available at www.sciencedirect.com

ScienceDirect

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

Comparison of the visco-elastic behavior of a pre-impregnated reinforced glass fiber composite with resin-based composite

Abdul S. Khan [a,*], *Monisha J. Phillips* [a], *K. Elizabeth Tanner* [a], *Ferranti S. Wong* [b]

[a] *Department of Materials, Queen Mary University of London, London, United Kingdom*
[b] *Centre for Oral Growth and Development, Barts & The London School of Medicine and Dentistry, London, United Kingdom*

ARTICLE INFO

Article history:
Received 20 May 2007
Received in revised form
11 March 2008
Accepted 14 March 2008

Keywords:
Fiber reinforced composite
Particulate composite
Visco-elastic
Dynamic mechanical analysis

ABSTRACT

Objectives. The visco-elastic behavior of a pre-impregnated reinforced glass fiber composite (everStick®) was compared with a resin-based particulate composite (Filtek™ P60) by using dynamic mechanical analysis (DMA) to determine their storage modulus (E') and damping ratio ($\tan\delta$).

Methods. These materials were subjected to three-point bend tests using a PerkinElmer DMA7. In temperature mode, the temperature was increased from 26 to 140 °C at 1 Hz. In frequency mode, the range was 1–10 Hz at a constant temperature of 37 °C.

Results. In both temperature and frequency modes, E' for everStick® was significantly higher and $\tan\delta$ was significantly lower than those for Filtek™ P60, indicating that the stiffness of the pre-impregnated glass fiber composite was higher and its damping property was lower than those for resin-based particulate composite.

Significance. The glass fiber restorative composite appears to absorb less energy in repeated stress and is less likely to retain external energy as residual stress.

1. Introduction

Resin-based particulate composites (RBCs) are well-established restorative materials [1–4]. Fiber reinforced resins have also been used for a variety of applications; these have included carbon fibers [5–8], ultra high modulus polyethylene fiber [9–12], and latterly glass fibers [13–18]. Particulate composites are heterogeneous and isotropic materials, while fiber reinforced composites (FRCs) are heterogeneous and often anisotropic if long fibers are packed in one direction. In the latter case, the modulus and strength enhancement will be in the direction of the fibers. Such systems are used for endodontic posts [17,19]. However, if woven fabric or chopped fibers are used, then the composite is more homogeneous. Such systems have been used for denture bases [11,20–22]. One form of FRC employs densely packed silanated glass fibers pre-impregnated in a polymer–monomer gel consisting of a light cure dimethacrylate monomer resin (Bis-GMA) and a linear polymer (PMMA). When this composite is polymerized, a semi-interpenetrating polymer network (semi-IPN) is formed [23,24].

In the present study, the visco-elastic properties of one FRC (everStick®, Batch Number: 2040413-EO-040, Stick Tech Oy, Finland) and one RBC (Filtek™ P60, Batch Number: 4720 A3, 3M ESPE, Germany) were measured by dynamic mechanical analysis (DMA) with respect to temperature and frequency.

* *Corresponding author at*: Department of Materials, Queen Mary University of London, Mile End Road, London E1 4NS, United Kingdom. Tel.: +44 787 162 3061; fax: +44 20 8981 8904.
E-mail address: a.s.khan@qmul.ac.uk (A.S. Khan).

0109-5641/$ – see front matter
doi:10.1016/j.dental.2008.03.017

Visco-elastic measurements are useful in that information can be obtained about the polymeric system on which the material is based, and on the propensity of the material to creep under load. Visco-elastic solids differ from elastic solids in that [25]:

(i) on application of a constant stress, after the instantaneous strain, the strain increases with time (creep);
(ii) on application of a constant strain, after the instantaneous stress, the stress decreases with time (stress relaxation);
(iii) if an alternating (sinusoidal) stress or strain is applied, stress and strain are out of phase. This phase angle is denoted by δ. In the corresponding theory, the Young's modulus (E) of classical elasticity is replaced by the so-called complex modulus (E^*), where E^* is given by

$$E^* = E' + iE'' \tag{1}$$

where E' is the storage modulus, and represents the elastic component of deformation, E'' is the loss modulus, and represents the viscous (inelastic) component, and $i = \sqrt{-1}$. These are related to δ:

