffer polymer chain that is unlikely to undergo plastic deformation and dissipate shrinkage stresses.

The resin composite demonstrating the lowest polymerization shrinkage stress, SureFil SDR flow (Stress Decreasing Resin, Smart Dentin Replacement), is filled 68% w/w, but employs a modified urethane dimethacrylate monomer, which produces a more flexible polymer chain and is said by the manufacturer to polymerize more slowly, allowing the resin to dissipate more stress by plastic deformation. The results of this study on polymerization shrinkage stress of SureFil SDR flow are confirmed in a prior study by Manhart J et al. [27] and Abe Y et al. [28].



**Figure 5:** Bar graph shows the polymerization shrinkage stresses at different times, the groups were divided into flowable and universal composites.

Based on the results of this study, the authors believe that the capabilities of the resin composites in preventing the tooth margins from polymerization shrinkage stresses is product specific and does not result solely from the consistency of the material. Thus, it is important for a clinician to learn about the chemical properties of the resin composites of their choice to understand and predict its clinical behavior [29-31].

### Conclusion

In general, flowable resin composites demonstrated higher polymerization shrinkage stresses values than universal resin composites, except for SureFil SDR flow showed the lowest polymerization shrinkage stress properties among all the materials tested. With the recent demand of flowable bulk-fill resin composites, among dental professionals, an increase of research is necessary in order to compare their different properties and understand how these may clinically affect the longevity of the restorations.

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