DENTAL MATERIALS 25 (2009) 188–197

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The mechanical properties of nanofilled resin-based composites: The impact of dry and wet cyclic pre-loading on bi-axial flexure strength

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ARTICLE INFO

Article history:
Received 1 February 2008
Received in revised form
25 April 2008
Accepted 2 June 2008

Keywords:
Cyclic fatigue
Bi-axial flexure strength
Nanofillers
Resin-based composite

ABSTRACT

Objectives. To determine the influence of nano-sized filler particles and agglomerates of nanoparticles ('nanoclusters') in resin-based composite (RBC) materials on the bi-axial flexure strength (BFS) following cyclic pre-loading and storage in a 'dry' or 'wet' environment.

Method. Seven commercially available RBC restoratives, Heliomolar (Ivoclar Vivadent, Schaan, Liechtenstein), Z100 MP Restorative, Filtek^{TM} Z250, Filtek^{TM} Supreme (3M ESPE, St. Paul, MN, USA) in Body (FSB) and Translucent (FST) shades, Grandio and Grandio Flow (VOCO, Cuxhaven, Germany), containing differing filler particle types and morphologies were investigated. Specimens were pre-loaded at 20, 50 or 100 N for 2000 cycles and stored in a 'dry' or 'wet' environment prior to BFS testing.

Results. A general linear model analysis of variance highlighted a reduction in the BFS following pre-loading, however, individual RBC materials responded differently. The RBCs containing agglomerated nano-sized particles or 'nanoclusters' (Filtek^{TM} Supreme) demonstrated distinctive and unique patterns of response to pre-loading. Cyclic pre-loading at 20 and 50 N significantly increased the Weibull modulus of both FSB (8.53 ± 1.91 and 10.23 ± 2.29) and FST (16.89 ± 3.78 and 10.91 ± 2.45) compared with FSB and FST control (no pre-cyclic load) specimens (5.98 ± 1.34 and 7.99 ± 1.78, respectively). BFS of FSB and FST was maintained or significantly increased compared with the other materials following 20 and 50 N cyclic pre-load ($P < 0.05$).

Significance. The 'nanoclusters' provided a distinct reinforcing mechanism compared with the microhybrid, microfill or nanohybrid RBC systems resulting in significant improvements to the strength and reliability, irrespective of the environmental storage and testing conditions. Silane infiltration within interstices of the nanoclusters may modify the response to pre-loading induced stress, thereby enhancing damage tolerance and providing the potential for improved clinical performance.

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doi:10.1016/j.dental.2008.06.003

1. Introduction

The demand by patients for tooth-coloured restorations, concerns regarding environmental impact and the adverse clinical reactions to amalgam filling materials have accelerated research into the development of alternative restoratives. However, despite the development of resin-based composite (RBC) materials the clinical longevity of dental amalgam remains superior [1]. Posterior amalgam restoratives exhibit a median survival time exceeding 11 years, whilst tooth-coloured materials, including RBCs possess median survival rates below 7 years [1]. A frequent cause of premature restoration failure is the occurrence of fatigue as a result of cyclic masticatory forces initiating crack propagation and manifested as fracture of RBCs following several years clinical service [2]. Following anterior placement restorations will typically be subjected to masticatory forces ranging from 100 to 200 N [3], whilst posterior restorations may be loaded to up to 800 N [4]. Although forces generated whilst chewing foodstuffs are considerably lower (∼10–20 N) [5], it is frequently the accumulation of localised microscopic loading induced damage that influences the survival rate of the restoration [6].

Hybrid RBCs consist of dispersions of individual silanated inorganic particles within an organic resin matrix. The development of RBCs as an alternative to dental amalgam has resulted in optimisation of the particle size distributions and filler loading, resulting in an improvement in the mechanical properties [7–9]. In order to achieve superior aesthetics, sub-micron fillers were introduced to the development of RBC materials. However, filler loading of the early 'homogeneous microfill' RBC types was reduced due to a high surface area to volume ratio, thereby limiting mechanical properties. The introduction of 'heterogeneous microfills' increased the filler loading (∼50 vol%) as prepolymers containing a high volume fraction of silanated nanofillers (∼50 nm) were incorporated into a resin matrix containing discrete sub-micron particles. Although the approach improved the flexural strength of 'heterogeneous' RBCs (80–160 MPa) compared with 'homogeneous' microfills (60–80 MPa) [10,11], the mechanical properties remained inferior to hybrid RBC systems, which are loaded to approximately 55–65 vol% and possess flexure strengths in the region of 120–145 MPa [11].

