

**SETTING SHRINKAGE STRESS OF
FIVE DIFFERENT BULK FILL COMPOSITES**

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Introduction

This study concerns polymerization shrinkage stress measurements on the composite ELS Bulk Fill Flowable (ELS BF F), Venus Universal Bulk Fill Flowable (Venus U BF F), X-tra Base Flowable (X-tra Base F), Filtek Bulk Fill (Filtek BF), and Tetric EVO Ceram Bulk Fill (Tetric EvoC BF) with the ACTA tensiometer. The materials were received in April 2015. The experiments were performed in the periods as mentioned in Table 1.

Materials and methods

Table 1. Materials tested for shrinkage stress.

Material	Manufacturer	Test period	Batch/shade/exp date
ELS Bulk Fill Flowable	Saremco	Mai-Jun 2015	C348//01.2019
Venus Universal Bulk Fill Flowable	Heraeus	Mai-Jun 2015	10106//01.2018
X-tra Base Flowable	VOCO	Mai-Jun 2015	1444571/U/01.2017
Filtek Bulk Fill	3M ESPE	Mai-Jun 2015	N633572/A2/09.2017
Tetric EVO Ceram Bulk Fill	Ivoclar Vivadent	Mai-Jun 2015	T02443/Bulk IVA/01.2018

Setting shrinkage stress measurements

The test setup shown in figure 1 was placed in an Instron 6022 Tensiometer. Composite paste was inserted between the glass plate and the flat surface of the steel bolt head and adhered to both these surfaces. During light curing and a period of 30 minutes following, the shrinkage stress development was measured, while the distance between the glass and the steel bolt head was kept constant. This simulated a restoration in a fully rigid situation where the cavity walls cannot yield to the contraction forces.

Technical procedures

Preparation of surface of the glass plate:

The glass plate (4 mm thick) was glued to a stainless steel tube (inner diameter approx. 3 cm), which had an outward speed to enable to screw it into the platform of the setup. The upper glass surface, on the spot where the specimen had to be adhered, was sandblasted with Al₂O₃ (50 µm) until an even “frosted” surface developed. Remaining Al₂O₃ was

removed by compressed air. One drop of Ceramic Primer (3M ESPE) was applied to this frosted surface and the solvent gently evaporated by an airflow. A small amount of Scotchbond MP resin (3M ESPE) was blown out over the surface into a thin layer and light cured for 20 seconds. The steel tube with the glued glass plate was then mounted in the platform of the setup and fixed with a lock nut.

Preparation of the bolt head:

The bolt head (diameter $D = 3.2$ mm) was wet-ground on 600 grit SiC sandpaper and then sandblasted with Al_2O_3 (50 μm), rinsed with acetone and treated in a Silicoater (Kulzer) to deposit a thin silica layer. A drop of fresh Silicoup (Kulzer) was applied to silanize the surface. After drying a thin layer of Scotch Bond MP (3M Espe) was blown out over the surface, and light cured for 20 seconds. Finally the bolt was fixed in the setup of the tensiometer.

Insertion of the composite and activation:

The distance between the surfaces of the glass plate and the bolt head was adjusted to 0.8 mm, which had to become the specimen thickness (H) in the experiments. Together with the specimen diameter (D) a C-value of $C = D/2H = 3.2/1.6 = 2$ was obtained (Feilzer *et al. J Dent Res* **66**:1636-9, 1987). The LDTV's (probes) of the tensiometer were reset to zero (zero position) and the crosshead of the tensiometer was lifted to enable to apply a small amount of composite paste on the bolt head. Then the crosshead was returned to its zero position. Excess of composite was removed with a spatula. The specimens were light cured through the glass with an Elipar Highlight (Espe) for 40 seconds in Standard mode (750 mW/cm^2).

Measurement

From the start of light curing the shrinkage stress development was measured during 30 minutes. The axial contraction of the specimens was continuously counteracted by a feedback displacement of the crosshead to keep the thickness of the specimen constant. The average shrinkage stress of each of the composites in table 1 was determined from five measurements ($n = 5$).

