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THE STRENGTHENING OF RESIN CEMENTED DENTAL CERAMIC MATERIALS

A thesis submitted to the University of Dublin in fulfilment of the requirements for

The degree of Doctor of Philosophy by

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Materials Science Unit

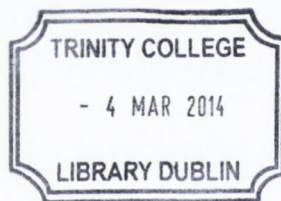
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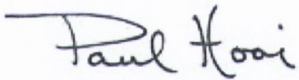
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The mentoring contribution of my supervisors in the preparation of the publications presented herein is respectfully acknowledged.



Paul Hooi



SUMMARY

The aim of the current investigation was to advance the understanding of the mechanism of resin-strengthening conferred to dental ceramic materials by resin-based composite materials. The investigation is presented as a series of manuscripts. In the first study (Manuscript 3.1), dental porcelain disc-shaped specimens were resin-coated with three resin-based composite materials with different flexural moduli at discrete resin thicknesses. The discs were loaded to failure in a biaxial flexure test and the maximum tensile stress calculated with an analytical solution to determine the magnitude of resin-strengthening. Linear regression techniques were utilised to identify the theoretical strengthening at a 'zero' resin-coating thickness. A finite element analysis model was employed to verify the analytical solution and validate the experimental data. The flexural modulus and thickness of the resin-based composite materials were observed to have a significant impact on the magnitude of resin-strengthening. A soda lime glass analogue material for dental porcelain was identified to reduce the variability associated with specimen preparation (Manuscript 3.2). The study examined the influence of pre-cementation operative variables (alumina particle air abrasion and HF acid-etching) and resin-cement coating on the biaxial flexure strength of the soda lime glass analogue for dental porcelain. The soda lime glass analogue demonstrated the same patterns of strength dependence influenced by pre-cementation parameters and resin-cementation variables characterised previously for dental porcelain. Load to failure testing of dental ceramic materials is typically performed at a constant standardised testing rate. The soda lime glass analogue for dental porcelain was employed to investigate the influence of testing rate and cementation seating load on the pattern of resin-strengthening at speeds relevant to the clinical masticatory cycle (Manuscript 3.3). Testing rate and resin-cementation seating load were observed to significantly modify resin-strengthening mediated by the resin-ceramic

hybrid layer. The impact of environmental moisture introduced during the resin-cementation process on the observed pattern of resin-reinforcement was difficult to establish. Moisture was subsequently included as a controlled factor during the resin-cementation of the soda lime glass discs to further elucidate information regarding the pattern of resin-reinforcement when resin-cemented in a moisture scavenged environment (Manuscript 3.4). The findings demonstrated the dynamic susceptibility of soda lime glass to slow crack growth by identifying a rate-dependent pattern resin-reinforcement. The decrease in penetration expected from the resin in cracks within the 'resin-ceramic hybrid layer' following the removal of the higher seating load was proposed as the associated modifying parameter for the resin-strengthening behaviour. The geometric form of dental ceramic restorations is complex and the impact of clinically relevant curvatures on the fracture behaviour of dental ceramic materials was investigated with the biaxial flexure testing of curved discs of different radii of curvature (Manuscript 3.5). The finite element method was utilised to determine the maximum tensile stress for the load at failure, in the absence of an analytical solution. Significant increases in the mean load to failure were observed as the radius of curvature was reduced (more severely curved). However, the maximum principal stress was not observed to demonstrate a similar sensitivity to radius of curvature for the curvatures investigated. The sensitivity of load to failure data to specimen shape was highlighted. The findings demonstrated that comparisons between load to failure data in isolation is problematic and the complications associated with the load to failure testing of full contour crowns.

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1. INTRODUCTION

1.1 INTRODUCTION TO DENTAL PORCELAIN

The increasing patient demand for aesthetic and functional restorative treatment of natural dentition continues to challenge the dental profession to explore methods for perfecting all-ceramic restorations. Dental ceramics do not elicit a pulpal response from vital teeth and resist wear in the oral environment extremely well which makes them an ideal candidate material to replace natural dentition. When compared with metal or porcelain-fused-to-metal (PFM) restorations, dental ceramic materials also offer improved aesthetics for use as porcelain laminate veneers (PLVs), dentine bonded crowns (DBC's) and fixed partial dentures (FPDs), despite their inherent brittleness (Piddock and Qualtrough, 1990). Unfortunately, Kingery et al. (1976a) proposed that ceramic materials were susceptible to brittle fracture initiated at defects which were either inherent to the material (grain size, microcracks) and/or introduced during the ceramic fabrication process (surface irregularities, porosities, impurities, geometric defects).

History of dental porcelain

Contemporary aesthetic restorative dental ceramics have evolved from the work of the French dentist Nicholas Dubois de Chemant in 1792 when he obtained a patent for a process to fabricate porcelain teeth using a mixture of kaolinite, potash feldspar and quartz (Koch, 1910). However, Chamant's dental porcelain was opaque and in 1838 Wildman achieved increased translucency by removing the clay component (kaolinite) from the formulation (Capon, 1927). The removal of the clay component from the mullite region of the silica-leucite-mullite ternary phase diagram, resulted in translucent aesthetics because the refractive

index of mullite was greater than that of the feldspathic glass matrix (Capon, 1927). Beers of California is credited with the idea of the gold shell crown in 1873 from which the porcelain jacket crown (PJC) is considered to have originated (Wells, 1901). The process of fabricating PJCs was developed by Charles Land (Land, 1903a,b) and the same method is universally used today, although improvements in materials and technique have occurred over the past century. However, the susceptibility of dental porcelain to fracture when employed clinically, under relatively low service loads, continues to be the predominant criticism of the dental profession.

