JOURNAL OF THE MECHANICAL BEHAVIOR OF BIOMEDICAL MATERIALS 29 (2014) 427–437

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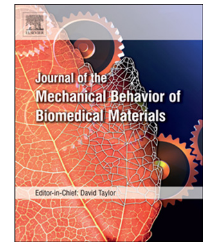


Research Paper

# Evaluation of the interfacial work of fracture of glass-ionomer cements bonded to dentin

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ARTICLE INFO

*Article history:*
Received 16 July 2013
Received in revised form
8 September 2013
Accepted 15 September 2013
Available online 10 October 2013

*Keywords:*
Work of fracture
Interfacial work of fracture
Resin-modified glass-ionomer
Conventional glass-ionomer

ABSTRACT

*Objective:* The aim of this study was to investigate the interfacial work of fracture of conventional (C-) and resin-modified (RM-) glass-ionomer cements (GICs) bonded to dentin.
*Methods:* One hundred and sixty five aries-free human molars were embedded in epoxy resin, sectioned and polished with 300- and 600- grit silicon carbide paper to remove enamel on the occlusal surface. Equilateral triangular-shaped plastic molds ($4\times4\times4\times5\ mm^4$) were clamped to the prepared dentin surfaces by a stainless steel test apparatus. Teflon tape was placed under one internal vertex of the mold to create a 0.1-mm notch at the material-dentin interface. Interfacial work of fracture ($\gamma_{wof}int$) in tensile fracture mode-I (opening) was determined for six C-GIC, three RM-GIC, and two GIC luting cements at a cross-head speed of 0.1 mm/min and a crosshead distance ($L$) from the interface of 4.3 mm. The debonded surfaces were evaluated for the predominant failure mode. SEM analysis of examples showing interfacial and notch areas was performed.
*Results:* ANOVA and Tukey's post hoc test demonstrated the highest mean $\gamma_{wof}int$ value ($90.16\pm16.6\ J/m^2$) of one RM-GIC was significantly different ($p<0.05$) from the other materials. 'High viscosity' GICs achieved lower results with the lowest recorded at $20.4\pm10.1\ J/m^2$. There was a significant difference observed ($p<0.05$) between the mean $\gamma_{wof}int$ of luting C-GIC and luting RM-GIC. Although differences were observed between different material mean $\gamma_{wof}int$, when comparing groups no significant differences ($p=0.181$) were observed. For all groups, mixed GIC-interface failure (41%) was the most commonly observed, followed by cohesive failure in GIC (25%) and adhesive failure (20%). SEM analysis revealed that specimens generally fractured from the notch initiation point into the GIC or along the dentin–GIC interface.
*Conclusion:* Within the limits of this study, significant differences ($p<0.05$) were observed in the $\gamma_{wof}int$ between different glass-ionomer materials. The null hypothesis that there is no difference in the $\gamma_{wof}int$ among different glass-ionomer materials bonded to human dentin was rejected.

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1751-6161/$ - see front matter 
http://dx.doi.org/10.1016/j.jmbbm.2013.09.020

*Relevance:* In the current study, the interfacial work of fracture ($\gamma_{wof}int$) of glass-ionomer adhesive interfaces has been reported using a simple method that can be used to study the fracture mechanics of an adhesive interface without the need for complicated specimen preparation.



## 1. Introduction

Variables such as test rig, biological substrate, dentin and enamel type and position in the tooth, storage of teeth, affect bond strength tests (Heintze, 2013). However the bond tests used are simple and easy to use, so they are still commonly reported in an effort to demonstrate the efficacy and ranking of adhesive dental materials. Comparing bond strength test results from different studies is not recommended as test conditions are invariably different among the various tests (Kelly et al., 2012; Scherrer et al., 2010; VanNoort, 1989). Several authors have questioned the validity of these tests and criticized the lack of standard protocols and relevance to clinical performance (Della Bona and Watts, 2013; Heintze, 2013; Stephen, 2012). Researchers have also suggested that a fracture mechanics approach is more appropriate than conventional shear or tensile bond strength tests (Salz and Bock, 2010).

