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# Shrinkage stress kinetics of Bulk Fill resin-based composites at tooth temperature and long time

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## ABSTRACT

**Objective.** To determine the shrinkage stress kinetics at up to 12 h after light exposure and at tooth temperature during placement of selected Bulk Fill resin-based composites (RBCs). **Methods.** Five representative Bulk Fill RBCs from four companies were chosen with a wide range of viscosity and filler volume content. The shrinkage stress kinetics at  $T = 33^\circ\text{C}$  was measured continuously over a period of 12 h using a modified tensometer with the ability to measure the cantilever beam deflection to better than 40 nm accuracy at a sampling rate of up to 200 samples/s, and thermally stable resulting in a measurement accuracy better than 0.05 MPa at 12 h. The tensometer compliance was  $0.105 \mu\text{m}/\text{N}$ . A custom made heater was used to control the RBC sample temperature at  $T = 33^\circ\text{C}$  with a temperature gradient across the sample of less than  $1^\circ\text{C}$ . The samples were irradiated for 20 s with irradiance of  $1.1 \text{ W}/\text{cm}^2$  and total energy density of  $22 \text{ J}/\text{cm}^2$ . Three samples ( $n = 3$ ) were used for each RBCs.

**Results.** The shrinkage stress at 12 h for the five Bulk Fill RBCs ranged from 2.21 to 3.05 MPa, maximum stress rate  $((dS/dt)_M)$  varied from 0.18 to 0.41 MPa/s, time at which the maximum stress rate occurred ( $t_{\text{Max}}$ ) were between 1.42 to 3.24 s and effective gel time ( $t_{\text{gel}}$ ) varied from 50 to 770 ms. Correlations were observed between  $(dS/dt)_M$  and  $t_{\text{Max}}$  ( $r = -0.946$ ),  $t_{\text{Max}}$  and filler volume fraction ( $r = -0.999$ ), and between the shrinkage stress at 12 h and  $t_{\text{gel}}$  ( $r = 0.994$ ). However, no correlation was observed between the stress at 12 h and filler volume fraction. **Significance.** The shrinkage stress for four of the five Bulk Fill RBCs were not significantly different ( $p < 0.05$ ) at 6 h and beyond after photo-curing and that fully developed stress induced by photo-cured RBCs may only be reached at times longer than 12 h.

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## 1. Introduction

Since the introduction of resin-based composites (RBCs) by Bowen in 1958 [1] as an alternative to dental amalgam toward

tooth restorations, tremendous research and development activities have followed [2–4]. The primary motivation for such activities is the esthetic restoration of the tooth to its original appearance with a long clinical life. Recently, developments in novel monomers, monomer blends and filler

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nanotechnology [5–10], photoinitiators [11–14], and improved RBC transparency to blue light [15,16] allow the placement and photocuring of up to 4 mm thick increment of “Bulk Fill” RBCs in the restoration [17–21]. Despite such improvements over a 50 year period, the fundamental property of shrinkage strain of the RBC upon photocuring and its links on the viability of the restoration remains [22]. The shrinkage strain and resulting development of built in stress on the tooth structure may result in cuspal deflection, marginal gap formation and leakage, enamel cracking and may eventually lead to restoration fracture, secondary caries, and ultimate clinical failure of the restoration [23].

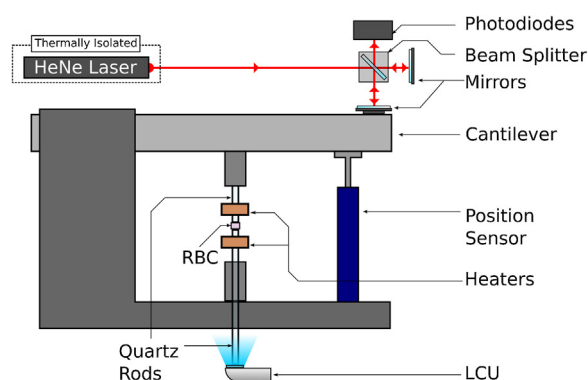
The issue of shrinkage stress on the tooth walls was identified and two general approaches were introduced to quantify its impact [24–33]. The ratio of the bonded to unbonded surface area of the RBC cylindrical shaped sample, the C-factor, was observed to play an important role toward the determination of the final stress value [31,34]. Its value was found to decrease with decreasing C-factor under otherwise the same experimental conditions. These results were interpreted in terms of RBC flow and strain relaxation during the pre-gel phase of the curing RBC sample. These results pointed out the role of the gel time and RBC viscosity during the development of the shrinkage induced stress where low viscosity flowable RBCs would favor strain relaxation via flow at the beginning of photo-polymerization before the gel time than highly viscous RBCs. In recent studies [35–37], the shrinkage stress kinetics was measured on conventional RBCs at both room and clinical mouth temperature and over a period of up to 1 h after light exposure [36]. It was observed that for all six RBCs tested [36] the mean shrinkage stress was significantly higher ( $p \leq 0.007$ ) at 60 min compared to that at 2 min after light exposure at 23 °C while the mean shrinkage stress was not significantly different ( $p \geq 0.112$ ) at both times at 37 °C. Shrinkage stress studies were performed on “Bulk Fill” RBCs at room temperature [17,18,38–40] with detailed analysis and conclusions drawn on samples with data collected over a period of time as short as 3 min. The longest duration experiments were carried out over 30 min.

