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Research paper

Fracture resistance curves and toughening mechanisms in polymer based dental composites

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ABSTRACT

The fracture resistance (R-curve behaviour) of two commercial dental composites (Filtek Z350® and Concept Advanced®) were studied using Double Cantilever Beam sandwich specimens loaded with pure bending moments to obtain stable crack growth. The experiments were conducted in an environmental scanning electron microscope to (a) accurately measure the applied energy-release rate for crack initiation, (b) measure the early (rising) part of the R-curve, and (c) provide direct microscopic evidence of the toughening mechanisms ahead of and/or in the wake of the crack tip. The two tested composites displayed distinctly different R-curve behaviours. The difference was related to different toughening mechanisms as the two composites had markedly different microstructures. Contrary to common experience, the composite with the finer microstructure (smaller particles), the Concept Advanced®, showed significantly higher fracture resistance than the composite with the coarser microstructure. The fracture properties were related to the flexural strength of the dental composites. The method, thus, can provide useful insight into how the microstructure enhances toughness, which is necessary for the future development of such materials.

1. Introduction

Resin based composites have been widely used in the dental restorative field as an alternative to traditional amalgam and their popularity continues to grow (Hickel, 2009; Christensen, 2007). The main reasons are (a) aesthetic aspects (Roulet, 1988; Lohbauer et al., 2006) and (b) the presence of the potentially toxic mercury in amalgam and the environmental pollution by mercury waste (Hickel et al., 2000; Hickel, 2009). However, the use of amalgam is not declining rapidly since the strength and durability of dental restorations carried out using dental resin composites are not as high as those of amalgam fillings. The properties of concern are polymerisation shrinkage, fracture, fatigue and wear (Christensen, 2007).

The most prominent reason for failure is secondary caries, which from a materials point of view is caused by polymerisation shrinkage during curing of the composite

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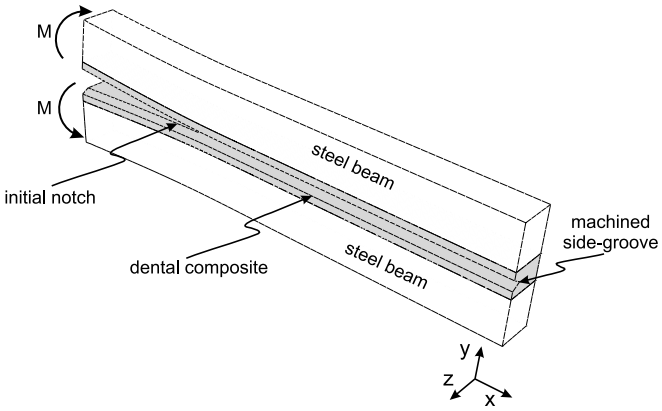

Fig. 1 – Double Cantilever Beam (DCB) specimen loaded with pure bending moments.

(Sakaguchi et al., 1992; Palin et al., 2005). The second most important reason for failure is fracture (Manhart et al., 2004) which can lead to chipping and/or bulk fracture (van Dijken, 2000) of the restoration. This problem is related to the low fracture resistance of these materials. Thus, there is a need for improving the fracture toughness in dental resin composites in order to diminish this failure mode of dental fillings.

These shortcomings have resulted in a continuous effort to develop resin composites with improved fracture resistance properties among other properties. Modifications of the resin and/or filler surface treatment have been reported (Musanje and Ferracane, 2004; Mante et al., 2010). Most studies, however, have been focused on the filler technology (Davis and Waters, 1987; Zhao et al., 1997) where the reinforcement is in the form of particles (Lohbauer et al., 2006; Watanabe et al., 2008; Bonilla et al., 2003) or whiskers/fibres (Krause et al., 1989; Xu et al., 1999, 2003). Currently particle reinforcement is most commonly used in practice. In the last few years the use of particles in the nano-size range is increasing. Due to the reduction of the particle size, a higher filler volume fraction can be achieved which results in improved mechanical properties (Beun et al., 2007; Mayworm et al., 2008).

Linear Elastic Fracture Mechanics (LEFM) has been extensively used in studying the fracture properties of dental materials (Kim et al., 1994; Indrani et al., 1995; Zhao et al., 1997; Manhart et al., 2000; Rodrigues et al., 2008). The resistance to fracture is described by a single material property, the mode I fracture toughness K_{IC}. However, crack growth in dental materials can be complicated among other things by the occurrence of crack bridging during crack growth. Such a toughening mechanism gives rise to fracture resistance with crack extension (R-curve behaviour) (Broek, 1986). Indeed, Shah et al. (2009a,b) have shown recently that resin based dental restorative composites exhibit R-curve behaviour and therefore the fracture of these materials should not in general be characterised in terms of LEFM. However, Shah et al. (2009a,b) were not able to measure the early part of the R-curve due to experimental limitations and the toughening mechanisms were studied by post failure examinations of fractured specimens.

Thus, the objective of the present work is to develop a test method which can be used to measure the entire R-curve accurately and *in-situ* observe the toughening mechanisms in great detail. Such a method is necessary to enhance toughness by controlling microstructure. To illustrate the usefulness of the method, two commercially available particle reinforced based dental composites, Filtek Z350® (3M ESPE) and Concept Advanced® (Vigodent), with approximately the same filler load, but with distinctly different microstructure and thus fracture mechanical properties, were investigated. In addition, three-point bending tests were performed for the two materials in order to assess their flexural strength properties and relate them to the fracture mechanics properties.

2. Analysis

In the present study, the fracture mechanical properties are determined by the use of a Double Cantilever Beam sandwich specimen loaded with pure bending moments, see Fig. 1. A flat dental polymer composite specimen, which has a notch in the one end, is glued to two steel beams that are mounted on a special fixture (described in the next section) that applies pure bending moments (Sørensen et al., 1998). As indicated in Fig. 1, the specimen has a side groove to guide the crack, so that the width of the cracking plane, b, is smaller than the overall specimen width, B.

The applied energy-release rate, $\mathcal{G}$, is calculated through the path independent J-integral (Rice, 1968). The application of the J-integral is valid also for a large-scale bridging (Bao and Suo, 1992). In the case of R-curve behaviour due to plasticity, the I-integral (Budiansky et al., 1983) can be applied under steady-state crack growth; as long as the specimen is not unloaded, the I-integral result for the specimen used here is identical to the J-integral result.

The J-integral is calculated along the external boundaries of the specimen Γ_i with $i = 1, 5$ (Fig. 2) and the only non-zero contributions come from the beam ends. The specimen is analysed as a plane problem with uniform width equal to B. The beams are subjected to pure bending moments, and if the beams are a few times longer than their height, the strain varies linearly across the height. Under these conditions, the