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Effect of sandblasting, etching and resin bonding on the flexural strength/bonding of novel glass-ceramics

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ABSTRACT

Objectives. To process novel leucite glass-ceramics and test the effects of surface treatment and resin bonding on the biaxial flexural strength (BFS) and shear bond strength (SBS).

Methods. Alumino–silicate glasses were ball-milled, and heat treated to form leucite glass-ceramics (LG-C, OLG-C), then sintered into ingots. Ingots were heat extruded into a refractory mould to form disc specimens (1.3 × 14 mm diameter). IPS e.max® was used as a commercial comparison. Glass-ceramic test groups were sandblasted (Groups. 1, 4, 6), sandblasted, etched and adhesively bonded (Groups. 2, 5, 7) or lapped, etched and adhesively bonded (Groups. 3, 8). Specimens were adhesively bonded with Monobond S, followed by the application of Variolink II® cement and light curing. BFS testing was at 1 mm/min and SBS testing at 0.5 mm/min. Samples were characterised using XRD, SEM and profilometry.

Results. XRD confirmed tetragonal leucite in LG-C/OLG-C and lithium disilicate/lithium orthophosphate in IPS e.max®. Mean BFS (MPa (SD)) were: Gp1 LG-C; 193.1 (13.9), Gp2 LG-C; 217.7 (23.0), Gp3 LG-C; 273.6 (26.7), Gp4 OLG-C; 255.9 (31); Gp5 OLG-C; 288.6 (37.4), Gp6 IPS e.max®; 258.6 (20.7), Gp7 IPS e.max®; 322.3 (23.4) and Gp8 IPS e.max®; 416.4 (52.6). The Median SBS (MPa) were Gp1 LG-C; 14.2, Gp2 LG-C (10 s etch); 10.6 and Gp3 IPS e.max®; 10.8. Mean surface roughness was 5–5.1 μm (IPS e.max®) and 2.6 μm (LG-C).

Significance. Novel leucite glass-ceramics with reduced flaw size and fine microstructures produced enhanced BFS and SBS by resin bonding. These properties may be useful for the fabrication of minimally invasive aesthetic and fracture resistant restorations.

1. Introduction

Leucite ($KAlSi_2O_6$) glass-ceramics are desirable for the fabrication of dental restorations due to their excellent aesthetic properties, which simulate natural tooth appearance and their low cytotoxicity [1]. The high thermal expansion coefficient of tetragonal leucite ($20.5 \times 10^{-6}/^{\circ}C$) [2], makes it a useful compo-

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nent in veneering materials for high strength metal-ceramic restorations [3]. Leucite glass-ceramics can also be fabricated into a variety of all-ceramic restorations adhesively bonded to dentine–enamel tooth structure [4,5], and encouraging a more conservative tooth preparation [6]. Restorations can be processed by heat extruding glass-ceramic ingots into a refractory mould prepared by the lost wax technique, then finished by extrinsically staining to simulate the natural characteristics of the tooth [7]. Heat extrusion increases densification and is associated with higher flexural strength due to crystallite dispersion and a more homogeneous crystal distribution [8,9]. Typical properties are a reported K_{IC} of 1.33 (0.08) MPa $m^{1/2}$ and flexural strengths in the range of 75.7–165 MPa [10,11]. Mackert et al. [12] suggested that inherent flaws associated with the cubic to tetragonal transformation were reduced by synthesizing crystals in a critical size range (<4 μm). The synthesis and heat extrusion of a fine grained (<4 μm) leucite glass-ceramic resulted in a high flexural strength of (mean (SD)) 245 (24.3) MPa and high reliability (weibull m = 11.9) [13]. Heat extrusion and processing including sandblasting and finishing are however, associated with a range of critical flaws, which when under tensile stress cause premature failure by various failure modes, initiated at occlusal contacts or cementation surfaces [14]. Resin bonding of leucite glass-ceramic restorations is advantageous in this respect as they are significantly strengthened by this modification to their internal surfaces [15]. Internal ceramic surfaces can be sandblasted and etched to gain micromechanical retention, followed by silane bonding agents wetting and bonding to the ceramic surface. The organo-functional group in the silane next forms a bond with the resin cement [16,17]. Effective resin-ceramic bonding of glass-ceramic restorations takes advantage of increased surface area for bonding to tooth structure to gain retention [15,18] and reinforcement [5], and a clinically acceptable marginal fit [19,20]. There is also the advantage of significant strengthening effects related to resin elastic modulus and thickness [21,22]. Some pre-resin bonding surface treatments such as sandblasting, in addition to improving micro roughness, can change critical flaw populations and degrade strength [23]. Hydrofluoric (HF) acid etching has also been found to reduce the biaxial flexural strength of leucite glass-ceramics [24], and the type of silane employed can influence bond strengths [25]. When developing new glass-ceramic formulations the glass/crystal phase chemistry, leucite crystal size, number and distribution [15], and physical properties influence the resultant bonding surface area and structure after pre-cementation treatments. The subsequent micromechanical retention and wettability of these surfaces is important to achieve effective adhesive resin bonding [26]. The authors have synthesised a unique range of new leucite glass-ceramics with high leucite volume fraction and small crystallite size for the first time [27]. It is therefore key to asses these ceramics after scale-up and following processing and cementation procedures, to realise the optimisation of this important category of materials and its benefits for minimally invasive adhesive dentistry. Therefore, the aims of this study were to process novel leucite glass-ceramics (LG-C, OLG-C) using heat extrusion and to analyse the effects of sandblasting, etching and resin bonding on the biaxial flexural strength and the shear bond strength of the glass-ceramics.

