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In vitro shear bond strength test and failure mechanism of zinc phosphate dental cement

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ABSTRACT

The cohesive shear bond strength (SBS) of hardened zinc phosphate cement (CeCe) was comparatively measured with those of the adhesive SBS of the human dentin–cement (DCe), artificial acrylic crown–cement (CrCe), dentin–cement–crown (DCeCr). Results from experiments found that the SBS of CeCe specimen is much higher than those of the adhesive SBS. The average maximum SBS of CeCe, DCe, DCeCr and CrCe specimens of approximately 6.91, 1.02, 0.67 and 0.25 MPa, are respectively obtained. Fractographs taken by scanning electron microscope and close-up camera images of the fracture surfaces were analyzed for their failure mechanisms. The crack initiation and propagation of DCe and CrCe bonding types occur along the bonding interface. In the DCeCr bonding type, the crack initiates at crown–cement interface, propagates downward and changes in to the dentin-cement interface until failure. The average fracture area ratio of the CrCe interface to DCe interface of about 85:15 are observed for the DCeCr de-bonded specimens. Results from force and stress analysis using the two point bonding model indicate that the shear stress is more evident for the bond fracture.

1. Introduction

In dental restoration for both intracoronal preparations such as gold or ceramics inlays and extracoronal preparations such as crown, onlays, bridges, adhesives or cements for bonding with a long term stability, high bond strength and non-toxicity are preferable. The classical zinc phosphate cement has been used as the permanent luting agent under indirect restorations and commonly used for comparative study to the new developed cements. Strong attachment in dental restorations can be accomplished by mechanical bonding or retention of hardened adhesive projections embedded in the crevices or pores at the adherend surface most of which has to be treated prior to making bond. The acid etch technique on the adherend surface of some dental ceramics containing glassy matrix can enhance the bond strength [1]. Apart from the attempt to improve the adhesive bond strength by surface treatment, the study for development of the cement bond efficiency with various methods has been reported. Addition of titania nanotubes to the resin based cement can significantly improve the fracture toughness, flexural strength and modulus [2]. However, the report from the bond strength test of dental adhesives added with nano silver particle found that no bond strength enhancement has been observed [3]. The long term adhesion of several dual polymerizing cementing agents to human dentin was studied by shear testing after 150 days of storage in water for one subgroup and after 150 days of storage in water plus 37,500 thermal cycles for the other subgroup [4]. Cementing agents or adhesive systems and the polymerization method are found to influence the long-term bond to hard dental tissues.

In the materials point of view, the bond strength in dental restorations is the one important factor and various methods of the bond strength tests have been used for the study such as tensile strength, fatigue, flexural and shear bond strength. Due the simplicity of testing procedure the shear bond strength (SBS) test is the most popular test for measurements and results of the measured SBS with various conditions were reported. The SBS test was employed for the studies of the bond strength of dental adhesives added with nano-silver particle [3] and also in the study of the long term adhesion of dual polymerizing cements to human dentine [4]. Effects of surface treatments with plasma spraying on zirconia ceramic surfaces were reported and found that the SBS of composite luting cement to zirconia ceramic surface increased significantly [5]. The SBS of resin cements to commercially pure titanium with various conditions of surface treatment were reported and found

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to depend on the type of cement [6]. The SBS of the resin composite luting cement tested on dental ceramics with various surface conditioning methods was greatly reduced after themocycling [1]. Fatigue testing in conjunction with SBS testing of enamel bonds with self etch and total-etch adhesive systems were studied and a better means for assessing the performance of adhesive systems are achieved [7]. The study on shear and tensile bond strengths of dentine and ceramics using dual polymerizing cements indicated that cementing agents or adhesive systems may influence the bond strength and all failures are found within the resin cement–dentine interfaces [8]. Results of SBS of resin luting cement to glass-infiltrated porous aluminum oxide cores which were surface treated with alumina and diamond blasting were reported [9]. The tensile shear force was applied via a wire loop which placed under the wings of the bracket in order to study the bond strength of different cementing agents in orthodontics [10]. Results from the study on the cement layer dependence SBS of zinc polycarboxylate dental cement found that in the specimen with a thin adhesive layer thickness lower than 100 μm, the joint failed at the adherend interface but, in the thick adhesive layer the joint failed cohesively with a contribution from tensile stresses [11].

