

Introduction

This study concerns polymerization shrinkage stress measurements on the polymers ELS Flow, Ice, Glacier, Premise Dentin, Ceram.X Mono and Charisma. Sections given the date by Royal of December 11, 2006 and Charisma was approved on December 11, 2006. The experiments were performed on January 4, 2006.

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Project number P2006.003 Shrinkage Stress

February 16, 2006

Material	Manufacturer	Time period	Reference
ELS Flow Composite	Saremco	Jan - Feb 2006	03-03-13-2006
Ice	SCH	Jan - Feb 2006	03-03-13-2006
Glacier	SCH	Jan - Feb 2006	03-03-13-2006
Premise Dentin Composite	Kerr	Jan - Feb 2006	01-03-2006
Ceram.X mono	Dynafly	Jan - Feb 2006	04-03-2006
Charisma	Heraeus	Jan - Feb 2006	01-03-2006

By order of Saremco

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Setting shrinkage stress measurements

The test setup shown in figure 1 was placed in an Instron 6022. The composite was placed on a glass plate (4 mm thick) which was fixed to a steel ball head and adhered to both these surfaces. The shrinkage stress measurement was performed during a period of 30 min following the curing. The shrinkage stress was measured, while the distance between the glass and the steel ball head was kept constant. The measurement is a fully rigid situation where the cavity walls cannot yield to the contraction forces.

Signature:



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Introduction

This study concerns polymerization shrinkage stress measurements on the composites ELS Flow, Ice, Glacier, Premise Dentin, Ceram.X mono and Charisma. Saremco gave the order by E-mail of December 9, 2005. Premise Dentin, Ceram.X mono and Charisma were received on December 21, 2005. ELS Flow, Ice and Glacier were received January 2006. The experiments were performed in the periods as mentioned in table 1.

Materials and methods

Table 1. Materials tested for polymerization shrinkage stress.

Material	Manufacturer	Test period	Batch/Shade/Exp date
ELS Flow Opaque	Saremco	Jan - Feb 2006	05/A3/12-2009
Ice	SDI	Jan - Feb 2006	0506129/A3/2008-06
Glacier	SDI	Jan - Feb 2006	0505101/A3/2010-05
Premise Dentin Opaque	Kerr	Jan - Feb 2006	014338/A3/2007-07
Ceram.X mono	Dentsply DeTrey	Jan - Feb 2006	0409000316/M3/2006-07
Charisma	Heraeus Kulzer	Jan - Feb 2006	010079/A3/2008-03

Setting shrinkage stress measurements

The test setup shown in figure 1 was placed in an Instron 6022 Tensilometer. 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 min 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) was blown out over the surface into a thin layer and light cured for 20 sec. 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 ($50 \mu m$), 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 s in Standard mode (750 mW/cm^2).

Measurement

From the start of light curing the shrinkage stress development was measured during 30 min. 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 three measurements ($n = 5$).