2. Materials and methods

2.1. *Preparation of sandblasted specimens*

An alumino–silicate glass with the following composition (mol %) was commercially synthesized (Lot nos: F-0356, 92100111, glasses supplied by Davis Schottlander Davis Ltd., UK and Cera Dynamics Ltd, Stoke-on-Trent, UK): SiO_2 (69.7%), Al_2O_3 (10.6%), K_2O (12.8%), CaO (1.5%), TiO_2 (1.3%), Na_2O (1.9%), Li_2O (1.6%), B_2O_3 (0.7%) by heating in a high temperature custom made furnace (Cera Dynamics Ltd, UK) at 10 °C/min to 1550 °C (5 h hold). The glass was air quenched and allowed to cool to room temperature. The glass frit was crushed, ball-milled for 1 h and screened to 125 μm (LG-C). To optimise the glass-ceramics another batch of glass was also produced using the same parameters but quenched in water and ball milled for 93 h, followed by spray drying (Niro Atomizer, Denmark) of the powder (OLG-C).

The glass powders (LG-C and OLG-C) were placed into refractory trays (IPS press Vest speed, Ivoclar-Vivadent, Lot no: Powder TL3033 and Liquid TL3022) and heated in a furnace (Lenton 1600, Hope Valley, UK) at 10 °C/ min to 592 °C (1 h hold), then ramped at 10 °C/min to 1040 °C (30 min hold). The leucite glass-ceramic (LG-C) and optimised leucite glass-ceramic (OLG-C) were air quenched, ball-milled and screened through a 125 μm sieve (Endescott Ltd, London, UK). To fabricate glass-ceramic ingots 1.6 g of LG-C or OLG-C powder was dry compacted using a custom-made steel die and punch (diameter 13.0 mm, Specac Ltd., Slough, UK), by applying 0.5 bar pressure for 30 s using a hydraulic press (Quayle Dental, HBP 153, UK). Compacted powder ingots were ramped from 538 °C at a rate of 38 °C to 1060 °C, under partial vacuum (55 h Pa) and held for 2 min in a porcelain furnace (Multimat 2 Touch + Press, Dentsply, Weybridge, UK).

IPS e.max® (LT A3, Ivoclar-Vivadent, Lot no: T45580) was used as a commercial comparison. Perspex (ICI Plastics, UK) discs (1.3 mm depth × 14 mm diameter) were sprued onto muffle bases with a surrounding silicon cylinder. Disc specimens were invested using 200 g of investment material (IPS Press VEST speed powder, Lot no: TL3033), mixed with 32 ml liquid (Lot no: TL3022) and 22 ml distilled water and vacuum mixed (Renfert twister; E10022C6, Germany) at 350 rpm for 2.3 min. The investment was poured into the moulds under vibration and allowed to set for 45 min, then placed into a furnace (Renfert Magma, B1099520, Germany) preheated to 850 °C and allowed to dwell for 60 min. The LG-C, OLG-C or IPS e.max® ingots (at room temperature) were placed into hot refractory moulds, then heat pressed using a press furnace (Programat EP 3000, Ivoclar-Vivadent, Schaan Liechtenstein) according to the protocols in Table 1. LG-C, OLG-C and IPS e.max® specimens were divested via a sandblasting unit (Renfert Basic Quattro, Germany) using 50 μm glass beads (07M509B, Bracon Ltd, UK) at 2 bar pressure. The sandblasting nozzle was held at 10 mm from specimen's surface and at 45° to the specimens. Sprue areas were cut off using a diamond disc (006

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