



The shear adhesion strength between the FRC substructure and denture base resin: Effects of FRC architecture, adhesive composition and hydrolytic degradation

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ABSTRACT

The adhesion strength between fiber reinforced composite (FRC) members and denture-base resin is pivotal for enhancing removable denture long lasting performance and patient comfort. FRC reinforcing rods based on glass fibers impregnated with light curing resin were used to model the FRC substructure. The influence of fiber architecture and adhesive layer composition on the shear adhesion strength, τ_a , between the FRC and denture-base resin was investigated both dry and in moist environment to assess the stability of the adhesive bond in the oral cavity. The obtained results suggest that for a given fiber architecture, adhesive composition and test conditions, the wetting of the FRC surface was the primary variable affecting the τ_a . In the case of good wetting and formation of adhesive bond between the substrates, interlaminar shear strength of the unidirectional FRC substrate was the limiting factor. In multidirectional FRC substrate, the shear strength of the outer resin rich layer was limiting factor for the maximum adhesion strength.

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1. Introduction

There is an increasing number of applications in modern dentistry using fiber reinforced composites (FRCs) in reinforcing crowns, constructing frameworks for fixed partial dentures, periodontal splints, post-orthodontic retainers, and manufacturing and repairing removable dentures as well as various acrylic orthodontic devices. Desired biomechanical performance, esthetics and patient comfort are achieved by combining easily formable load bearing FRC substructures with composite veneers, denture-base resins, flowable composites and resin based adhesives. For achieving long lasting dental device, a good adhesion between all the components is a very important prerequisite.

In addition to economical constraints, there are many clinical indications requiring replacement of missing teeth or restoring of normal function and appearance of the patient's oral cavity using removable denture. Acrylic denture base resins based on lightly cross-linked poly-methylmethacrylate (PMMA) are routinely used to manufacture removable dentures employing the “dough” technique. Easy handling, low cost, stability in the oral environment and the aesthetics are the main reasons why they became widely

used. However, their limitations include low fracture toughness, poor fatigue behavior and low stiffness. Metal wires embedded in the acrylic resins are commonly used to partly obviate these deficiencies as well as to reinforce fractured dentures and other devices during their repair. Poor adhesion to the acrylic resins and poor esthetics of metal wires result in rigid, low comfort dentures prone to brittle failure. In order to address these shortcomings, FRCs have been introduced to dentistry more than a two decades ago [1]. In dentistry, various preformed FRC components replacing metal load bearing substructures or reinforcing elements are gaining popularity in a growing number of prosthetic and orthodontic applications [2–12]. Rigidity and strength of FRC are influenced by fiber type and volume fraction, fiber orientation and adhesion to the polymer matrix as well as by the quality of impregnation with the resin matrix [13].

Upon curing to the final shape, the FRC components are either covered with esthetic particulate filled composites (crowns, bridges, splints, retainers, posts) to improve patient's comfort and maintain good oral hygiene or embedded in the acrylic resin when used as reinforcing elements (removable dentures, repaired dentures, orthodontic devices) [14–17]. The quality of adhesion between the FRC substructure and the resin based esthetic materials is one of the main concerns in improving the service life of current removable prostheses and other acrylic dental devices. Since the chemistry of resins used to manufacture both dental FRCs and denture base resins is substantially similar, desired formation of

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a chemical adhesive bond can be achieved [18]. However, as shown previously [19], the factors affecting the adhesion strength of a given joint also include substrate and adhesive toughness, substrate geometry, adhesive layer thickness and mode of loading the adhesive joint. Most of the published data were obtained using components cured the same way (light, heat, dual). Even though the mechanism of cure remains the same, the cure kinetics and degree of cure differ significantly for light and heat cured dental resins [20,21]. At the same time, despite its importance, little is known about the adhesion between light curing FRC and heat curing acrylic resins processed using the dough technology [22].

The addition of nanoparticles with large specific surface area ($200\text{--}1000\text{ m}^2/\text{g}$) compared to micrometer size fillers ($0.2\text{--}5\text{ m}^2/\text{g}$) into a polymer network modifies both cure kinetics and mechanical behavior below and above its glass transition temperature, T_g . It was shown that below T_g , chain packing, density fluctuations, and segmental relaxations vary proportionally to the chain–particle interface area and interfacial interaction strength similar to that observed in polymers with antiplasticizers. Nanoparticles dispersed in monomer mixtures can also self-assemble during cure into extended structures similar to colloids. Due to their extremely large specific surface area providing large interface area between the solid surface and reacting monomer mixture even at low nanoparticle content, presence of nanoparticles can affect the partitioning of reactive species in the mixture resulting in modified network structure and cure kinetics [23].

The aim of this paper was to investigate the shear adhesion strength, τ_a , between the light cured FRC substrate and lightly cross-linked, heat cured acrylic denture base resin. Nanocomposite adhesive layer with varying composition was used and the adhesion strength was investigated using modified pull-out test with varying joint geometry. To assess the longevity of the bond in the moist environment of oral cavity, joints were exposed to water for the period of up to 4 months. In addition, investigation of the loci of failure and the crack path was attempted using scanning electron microscopy (SEM) of the failed test specimens. Fracture mechanics protocol was used to analyze the experimental data.

