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Microtensile bond strength of a resin cement to a novel fluorcanasite glass-ceramic following different surface treatments

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ABSTRACT

Objectives. This study evaluated the effect of surface treatments on the bond strength of fluorcanasite and lithium disilicate glass-ceramics, with the possibility of eliminating HF etching of these ceramics.

Methods. Fifteen blocks of an experimental fluorcanasite and a lithium disilicate glass-ceramic (IPS e.max CAD®) were assigned to one of the following three surface treatments: (1) machined with 60 μm finish, (2) machined and grit blasted, (3) machined and HF etched. The ceramic blocks were duplicated in composite resin (Spectrum®) and cemented together with a resin luting agent (Variolink II®). Thirty microbars per group (1.0 × 1.0 × 20 mm) were obtained and subjected to a tensile force at a crosshead speed of 0.5 mm/min using a universal testing machine until failure. The mode of failure was determined using scanning electron microscopy. The appropriate bonding procedure was assessed for durability by storing in water at 100 °C for 24 h. Statistical analyses were performed with ANOVA and Tukey's test ($P < 0.05$).

Results. Machining alone significantly increased the bond strength (MPa) of the fluorcanasite (27.79 ± 6.94) compared to the lithium disilicate (13.57 ± 4.52) ($P < 0.05$). HF etching resulted in the lowest bond strength (8.79 ± 2.06) for the fluorcanasite but the highest for the lithium disilicate (24.76 ± 9.38). Regarding durability, the machined fluorcanasite (15.24 ± 5.46) demonstrated significantly higher bond strength than the machined and HF etched lithium disilicate (12.28 ± 3.30).

Significance. The fitting surface of the fluorcanasite glass-ceramic should retain the machined finish and be directly treated with silane. The use of HF acid is contraindicated.

1. Introduction

Chain silicates, or inosilicates, are polymeric crystals in which single or multiple chains of silica tetrahedra form the structural backbone. In the late 1970s, Beall [1] demonstrated that glass-ceramics based on modified chain silicate compositions (enstatite, potassium fluorrichterite and canasite) have a particularly high fracture toughness (3–5 MPa $m^{1/2}$) and bending strength (200–300 MPa). Fluorcanasite is a synthetic double

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chain silicate glass-ceramic displaying a combination of high flexural strength and fracture toughness in comparison with currently available resin-bonded glass-ceramic restorative systems and is a potential material for all-ceramic restorations. In addition, fluorcanasite, unlike many high strength dental ceramics, has a surface that could be bonded with an adhesive composite resin luting agent via a silane coupling agent.

Success with resin-bonded all-ceramic restorations is highly dependent on obtaining a durable and reliable bond, which has to integrate all parts of the system into one coherent structure. This bond is usually created by: (1) micromechanical retention by hydrofluoric acid etching and/or grit blasting, and; (2) chemical bonding by a silane coupling agent [2,3]. Etching the inner surface of a restoration with hydrofluoric acid followed by the application of a silane coupling agent is a well known and recommended method to increase the bond strength [4–6]. However, previous studies have challenged this protocol. Hooshmand et al. [2] and Aida et al. [7] found that the hydrofluoric acid etching stage could be eliminated for the bonding procedure whereas Sorensen et al. [8] reported that the use of a silane coupling agent was of no significant benefit. Shimada et al. [9] reported that hydrofluoric acid etching glass-ceramics adversely affects ceramic bonding and is probably not necessary for clinical applications. Glass-ceramics with a fine crystalline structure such as fluorcanasite may not benefit from hydrofluoric acid etching. Other researchers have demonstrated that the new generation of ceramic primers can strongly couple to machinable glass-ceramics without prior gritblasting or hydrofluoric acid etching of the ceramic surface [10,11].

There are possibly three good reasons why it would be desirable to remove the hydrofluoric acid etching step from the procedure: (1) hydrofluoric acid is a highly toxic chemical, representing a potentially serious health hazard [12]; (2) it has been reported that hydrofluoric acid etching of silica-based ceramics produces insoluble silica-fluoride salts, which can remain as by-products on the surface [9]. If not removed, these by-products can interfere with the bond strength to the resin; (3) its elimination from the bonding procedure would be highly advantageous, but would only be possible if the silane bond can be shown to be adequate [2].

Various investigations have demonstrated that using adhesive composite resin cements increases the fracture resistance of glass-ceramic restorations, provides high retention, improves marginal adaptation and prevents microleakage by penetrating surface flaws and irregularities and inhibiting crack propagation [13–16]. Fracture resistance of the ceramic–resin bond is controlled primarily by the microstructure and surface treatment of the ceramic [17,18]. Therefore, it is essential that an optimal bonding protocol is developed. Because fluorcanasite is a chain silicate glass-ceramic, it is hypothesized that it is possible to achieve a reliable bond using a silane coupling agent and resin cement. Due to the fine grain, acicular microstructure of fluorcanasite, it may be possible to eliminate the hydrofluoric acid etching stage from the cementation procedure.

2. Materials and methods

2.1. Ceramic materials

Two CAD/CAM machinable glass-ceramic core materials were employed in this study; an experimental fluorcanasite glass-ceramic (University of Sheffield) and a commercial lithium disilicate glass-ceramic (e.max CAD®, batch number JO8179, Ivoclar Vivadent AG, Schaan, Liechtenstein).

2.2. Surface preparation

Four different surface treatments were performed on disc specimens of the fluorcanasite and lithium disilicate glass-ceramic:

- Polished to 1 μm finish with 400–1200-grit wet silicon carbide paper, then 3 and 1 μm diamond polishing paste using a polishing machine (Buehler Metaserv, UK).
- Machined finish using a 60 μm diamond bur (Henry Schein, Germany).
- Machined and grit blasted with 50 μm aluminium oxide particles (MicroEtcher, Danville Engineering, San Ramon, CA).
- Machined and etched with hydrofluoric acid (HF) (Ultradent Porcelain Etch 9.5% Buffered, Ultradent Products, South Jordan, UT) for 1 min, then rinsed and air dried for 1 min.

A surface roughness profile was determined for each of the groups using a profilometer (Mitutoyo Surftest 301, Mitutoyo America Corp, Aurora, IL). A diamond stylus (5 μm radius) was used under a constant measuring force of 3.9 N. The instrument was calibrated using a standard reference specimen, and then set to travel at a speed of 0.1 mm/s with a range of 600 μm during testing. The roughness of the specimen was analyzed by performing two passes of the profilometer, with one pass at a 90° angle to the other. Ten recordings per specimen ($n = 3$) in each surface treatment group were obtained.

Following completion of the profilometric evaluation, SEM analysis was performed to ascertain the effects of the different surface treatments on the microstructure of the core materials. The specimens were gold coated with a sputter coater (Evaporation unit, Edwards, UK), mounted on coded brass stubs and examined using scanning electron microscopy (Philips XL-20).

2.3. Microtensile bond strength testing

Fifteen 1 × 1 × 1 cm blocks were prepared from the fluorcanasite and the lithium disilicate glass-ceramics. The specimens were polished with 400-grit through to 1200-grit wet silicon carbide paper using a polishing machine (Buehler Metaserv, UK). Following this, the ceramic blocks were ultrasonically cleaned (Biosonic UC300, Whaledent, Altstätten, Switzerland) in distilled water for 5 min to remove any contamination from the silicon carbide papers. Each ceramic block was duplicated in composite resin (Spectrum TPH, batch no. 0506003114, Dentsply DeTrey GmbH, Konstanz, Germany) with the same dimensions using a mould made of a polyvinylsiloxane impression material (Aquasil, Dentsply DeTrey GmbH, Kon-