DENTAL MATERIALS XXX (2015) XXX–XXX

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/dema

Simple optical method for measuring free shrinkage

Daranee Tantbirojn[a], *Carmem S. Pfeifer*[b], *Arianna N. Amini*[c], *Antheunis Versluis*[d,*]

[a] *Department of Restorative Dentistry, College of Dentistry, University of Tennessee Health Science Center, Memphis, TN, USA*

[b] *Biomaterials and Biomechanics, School of Dentistry, Oregon Health & Science University, Portland, OR, USA*

[c] *Sophomore at Rhodes College, Memphis, TN, USA*

[d] *Department of Bioscience Research, College of Dentistry, University of Tennessee Health Science Center, 875 Union Avenue, Memphis, TN 38163, USA*

ARTICLE INFO

Article history:
Received 14 November 2014
Received in revised form
10 April 2015
Accepted 14 August 2015
Available online xxx

Keywords:
Polymerization shrinkage
Resin composite
Free shrinkage
Stereomicroscope
Dilatometer
Image analysis software
Finite element analysis

ABSTRACT

Objectives. A simple optical method for measuring polymerization shrinkage of dental composites is compared with an established dilatometer.

Methods. Five restorative composites were used to test the methods: Filtek Supreme Ultra (3M ESPE), Filtek LS (3M ESPE), Premise (Kerr), Gradia Direct (GC), and GC Kalore (GC). Uncured composites were attached to sandblasted silane-treated glass slides. The slides were placed sample side inside a mercury-filled dilatometer (ADAF). The mercury levels were recorded as the materials were light-cured through the glass-slides (40 s). Mercury levels, which correlated with volumetric shrinkage, were recorded for 60 min ($N = 6$). For the optical method, uncured composite was placed on a smooth silicone platform. A pre-polymerization image was captured under a stereomicroscope, and the specimen was light-cured (40 s). Post-polymerization images were captured at 2, 10, 60, and 90 min ($N = 10$). Composite outlines were traced to obtain projected surface areas (ImageJ) and volumetric shrinkage was calculated. Results were analyzed using two-way ANOVA ($\alpha = 0.05$) and Pearson Correlation tests. Shrinkage deformation for both methods was modeled using finite element analysis.

Results. Volumetric shrinkage at 60 min ranged between 1.24% and 2.24% for dilatometer and 1.35–2.68% for optical methods. Optical method shrinkage was consistently higher than the dilatometer ($P = .0001$), but the ranking of the composites was the same (Pearson Correlation Coefficient 0.9997). Finite element analysis showed that lower shrinkage values of the dilatometer method could be attributed to bonding of its samples.

Significance. The optical method using a general-purpose stereomicroscope and public-domain software is a simple and accurate alternative to measure free shrinkage.

* *Corresponding author.* Tel.: +1 901 448 6372.
E-mail addresses: dverslui@uthsc.edu (D. Tantbirojn), pfeiferc@ohsu.edu (C.S. Pfeifer), amian-17@rhodes.edu (A.N. Amini), antheun@uthsc.edu (A. Versluis).

http://dx.doi.org/10.1016/j.dental.2015.08.150
0109-5641/

1. Introduction

Volumetric shrinkage is a consequence of polymerization of resin-based materials. It happens when formation of a polymer network creates a denser material. Shrinkage causes dimensional changes that can cause residual stress when it is hindered. Polymerization shrinkage is a concern in dentistry ever since dental composites were first developed as a restorative material [1]. Reduction of polymerization shrinkage remains one of the critical design properties in the development of new dental composites.

Various methods have been used to measure polymerization shrinkage. Volumetric shrinkage can be derived from changes in density between uncured and cured composites, measured using the buoyancy principle explained by Archimedes [2,3]. Another approach is measuring the displacement of liquids, such as water or mercury, in so-called dilatometers [4–9]. Shrinkage can also be determined by measuring changes in dimensions. They can be measured in one dimension or 'linear' (for example, linometer [10,11] and strain gauges [12]), or spatial (for example, Accuvol [13] and micro-computed tomography [14]).