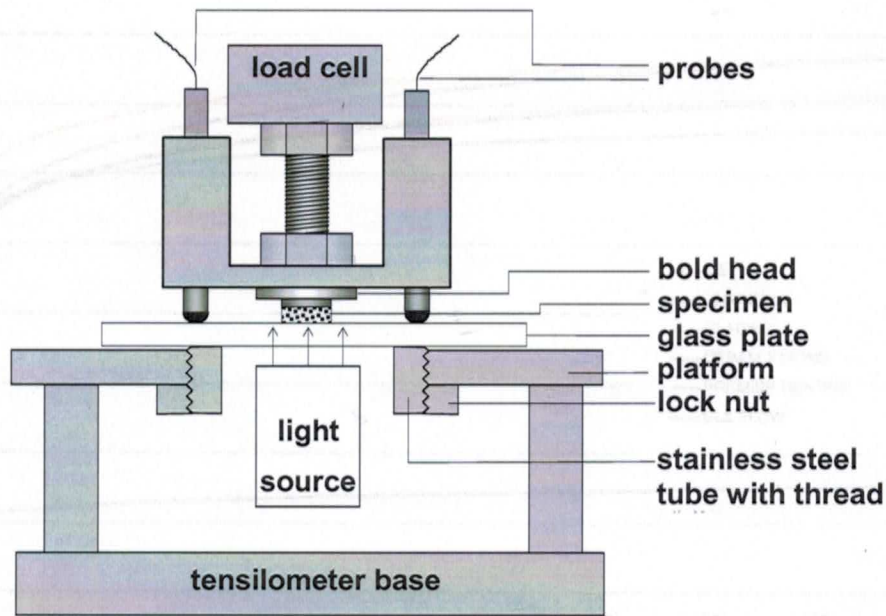


Figure 1. Setup in the tensiometer. The specimen was bonded with a very thin layer of Scotchbond MP resin (3M) to the surface of the bold head (silica coated and silanated) and the surface of the glass plate silanated with Ceramic Primer (3M).

Results

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

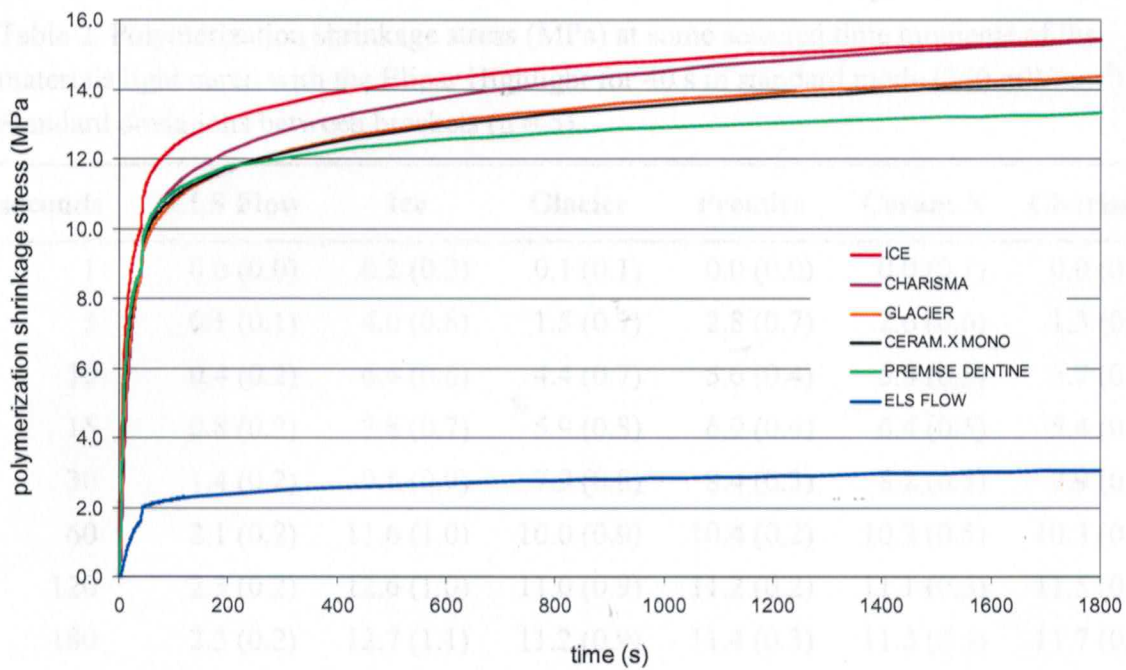


Figure 2. Polymerization shrinkage stress development (MPa) of the investigated materials (n = 5) during 30 minutes of setting after the start of light curing with the Elipar Highlight for 40 s in standard mode (750 mW/cm²).