The objective of this work was to investigate the shrinkage stress kinetics for up to 12 h at  $T = 33$  °C for selected commercial Bulk Fill RBCs. The null hypotheses were: (1) the shrinkage stress is the same at 2 min, 1 h and 12 h for each RBC at a RBC temperature of 33 °C, (2) there is no correlation between the maximum shrinkage stress rate and time at which it occurs  $t_{Max}$ , (3) there is no correlation between  $t_{Max}$  and the filler volume fraction, (4) there is no correlation between the shrinkage stress at 12 h and the filler volume fraction, and (5) there is no correlation between the shrinkage stress at 12 h and effective gel time.

## 2. Materials and methods

### 2.1. Materials

Five commercial Bulk Fill RBCs were selected in this study. The RBC compositions as described by the manufacturers are given in Table 1. Two low-viscosity (3M ESPE Filtek Flowable Restorative A2 shade and DENTSPLY Surefil SDR flow Posterior



**Fig. 1 – Modified ADAF tensometer to measure accurately the shrinkage stress at tooth temperature and long time. A Michelson interferometer was set up to measure the cantilever beam deflection.**

A3 shade), two medium viscosity (3M ESPE Filtek Posterior A2 shade and Voco GmbH X-tra fil) and one high viscosity (Ivoclar Vivadent Inc., Tetric Evoceram Posterior Bulk Fill IVA shade) were used to study the shrinkage stress with time, viscosity, gel time, and filler volume fraction.

### 2.2. Shrinkage stress and gel time measurements

A schematic diagram of the modified tensometer (American Dental Association Foundation, Paffenbarger Research Center, Gaithersburg, MD, USA) [28] used to measure the shrinkage stress kinetics and gel time of RBCs at 33 °C is shown in Fig. 1. The cantilever beam deflection was measured simultaneously with the built-in tensometer LVDT (Linear Variable Differential Transformer) position sensor and with an optical technique. The optical technique consists of using an interferometer where the “moving” mirror is mounted on the cantilever beam and the HeNe laser, beam splitter, fixed reference mirror, and Silicon photodiodes were mounted rigidly on the tensometer base. As the beam deflects caused by the photocuring of the RBC, constructive and destructive interferences of the recombined laser beam occur at the photodiodes. For instance, the cantilever beam deflecting from 0 to  $\lambda/2$  at the “moving” mirror where  $\lambda$  is the HeNe emission wavelength (632.8 nm) results in a full cycle (fringe) of constructive to destructive to constructive laser light interference at the photodiodes. The interferometric technique is highly sensitive to beam deflection where a deflection of 1/8th of a cycle (fringe), corresponding to a beam deflection of 40 nm at the moving mirror, can be easily measured. In contrast, the detection limit of the LVDT position sensor provided with the apparatus is 100 nm. This measurement approach toward monitoring an oscillatory instead of a slowly varying signal is not sensitive to electronic drift in the detection system.

A simultaneous calibration done at room temperature using the LVDT position sensor and interferometric photodiode signal was performed by applying a calibrated force at the sample position and measuring the beam deflection at the position sensor and photodiodes. An effective cantilever spring constant of  $k_{eff} = 4.283 \text{ N}/\mu\text{m}$  was obtained using the position sensor. Using the overall beam length  $L = 29.3 \text{ cm}$  and

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