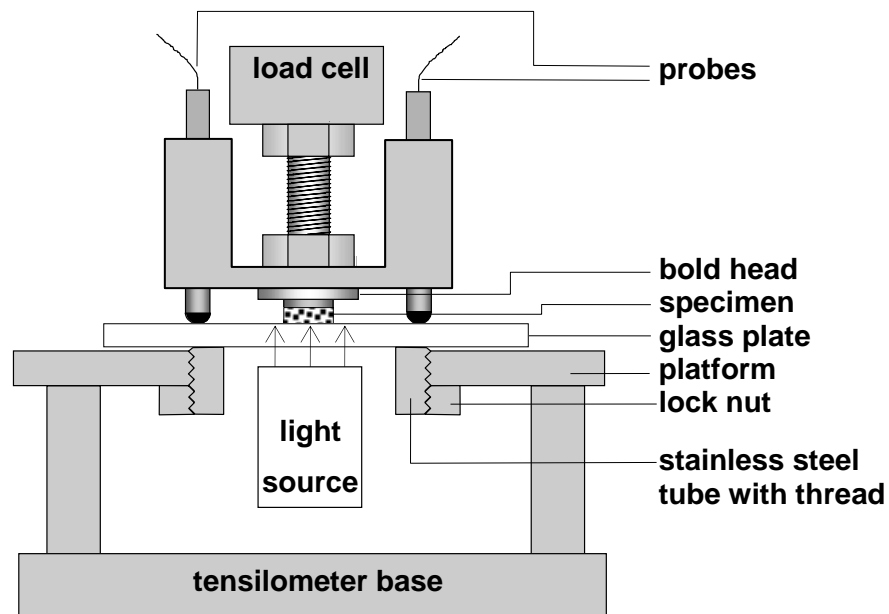


Figure 1. Setup in the tensiometer. The specimen was bonded with a very thin layer of Scotchbond MP resin (3M) to the surface of the bold head (silica coated and silanated) and the surface of the glass plate silanated with Ceramic Primer (3M).

Results

The results of the shrinkage stress development are given graphically in Figure 2 representing the measurement period of 30 minutes and in Figure 3 representing the first 60 seconds of curing. Table 2 shows some numeric data at various time moments.

Table 2. Polymerization shrinkage stress (MPa) at some selected time moments of the materials light cured with the Elipar Highlight for 40 seconds in standard mode (750 mW/cm²). Standard deviations between brackets (n = 5).

seconds	ELS BF F	Venus U BF F	X-tra Base F	Filtek BF	Tetic-EvoC BF
1	0.0 (0.0)	0.0 (0.0)	0.1 (0.1)	0.0 (0.0)	0.0 (0.0)
5	0.2 (0.1)	0.6 (0.4)	0.7 (0.2)	0.9 (0.6)	1.2 (0.5)
10	0.5 (0.1)	2.3 (0.9)	3.8 (0.7)	2.7 (1.2)	2.8 (1.0)
15	1.0 (0.1)	3.4 (0.9)	6.4 (0.9)	4.1 (1.3)	4.0 (0.9)
30	1.5 (0.1)	4.3 (0.8)	7.8 (1.1)	5.6 (1.5)	5.1 (0.9)
60	1.6 (0.2)	4.6 (0.7)	8.3 (1.1)	6.3 (1.6)	5.7 (0.9)
180	1.7 (0.2)	4.9 (0.7)	8.6 (1.1)	6.7 (1.7)	6.0 (0.9)
240	1.8 (0.1)	5.0 (0.7)	8.8 (1.1)	7.0 (1.7)	6.3 (0.9)
300	1.9 (0.1)	5.2 (0.7)	9.0 (1.1)	7.2 (1.8)	6.4 (0.9)
600	2.1 (0.2)	5.6 (0.8)	9.6 (1.1)	7.9 (1.8)	7.1 (0.9)
900	2.2 (0.2)	5.9 (0.8)	10.0 (1.2)	8.3 (1.9)	7.5 (0.9)
1200	2.3 (0.2)	6.1 (0.8)	10.3 (1.2)	8.6 (1.9)	7.8 (0.9)
1500	2.4 (0.2)	6.2 (0.8)	10.5 (1.2)	8.9 (1.9)	8.0 (0.9)
1800	2.4 (0.2)	6.3 (0.8)	10.7 (1.2)	9.1 (2.0)	8.2 (1.0)