In the shear test under actual experimental conditions, it is quite difficult to produce pure shear failure [12,13]. Effects of knife indenter on stress concentration and shear bond strength of dental adhesive were reported and found that the highest stress concentration was occurred at the bonded interface [14]. The degree of stress concentration depended on the type of loading with the largest effect resulting from single point loading was reported [15]. To avoid the high stress concentration due to the single point contact from the knife edge indenter, the Ultradent Product Inc., South Jordan, Utah has developed the restricted Ultradent device which is a semicircular edge indenter. As a result, the highly uniform load distribution spread over the surface of the cylindrical shape specimen under the shear test. The results from SBS comparative tests using the knife edge indenter and Ultradent device of the dentin-light polymerized adhesive interface found that the SBS tested by the ultradent device was higher than that of the knife device [16]. Moreover, the distance between the point of load application and the bonded interface in shear tests also affects stress distribution. If the applied load moved farther away from the interface, the more likely that tensile failure rather than shear failure will occur because the potential for bending stress would increase [12]. A bending force can produce all three types of stresses in a structure of materials under test which are tensile stress, compressive stress and shear stress. However, most case of fracture occurs because of the tensile stress component [11–14,17]. Results from the finite element analysis (FEA) for evaluation of the stress distribution in shear and micro-shear test set ups of dental adhesives indicated that the loading distance is the important parameter [13] and the vertical shear stress is found to vary with an increase of the distance below the loading site [15]. Furthermore the analysis results from FEA also confirmed that there was a predominance of tensile stresses causing fracture in the SBS tests [17].

From many research works as afore mentioned, the applied shear force gives rise to the appearance of tensile stress, compressive stress and shear stress and the bond failure is likely to occur due to tensile stress. In order to provide a clearer image of forces or stresses induced at the adherend interface and their relationship, in this work the in vitro tests of the shear bond strength of adhesion bonds between dentine–cement (DCe), crown–cement (CrCe), dentine–cement–crown (DCeCr) and the cohesion bond of cement–cement (CeCe) were comparatively measured. The commercial cement and another two types of laboratory prepared cements were used for bonding preparation of all specimens. All three types of stresses and forces at the adherend interface were analyzed using the two points bonding model.

2. Experimental details

2.1. Materials and methods

Two types of zinc phosphate dental cements were prepared from reagent grade chemicals with the weight percentage composition of: 90 ZnO–8.2 MgO–0.1 Bi_2O_3–0.5 SrF_2–1.2 SiO_2. The powder mixture was heated at 1200 °C for 5 h in air, ground to the particle size of about 10 μm after cooling to room temperature and denoted as the cement L1. The second type cement (cement L2) was prepared by the extra addition of 0.5 wt% silica nanofiller to the calcined powder mixture of the cement L1. The prepared cements were comparatively tested with the commercial zinc phosphate cement (ZPC, Hybrid, SHOFU, Japan, lot no. 4080).

Non-carious and non-endodontically treated permanent human molars were selected for the bond strength test and the artificial acrylic crown was used for the comparative test. The teeth were cleaned and stored in 0.1% thymol solution for not exceeding 6 months before the bond strength test. The root of each tooth was removed and the crown was embedded in a clear resin and left the buccal side outward. The embedded tooth was ground in order to obtain a flat dentin surface with the area of at least 10 mm^2 which was suitable for the bonding procedure. The dentin surface was polished with a 600 grit SiC paper then cleaned with distilled water and dried in air.

The self-cure-acrylic resin (UNIFAST, ivory color, GC Dental Products Corporation, Japan) was used for preparation of artificial crowns and substrate. The crown substrate for bonding strength test was prepared from the hardened artificial crowns using the same surface preparation as that of the tooth substrate. The cylindrical crown rod with a diameter of 3 mm and 4 mm height was prepared using stainless steel mold. The crown substrate and the crowns rod surfaces were ground with a 600 grit SiC paper, then etched by 9% hydrofluoric acid and dried in air.

The DCe and CrCe specimens for shear test were prepared by placing the 2 mm thick wax sheet with a hole diameter of 3 mm on the tooth surface or crown substrate being bonded with cements. The cement paste was poured into the wax hole and kept at 37 °C in air under a high relative humidity of about 90% for 1 h. After setting, the wax was removed carefully while the specimens were continually kept for 24 h prior to the shear test.

On preparation of the DCeCr specimen, the 3 mm diameter cylindrical crown rods were bonded with cements to the tooth substrate and aligned in to a vertical direction for the DCeCr shear test specimens. The constant compressive load of 150 N was applied for 5 min in order to remove the excess cement and to ensure the consistency of the cement film thickness of about 25 μm [18]. The DCeCr specimens were kept and tested with the same condition as that of the DCe specimen.

The specimen for the cohesion shear strength test of pure cement (CeCe) was prepared in to a cylindrical rod shape with a diameter of 3 mm and 6 mm height. The cement paste was poured into the stainless steel mold within 2 min and kept for 1 h at 37 °C in air with a relative humidity of 90–100%. After cement setting, the cement rod was removed from the mold and the surface was ground with 600 grit SiC paper, and then continually kept with the same condition for 6 h. After that, the part of the cement rod was embedded in a clear resin and left the part with 3 mm height above the resin surface and kept for 24 h prior to shear testing with the same condition as that of the DCe specimen.

2.2. Wettability test

In these experiments the as-prepared zinc phosphate cement is homogeneous viscous liquid containing fine solid particles with