Not all these methods measure the same shrinkage. Obviously, there are differences in which dimensions are measured and the type of shrinkage. But those differences are well recognized (post-gel versus total shrinkage) and conversions between dimensional expressions (linear versus volumetric) are well established [15]. However, a more fundamental concern that is generally overlooked is that although most methods — except post-gel shrinkage — assume to measure total (free) shrinkage, they often require attachment of the samples to a substrate to keep them in place (for example, to prevent the sample floating away or falling of the stage) or for attaching targets (for example, to allow a displacement sensor to contact or detect the sample) [16–18]. The results of such shrinkage methods may therefore not determine actual free shrinkage. Despite being a simple and well-defined property, measurement of shrinkage for dental materials has not been trivial. The majority of methods that are currently used require dedicated and sometimes costly equipment and/or devices that often are specifically designed for shrinkage measurements.

The objective of this study was to evaluate a simpler approach to measure free shrinkage [19]. This method used pre- and post-polymerizing images captured by a general-purpose stereomicroscope and processed with public-domain image analysis software. To validate the optical method, shrinkage values of five restorative composites were compared with a well-accepted shrinkage measurement technique (dilatometer). Finite element analysis was added to further compare the outcomes of the two methods.

2. Materials and methods

2.1. Materials

Five restorative composites were used to test the shrinkage methods: universal composite (Filtek Supreme, 3M ESPE, St Paul, MN, USA), anterior composite (Gradia Direct, GC Corporation, Tokyo, Japan), and three low-shrink composites (Filtek LS, 3M ESPE; Premise, Kerr Corporation, Orange, CA, USA; GC Kalore, GC Corporation). Material information is listed in Table 1.

2.2. Dilatometer method

Shrinkage was measured with an ADAF Mercury Dilatometer (Gaithersburg, MD, USA). Uncured composite was placed on a sandblasted and silane-treated glass slide. Samples were dome-shaped, approximately 6 mm in diameter and 1.5–2 mm high. The glass slide was clamped to a column filled with mercury. A linear variable differential transformer (LVDT) probe was seated on top of the mercury column. The composite was photopolymerized from the bottom with a quartz–tungsten halogen light source (DENTSPLY Caulk, Milford, DE, USA) curing light (550 mW/cm^2) for 40 s. The LVDT probe recorded displacements of the mercury column for 60 min at 0.1119 Hz. A second irradiation was performed to account for material expansion due to heat generated by the light source and exotherm. The second reading was offset from the original reading. Volumetric shrinkage was calculated from linear shrinkage based on the mass/density of the specimens, LVDT displacement, and temperature. Specimen densities were determined using the Archimedean principle using a density attachment coupled with a scientific balance (0.1 mg precision). The densities were 2.0788, 1.5166, 2.0263, 1.9515, and 1.9857 g/ml for Supreme, Gradia, Filtek LS, Premise, and GC Kalore, respectively. Sample size was 6 per group, which for a standard deviation of 0.22 could detect differences of more than 0.18% volumetric shrinkage among groups with 95% confidence.

2.3. Optical method

Uncured composite was dispensed on a silicone platform made from a light body polyvinylsiloxane impression material (Express, 3M ESPE) (Table 1). The amount of composite was gauged using a metal ring, 6 mm in diameter and 1.5 mm thick. The composite was shaped into a rounded disk on the silicone platform (Fig. 1A). The platform was placed under a stereomicroscope with charge-coupled device camera (SZX16 & UC30, Olympus, Tokyo, Japan) to capture the pre-polymerization image (Fig. 1B). The microscope lighting was only turned on during capturing an image to avoid premature conversion. Magnification was 1.25×, which was determined by the size of the sample. Although shrinkage is the relative change in dimensions and thus independent of magnification, for best results the largest magnification that still showed the entire sample should be chosen. Immediately following the capturing of the pre-polymerization image, the composite sample was photopolymerized for 40 s using a quartz–tungsten–halogen curing light (VIP Junior, Bisco, Schaumburg, IL, USA) with an intensity of 570 mW/cm^2 measured with a radiometer (Model 100, Demetron Research Corp, Danbury, CT, USA). Post-polymerization images were captured at 2, 10, 60, and 90 min. Focus of the microscope was not changed during the experiment. All experiments were conducted at room temperature. Sample size was 10 per group,

Please cite this article in press as: Tantbirojn D, et al. Simple optical method for measuring free shrinkage. Dent Mater (2015), http://dx.doi.org/10.1016/j.dental.2015.08.150