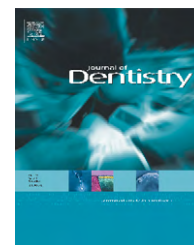


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Development and testing of multi-phase glazes for adhesive bonding to zirconia substrates

Polyxeni Ntala^a, Xiaohui Chen^b, Jason Niggli^a, Michael Cattell^{a,*}

^a Centre of Adult Oral Health, Queen Mary University of London, Barts and The London School of Medicine and Dentistry, Institute of Dentistry, Turner Street, London, E1 2AD, UK

^b Dental Physical Sciences, Queen Mary University of London, Barts and The London School of Medicine and Dentistry, Institute of Dentistry, Mile End Road, E1 4NS, UK

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ABSTRACT

Objectives: The aims of the study were to develop and test multi-phase glaze coatings for zirconia restorations, so that the surface could be etched and adhesively bonded.

Methods: Zirconia disc specimens ($n = 125$, 16 mm × 1 mm) were cut from cylinders of Y-TZP (yttria-stabilized tetragonal zirconia polycrystals) ZS-Blanks (Kavo, Everest) and sintered overnight. Specimens were subjected to the recommended firing cycles, and next sandblasted. The specimens were divided into 5 groups of 25, with Group 1 as the sandblasted control. Groups 2–5 were coated with overglaze materials (P25 and IPS e.max Ceram glazes) containing secondary phases. Group 2 was (wt%): 10% hydroxyapatite (HA)/P25 glaze, Group 3: 20% IPS Empress 2 glass–ceramic/glaze, Group 4: 20% IPS Empress 2 glass/glaze and Group 5: 30% IPS Empress 2 glass/glaze. After sintering and etching, Monobond-S and composite resin cylinders (Variolink II, Ivoclar-Vivadent) were applied and light cured on the test surfaces. Specimens were water stored for 7 days. Groups were tested using the shear bond strength (SBS) test at a crosshead speed of 0.5 mm/min. Overglazed and the fractured specimen surfaces were viewed using secondary electron microscopy. Room and high temperature XRD and DSC were carried out to characterize the materials.

Results: The mean (SD) SBS (MPa) of the test groups were: Group 1: 7.7 (3.2); Group 2: 5.6 (1.7); Group 3: 11.0 (3.0); Group 4: 8.8 (2.6) and Group 5: 9.1 (2.6). Group 3 was significantly different to the control Group 1 ($p < 0.05$). There was no significant difference in the mean SBS values between Group 1 and Groups 2, 4 and 5 ($p > 0.05$). Group 2 showed statistically lower SBS than Groups 3–5 ($p < 0.05$). Lithium disilicate fibres were present in Groups 3–5 and fine scale fibres were grown in the glaze following a porcelain firing cycle (Groups 4 and 5). XRD indicated a lithium disilicate/minor lithium orthophosphate phase (Group 3), and a tetragonal zirconia phase for the sintered Y-TZP ZS-Blanks. DSC and high temperature XRD confirmed the crystallization temperatures and phases for the IPS Empress 2 glass.

Conclusions: The application of a novel glass–ceramic/glaze material containing a major lithium disilicate phase might be a step in improving the bond strength of a zirconia substrate to a resin cement.

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* Corresponding author. Tel.: +44 (0)207 377 0000x2160; fax: +44 (0)207 377 7064.

E-mail address: m.cattell@qmul.ac.uk (M. Cattell).

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1. Introduction

Computer-aided design/computer-aided machining (CAD/CAM) systems allow for the routine use of high strength ceramic materials in the fabrication of indirect aesthetic restorations. Improved load bearing capacity, reliability and biocompatibility are reported for yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramics.^{1,2} The versatility of the Y-TZP ceramics means their applications have broadened from core materials for crowns and fixed partial dentures to implant abutments and posts.^{3,4} “Y-TZP” ceramics have superior mechanical properties with flexural strengths of 900–1200 MPa and a fracture toughness of 9–10 MPa m^{1/2}.⁵ These ceramics can also benefit from phase transformations and microcrack toughening mechanisms, with subsequent R curve behaviour.⁶