Using a fracture mechanics approach, a crack is introduced into the bond interface and the system's strength to resist crack propagation across the adhesive interface has also been investigated (Hashimoto, 2010; Salz and Bock, 2010). Critical stress intensity factor (KC), describes the ability to resits crack propagation. Linear elastic fracture toughness ($K_{IC}$) studies the stress region ahead of crack propagation in tensile (mode – I) failure (Soderholm, 2010). Various methods have been employed to investigate interfacial fracture toughness of adhesives to teeth and biomaterials to determine the work of fracture which has been abbreviated as $W_f$ and$W_i$, plane strain interfacial fracture toughness ($K_{IC}int$), the adhesive (elastic-plastic) fracture energy ($J_{IC}$) and the critical plane strain energy release rate ($G_{IC}int$) (Armstrong et al., 1998, 2001; Barker, 1977; Cheng et al., 1999; De Munck et al., 2013; Della Bona et al., 2006; Howard and Söderholm, 2010; Jancar, 2011; Lin and Douglas, 1994; Rasmussen, 1984; Rasmussen and Patchin, 1984; Tam and Pilliar, 1993, 1994; Tam and Yim, 1997; Toparli and Aksoy, 1998; Walshaw et al., 2003). Studies have also been performed on bone cements, which show adhesive failure occurring due to stress cracking at some point in their lifecycle (Lucksanasombool et al., 2003; Tong, 2006; Tong et al., 2007; Wang and Pilliar, 1989; Wang and Agrawal, 1997, 2000; Wang et al., 1994). Interfacial fracture toughness has also been measured for glass-ionomer materials (Akinmade and Hill, 1992; Mitsuhashi et al., 2003; Setien et al., 2005; Tam et al., 1995). Furthermore, a series of reviews on bond tests have recommended that a fracture mechanics approach be revisited as the preferred test method for adhesive strength evaluation (De Munck et al., 2005; Kinloch, 1979; Salz and Bock, 2010; Scherrer et al., 2010; Soderholm, 2010).

A contributing factor as to why adhesive interfacial fracture toughness tests have not been commonly reported is because current tests are complicated and often require specialized apparatus to prepare the dentin and dental material into the desired configuration. Attempts have been made to develop less complex systems, including adaptations of a common shear bond strength test using triangular shaped adhesive areas, (Cheng et al., 1999; Tantbirojn et al., 2000) and a notchless triangular prism specimen developed by Ruse et al. (Ruse et al., 1996). There is currently no available standardized test method for determination of interfacial fracture toughness properties of adhesive dental materials.

Tattersall and Tappin introduced a simple method to determine the work of fracture of materials using a specimen with a square cross section and triangular fracture surface (Tattersall and Tappin, 1966). Further work by Rasmussen et al. demonstrated a test method to study the fracture properties of enamel and dentin, and determined the work of fracture ($W_f$ or $\gamma_{wof}$), calculated by dividing the total energy ($J$) required to initiate fracture by twice the surface area ($m^2$) of a triangular-shaped fractured surface (Rasmussen, 1984; Rasmussen and Patchin, 1984; Rasmussen et al., 1976). The test method was also adapted to investigate interfacial work of fracture ($W_i$ or $\gamma_{wof}int$) for porcelain-gold and enamel-composite adhesion (Rasmussen, 1978). Subsequent studies have also investigated fracture mechanics of different materials and interfaces using a $\gamma_{wof}$ approach (Sakai and Bradt, 1993).

The aim of this study was to compare the interfacial work of fracture ($\gamma_{wof}int$) and failure modes of several glass-ionomer cements bonded to dentin, using a new simplified test method. The null hypothesis is that there is no difference in the interfacial work of fracture ($\gamma_{wof}int$) among different glass-ionomer cement materials bonded to human dentin.

## 2. Materials and methods

### 2.1. Teeth preparation

Human ethics approval (#1033315.1) for the use of human teeth was obtained from the University of Melbourne. One-hundred and sixty-five caries-free human molars were selected from a tooth bank. No information on the age of teeth was available. Teeth were stored in a refrigerated 0.5% chloramine T solution and used within six months of collection date. This method of storage followed guidelines described in ISO TS 11405 Dental materials – testing of adhesion to tooth structure. The teeth were cleaned with a slow-speed prophylaxis polisher (Zen; Philips, CA) and wet pumice, rinsed and stored in de-ionised water for approximately 24 h prior to embedding. Three stainless steel mold