2. Materials and methods

Commercial Bis-GMA/TEGMA monomer mixture *Evicrol* (Kerr-Dental, Czech Republic) was used as the resin matrix to prepare FRC rods, particulate filled composite (PFC) rings and adhesive interlayer. The photoinitiation complex consisted of 0.2 wt.% camphoroquinone (*Sigma-Aldrich*, USA) and 0.2 wt.% *N,N*-dimethylaminoethylmethacrylate (*Sigma-Aldrich*, USA).

FRC cylindrical rods were prepared using either continuous S2-glass roving (AGY, Belgium) or E-glass fiber braids (ADM, a.s., Czech Republic). Fiber volume fraction, v_f , in all the FRCs investigated was kept constant at $v_f = (0.38 \pm 0.02)$. Glass fiber bundles of approximately 100 mm in length were introduced into a round clear glass tube (length approx. 40 mm, inner diameter approx. 1.5 mm). Fiber bundles protruding from the glass tube were impregnated with the monomer mixture. Then, the impregnated portion of the fiber bundle was pulled into the tube to resume its symmetrical round cross-section rod shape. The pre-impregnated fiber bundle was light cured through the glass tube using the *Targis Power* (Ivoclar Vivadent AG, Liechtenstein) light cure chamber for 10 min at room temperature. The sample was then removed from the tube and its surface was cleaned with acetone [24]. Round cross section braids were impregnated using the same procedure as described above.

Nanocomposite adhesive was prepared by adding desired amount of fumed silica with average particle size of 5 nm and specific surface area of $200\text{ m}^2/\text{g}$ (*Cab-O-Sil M-5*, Cabot, USA) into resin and mixture was vigorously stirred for 10 min at room

temperature. Three adhesive compositions with silica content of 0, 1.7 and 7.9 vol.% were prepared and stored in a dark container to prevent their premature cure.

Heat curing denture-base acrylic resin *Superacryl Plus* (Kerr-Dental, Czech Republic) was processed according to manufacturer's instructions. Two weight parts of the powder containing PMMA, dibutylphthalate, zinc oxide and pigments were mixed with one weight part of the liquid containing methylmethacrylate and glycoldimethacrylate to form plastic dough. The dough was processed according to manufacturer's direction and desired amount was placed into the silicon rubber mold to form the cylindrical test specimen (Fig. 1).

Reference test specimens (Fig. 1a) were prepared by placing the cured FRC rod into a rubber mold (Lukopren 1522, Lucebni zavody Kolin, Czech Republic) with a symmetrical cylindrical cavity. Rod was aligned with the axis of symmetry of the cylindrical cavity. Then, the cavity was filled with either the denture-base dough (sample group I) or adhesive (sample group II) to form a coaxial

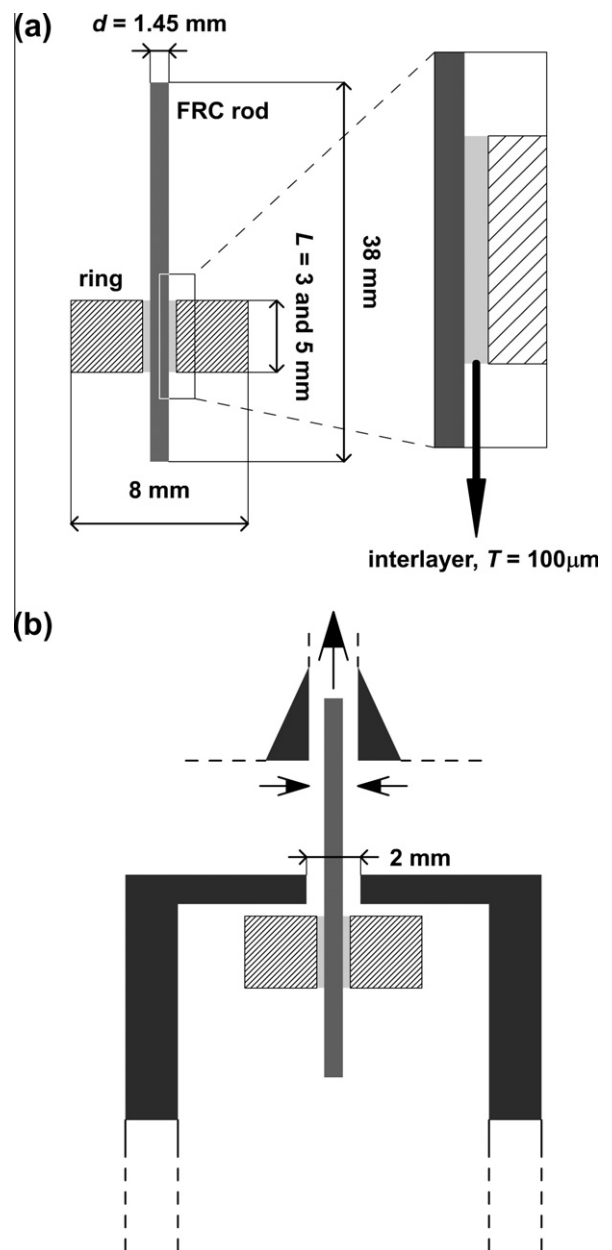


Fig. 1. (a) Dimensions of test specimen, and (b) diagram showing test geometry.

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