The clinical success of fixed ceramic prostheses are heavily dependent on the cementation procedure,⁷ with current Y-TZP prostheses cemented using conventional cements (zinc phosphate and glass ionomer) recommended by the manufacturers.⁸ The retention of non-adhesively bonded restorations is however, subject to the geometry of the crown preparation, which if less than ideal can be linked to restoration failure.⁹ Adhesive resin bonding of acid etched ceramic restorations to etched tooth structure is beneficial as there are increases in surface area for bonding, improved retention^{10,11} and reduced microleakage¹² compared with conventional cements. Strengthening of the ceramic restoration^{13,14} and the abutment tooth are also consequences of the adhesive bonding procedures.¹⁰ A successful bond between the ceramic and resin cement is achieved through the formation of chemical bonds and the micromechanical interlocking of resin to the ceramic surface,¹¹ and is well established for glass–ceramics with high silica content.¹⁵ The bond is provided by selective etching of glassy or crystalline components in the ceramic to produce a microporous surface. The increased surface area encourages resin penetration into the microretentive ceramic surface. The addition of a silane bonding agent produces a siloxane bond between the silica contained in the ceramic and the organic matrix of the resin cement to enhance this bond.^{16,17} Silane coupling agents also wet the substrate surface, lower surface tension and allow hydrophobic resin composites to adhere to hydrophilic silica based glasses and glass–ceramics.¹⁸ Zirconium dioxide ceramics are not silica based and present a challenge for reliable resin bonding as chemical silica–silane bonds cannot be established. Due to their high crystalline content and limited glassy phase, the application of acidic agents (hydrofluoric or phosphoric acid) will not create a sufficiently roughened surface for enhanced micromechanical retention.^{19,20} To overcome this problem, surface pre-treatments have been suggested including; tribochemical silica coating by particle abrasion,²¹ PyrosilPen-Technology using flame treatment/silane deposition,²² selective infiltration etching (SIE)²³ of the surface and the use of cements containing the phosphate ester monomer 10-methacryloyloxydecyl dihydrogenphosphate (MDP).²⁴ Durable bis-GMA resin bonds to Y-TZP ceramics were not established using some of these methods,²⁵ and roughness created by sandblasting was thought to be the main bonding mechanism for MDP monomers.²³ Several of these methods (SIE and MDP monomers) have also been combined with novel reactive primer monomers giving initial

high bond strengths which were reduced after thermocycling.²⁶ Organophosphate/carboxylic acid monomers used as a primer for zirconia substrates²⁷ or the deposition of a surface Si_xO_y “seed” layer are also promising,^{27,28} but their long term durability and hydrolytic stability is unknown.

Two-phase glaze coatings applied to glass–ceramic surfaces and acid etched previously provided high shear bond strengths to adhesive resins and increases in glass–ceramic flexural strength.²⁹ This approach was thought useful to enhance adhesive bonding to zirconia substrates via the creation of favourable micromechanical and chemical bonds. Viscous flow of the applied glaze should heal substrate processing damage,³⁰ seal the surface from moisture and reduce the chances of low temperature strength degradation.^{6,26,31} Therefore, the aims of the study were to develop and test multi-phase glaze coatings which could be applied to the fit surface of zirconia restorations, so that the surface could be effectively etched and adhesively bonded. The hypotheses were that the shear bond strength of the zirconia substrate would be improved by the application of a two phase etched glaze material.

2. Materials and methods

2.1. Zirconia specimen preparation

One hundred and twenty five zirconia disc specimens (16 mm diameter × 1 mm thickness) were cut from cylindrical Y-TZP ZS-Blanks (Lot nos; 100691445, 100709358 and 10078572, Kavo, Germany) using a diamond disc (NR 1575202, Girrbach Dental, Germany) mounted in a cutting machine (Cutman 100, Zeiser II system, Girrbach Dental, Germany). Specimens were sintered at 1500 °C in a furnace (Kavo Everest[®] Therm, Kavo, Germany) according to the manufacturer’s instructions. Specimens were then fired in a porcelain furnace (Multimat MCII, Dentsply, UK) using one zir liner, two dentine, one stain and one glaze firings according to the manufacturer’s recommendation firing cycles (IPS e.max[®] ZirPress manual, Ivoclar-Vivadent, Liechtenstein), to mimic restoration construction. All specimens were sandblasted using 50 μm alumina oxide at 2.5 × 10⁵ Pa pressure for 13 s at a distance of 10 mm. This surface treatment was applied to the entire disc surface to be adhesively bonded.

2.2. Experimental overglaze material development

Four overglaze coating materials (Table 1) were developed by mixing hydroxyapatite (HA, P209S, Plasma Biotol Ltd., UK), IPS Empress 2 glass–ceramic (Ivoclar-Vivadent) or IPS Empress 2 glass powder with commercially available glaze materials. The glazes used were P25 glaze (Lot no: 326389, Pemco Corp., Canada) and IPS e.max Ceram glaze (Lot no: J27558, Ivoclar-Vivadent).

2.3. Test specimen fabrication

The sandblasted zirconia disc specimens (125) were divided into 5 groups of 25, one of which was used as a control (Group 1). The overglaze materials were mixed with modelling liquid (IPS Classic, Lot no: E15945, Ivoclar-Vivadent) and applied to

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