$$\tan\delta = \frac{E''}{E'} \tag{2}$$

$\tan\delta$ is a measure of energy loss, sometimes referred to loosely as damping capacity. It is related to the fraction of energy retained, resilience (R):

$$R = \exp(-\pi \tan\delta) \tag{3}$$

At the glass transition temperature (T_g), E' decreases dramatically over a short temperature range, E'' decreases initially and then increases, and $\tan\delta$ go through maxima, consequent on the enhanced molecular mobility; some polymers show secondary transitions. Beyond T_g the polymer is in the rubber-like state, where deformation is a function of entropy only. Visco-elastic properties of polymeric dental materials have been occasionally studied in the literature [26–29]. DMA measures the ratio of the amplitudes of stress to the applied strain, and the phase angle δ, and computes E', E'', and $\tan\delta$. In the current study the samples were tested in the three-point bending mode.

2. Materials and methods

The everStick® is a light cured, radiopaque unidirectional continuous fiber composite containing E-glass fibers (55% SiO_2, 15% CaO, 15% Al_2O_3, 6% B_2O_3, 0.5% MgO, and 1.0% Fe + Na + K). The glass fibers are pre-impregnated with a silane coupling agent and a dimethacrylate resin matrix that is surrounded by a coating of PMMA and Bis-GMA. The Filtek™ P60 is a visible-light activated, radiopaque, restorative composite. The fillers are zirconia/silica (61% by volume without silane treatment). The filler particle size ranges from 0.01 to 3.5 μm with an average particle size of 0.6 μm. The resins are Bis-GMA, urethane dimethacrylate (UDMA) and bisphenol A polyethylene glycol diether dimethacrylate (Bis-EMA).

2.1. *Sample preparation*

Polytetrafluorethylene (PTFE) molds were made in order to manufacture test samples (24 mm × 2 mm × 1.5 mm in length, width and height, respectively). Prior to use the FRC specimens were stored at 4 °C and it was maintained to avoid them from direct light source. The samples were cut with sharp scalpel and were soaked in a dimethacrylate monomer (Scotchbond Multipurpose Adhesive, 3M ESPE, USA) in a Petri dish for 10 min in a low light environment. Prior to polymerization, excess resin was removed with soft tissue paper. The specimens were then placed in the mold and were polymerized using blue visible light (DeTrey, Dentsply, Germany, wavelength ≈ 470 nm) for 60 s. The distance of blue visible light was maintained constant for all samples. After removing the sample from the mold, the edges and the rough surfaces were polished by dry silica paper using 400 grade followed by 800 and 1000 grades to improve the surface finish. Twelve rectangular-shaped beam specimens were prepared and were stored in a low light environment before DMA testing. For the RBC sample, the samples were used as it was received from the manufacturer and were handled according to manufacturer's instruction. The material was injected directly into the PTFE mold (24 mm × 2 mm × 1.5 mm) without soaking in a monomer and 12 specimens were prepared. The specimens were polymerized by using blue visible light (DeTrey, Dentsply, Germany, wavelength ≈ 470 nm) for 60 s. The distance of blue visible light was constant for all samples. After removing the sample from the mold, the edges and the rough surfaces were polished by dry silica paper using 400 grade followed by 800 and 1000 grades to improve the surface finish.

2.2. *DMA*

A PerkinElmer DMA7 (PerkinElmer Corp., USA) in three-point bending mode was used to measure the dynamic mechanical properties of the two materials. For a specimen of known geometry if, L = distance between the two supports, b = width, and t = depth, the oscillating strain (ε_o) is given by

$$\varepsilon_o = \frac{3ty_0}{L^2} \tag{4}$$

where y_0 is the displacement amplitude.

The maximum oscillating stress (σ_o) occurs on the upper and lower surfaces and was given by

$$\sigma_o = \frac{3F_0L}{2bt^2} \tag{5}$$

where F_0 is the axial force amplitude. Therefore, by substituting for stress and strain, the complex modulus (E^*) was given by

$$E^* = \frac{F_0L^3}{2y_0bt^3} \tag{6}$$

The support separation in three-point bend test was 20 mm and the specimen length was 24 mm. The width and height were nominally 1.60 and 0.8 mm, respectively for everStick®. For Filtek™ P60, the nominal width and height were 1.72 and