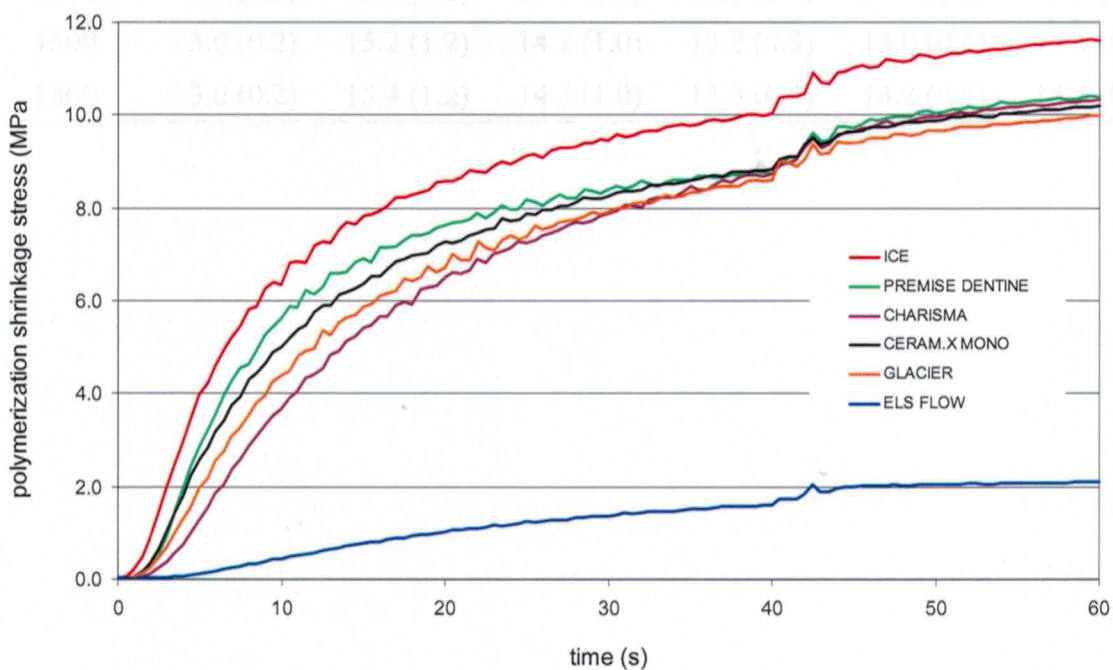


Figure 3. Polymerization shrinkage stress development (MPa) of the investigated materials (n = 5) during the first 60 seconds of setting after the start of light curing with the Elipar Highlight for 40 s in standard mode (750 mW/cm²).

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

seconds	ELS Flow	Ice	Glacier	Premise	Ceram.X	Charisma
1	0.0 (0.0)	0.2 (0.2)	0.1 (0.1)	0.0 (0.0)	0.0 (0.1)	0.0 (0.0)
5	0.1 (0.1)	4.0 (0.8)	1.5 (0.7)	2.8 (0.7)	2.6 (0.6)	1.3 (0.4)
10	0.4 (0.2)	6.4 (0.6)	4.4 (0.7)	5.6 (0.4)	5.0 (0.5)	3.7 (0.5)
15	0.8 (0.2)	7.8 (0.7)	5.9 (0.8)	6.9 (0.4)	6.4 (0.5)	5.4 (0.6)
30	1.4 (0.2)	9.5 (0.9)	7.9 (0.8)	8.4 (0.3)	8.2 (0.5)	7.9 (0.9)
60	2.1 (0.2)	11.6 (1.0)	10.0 (0.9)	10.4 (0.2)	10.2 (0.5)	10.3 (0.9)
120	2.3 (0.2)	12.6 (1.0)	11.0 (0.9)	11.2 (0.2)	11.1 (0.5)	11.5 (0.9)
180	2.3 (0.2)	12.7 (1.1)	11.2 (0.9)	11.4 (0.3)	11.3 (0.5)	11.7 (0.9)
240	2.4 (0.2)	13.1 (1.1)	11.7 (0.9)	11.7 (0.2)	11.7 (0.5)	12.2 (0.9)
300	2.5 (0.2)	13.4 (1.1)	12.0 (1.0)	11.9 (0.2)	12.0 (0.5)	12.6 (0.9)
600	2.7 (0.2)	14.2 (1.1)	12.9 (1.0)	12.5 (0.3)	12.9 (0.6)	13.7 (0.9)
900	2.8 (0.2)	14.7 (1.1)	13.5 (1.0)	12.8 (0.3)	13.4 (0.6)	14.3 (0.9)
1200	2.9 (0.2)	15.0 (1.1)	13.8 (1.0)	13.0 (0.4)	13.7 (0.6)	14.8 (0.9)
1500	3.0 (0.2)	15.2 (1.2)	14.1 (1.0)	13.2 (0.3)	14.0 (0.6)	15.1 (0.9)
1800	3.0 (0.2)	15.4 (1.2)	14.3 (1.0)	13.3 (0.3)	14.2 (0.6)	15.3 (0.9)