A recent response to the challenge of combining strength with aesthetic appearance and working characteristics uses a combination of individually dispersed nano-sized filler particles and agglomerations of these particles, described as 'nanoclusters'. Filtek^{TM} Supreme (3M ESPE, St. Paul, MN, USA) contains silica and zirconia nanoparticles, which are partially calcined to produce micron-sized porous clusters that are infiltrated with silane prior to incorporation into a resin matrix (Table 1; Fig. 1). The manufacturers claim that the system matches hybrid RBCs for strength and microfills for surface finish [12]. A previous study suggested nano-sized particles and "nanoclusters" provided distinct mechanical properties as water uptake and subsequent strength loss was modified by the size, morphology and resulting surface area of the fillers [13]. In the first part of this work [14] discrete filler particles separated from the resin matrix were loaded using a micromanipulation technique. The authors demonstrated that the 'nanoclusters' of Filtek^{TM} Supreme exhibited multiple fractures and a higher force at fracture compared with the spheroidal and irregular filler technologies [14]. This was attributed to the ability of the "nanocluster" to deform and collapse into pre-existing cluster porosities and through progressive fragmentation of the main cluster structure, which subsequently acted to absorb and dissipate propagating cracks [14]. The authors suggested that incorporation of the 'nanocluster' particles into the resin matrix as a complete system would have the potential to produce unique mechanical properties since deformation of the particle may enhance the resistance to crack propagation of the RBC

Table 1 – Summary of the constituents and quantities/ratios of components contained in the seven RBCs investigated

	Classification	Resin	Filler	Total filler content	
Heliomolar (HM)	Microfill	BisGMA, UDMA, DEMA	Pre-polymer (containing silica) Ytterbium trifluoride: 40–200 nm (66.7 wt%)	66.7 wt%	46.0 vol%
Filtek Z250 (FZ)	Microhybrid	BisGMA, UDMA, BisEMA$_6$, TEGDMA	Zirconia/silica: 0.01–3.5 µm (84.5 wt%)	84.5 wt%	60.0 vol%
Filtek Z100 (Z100)	Microfill	BisGMA, TEGDMA	Zirconia/silica: 0.01–3.5 µm (84.5 wt%)	84.5 wt%	66.0 vol%
Filtek Supreme Body (FSB)	Nanofill	BisGMA, UDMA, BisEMA$_6$, TEGDMA	Silica: 5–20 nm nanoparticle (8 wt%); zirconia/silica: 0.6–1.4 µm nanocluster (71 wt%)	79.0 wt%	59.5 vol%
Filtek Supreme Translucent (FST)	Nanofill	BisGMA, UDMA, BisEMA$_6$, TEGDMA	Silica: 75 nm nanoparticle (40 wt%); silica: 0.6–1.4 µm nanocluster (30 wt%)	70.0 wt%	57.5 vol%
Grandio (GR)	Nanohybrid	BisGMA, TEGDMA	Silica: 20–60 nm; barium-alumia borosilicate: 0.1–2.5 µm (87 wt%)	87.0 wt%	71.4 vol%
Grandio Flow (GF)	Nanohybrid	BisGMA, TEGDMA, HEDMA	Silica: 20–60 nm; barium-alumia borosilicate: 0.1–2.5 µm (80.2 wt%)	80.2 wt%	65.6 vol%

BisGMA: bisphenol A diglycidy ether dimethacrylate; TEGDMA: triethyleneglycol dimethacrylate; BisEMA$_6$: bisphenol A polyethylene glycol diether dimethacrylate; UDMA: urethane dimethacrylate; HEDMA: hydroethyl dimethacrylate; DEMA: decandiol dimethacrylate.

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