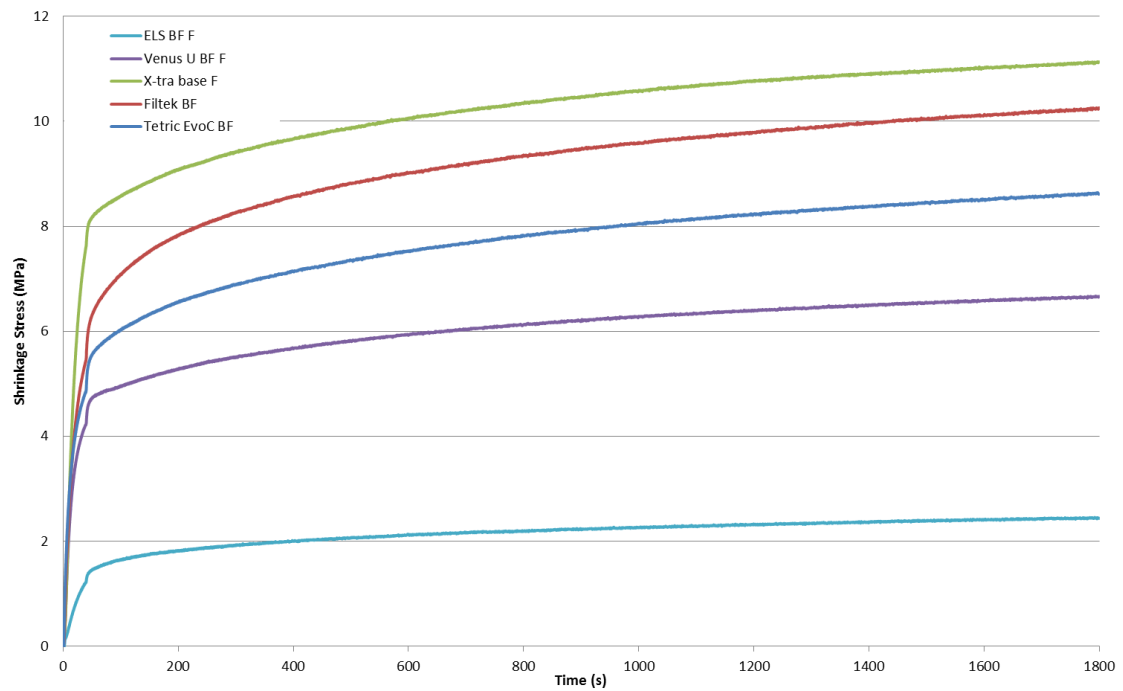


Figure 2. Polymerization shrinkage stress development (MPa) of the investigated materials ($n = 5$) during 30 minutes of setting after the start of light curing with the Elipar Highlight for 40 seconds in standard mode (750 mW/cm^2).

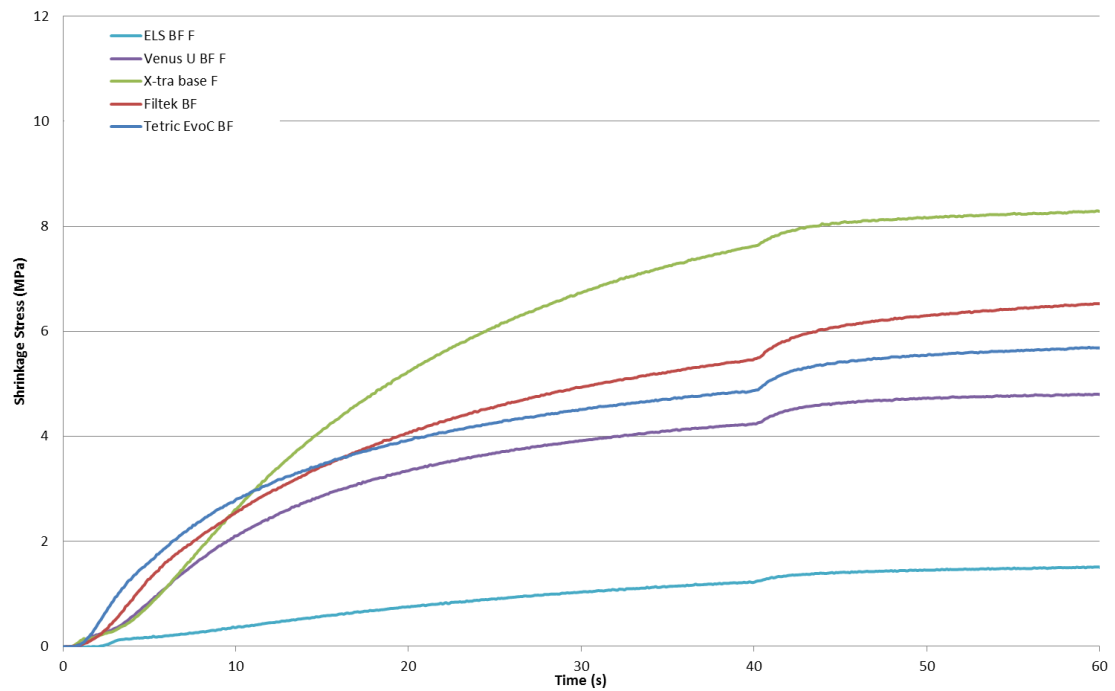


Figure 3. Polymerization shrinkage stress development (MPa) of the investigated materials (n = 5) during the first 60 seconds of setting after the start of light curing with the Elipar Highlight for 40 seconds in standard mode (750 mW/cm²).