The patenting of the first commercially successful dental gold alloy by Weinstein et al. (1962) offered the first real opportunity to strengthen dental porcelain resulting in the beginning of PFM restorations. In essence the metal substructure married the structural integrity of the cast metal alloy provided by the mechanical properties (strength, rigidity) while the aesthetic properties of the dental porcelain. However, while PFM restorations are still in use today, their aesthetic appeal is limited by difficulties in masking the underlying metal. Developments in ceramic materials were championed by Binns (1962) who attempted to dispersion strengthen glasses with heat pressed mixtures of alumina or zirconia crystals. Binns (1962) demonstrated that a strength increase was obtained when the proportion of dispersed ceramic crystal was increased, albeit in the absence of (i) a sudden volume change associated with the crystal structure or (ii) a difference in the thermal expansion coefficients between the glass and the dispersed ceramic inclusion. For a crack to propagate in a composite system, Binns (1962) highlighted that where the thermal expansion coefficients of both the glassy matrix and inclusion crystals were similar, the fracture path travelled indiscriminately through both glass and crystal phases. Therefore, the increased strength and elastic modulus of the inclusion crystals carried a greater proportion of the load and the

energy required to fracture the composite material was greater than that for the glass alone and dispersion strengthening was born.

McLean and Hughes (1965) selected alumina as the ideal candidate crystalline ceramic oxide to dispersion strengthen feldspathic porcelain on the basis of fusion temperature, resistance to thermal shock, thermal expansion coefficient, mechanical strength in the fired state, bonding properties with dental porcelain, colour and aesthetics, which led to the development of aluminous core porcelains. Figure 1.1 which was taken from the McLean and Hughes (1965) manuscript highlights how the thermal expansion coefficient of both the feldspathic porcelain glass and alumina inclusion crystals were matched as the fracture path travelled indiscriminately through the composite material structure.

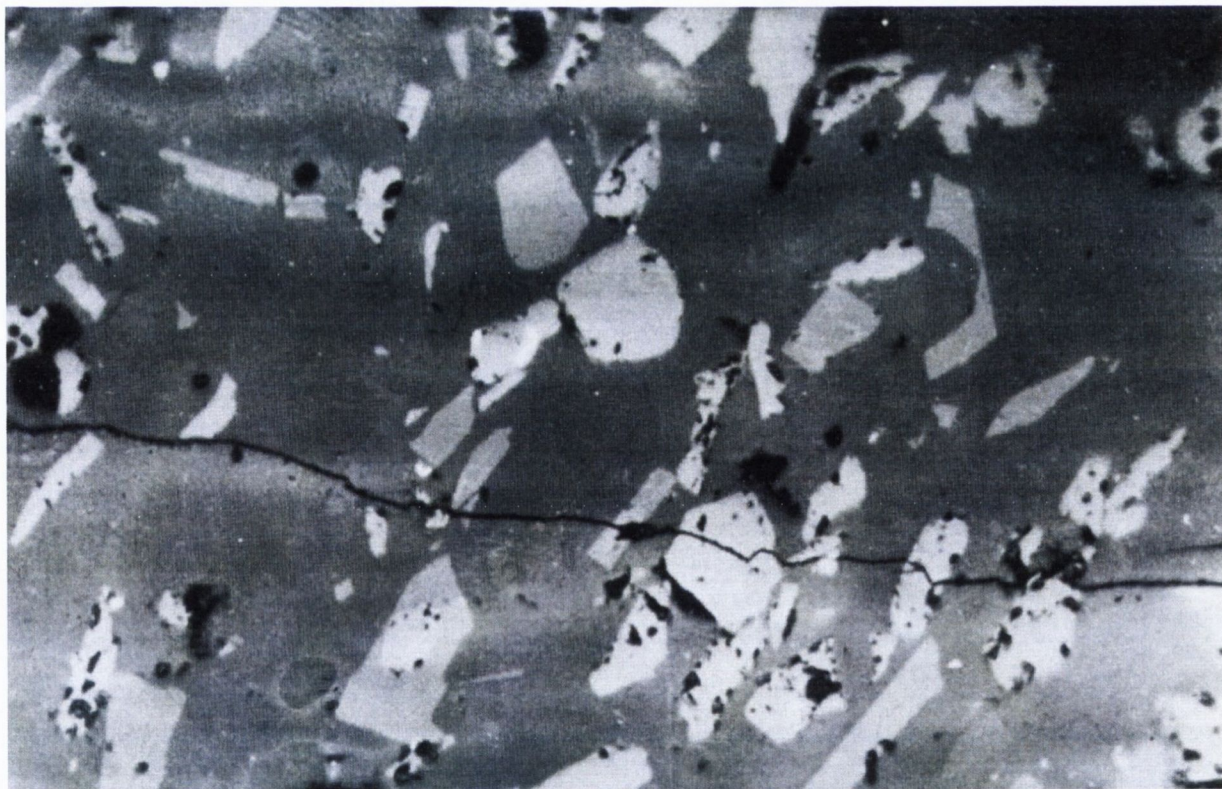


Figure 1.1: Scanning electron micrograph showing the fracture path in a two-phase glass-crystal material, each with equal thermal coefficients of expansion which illustrates the fracture path progressing indiscriminately through both the glass and crystal phases. Adapted from McLean and Hughes (1965).

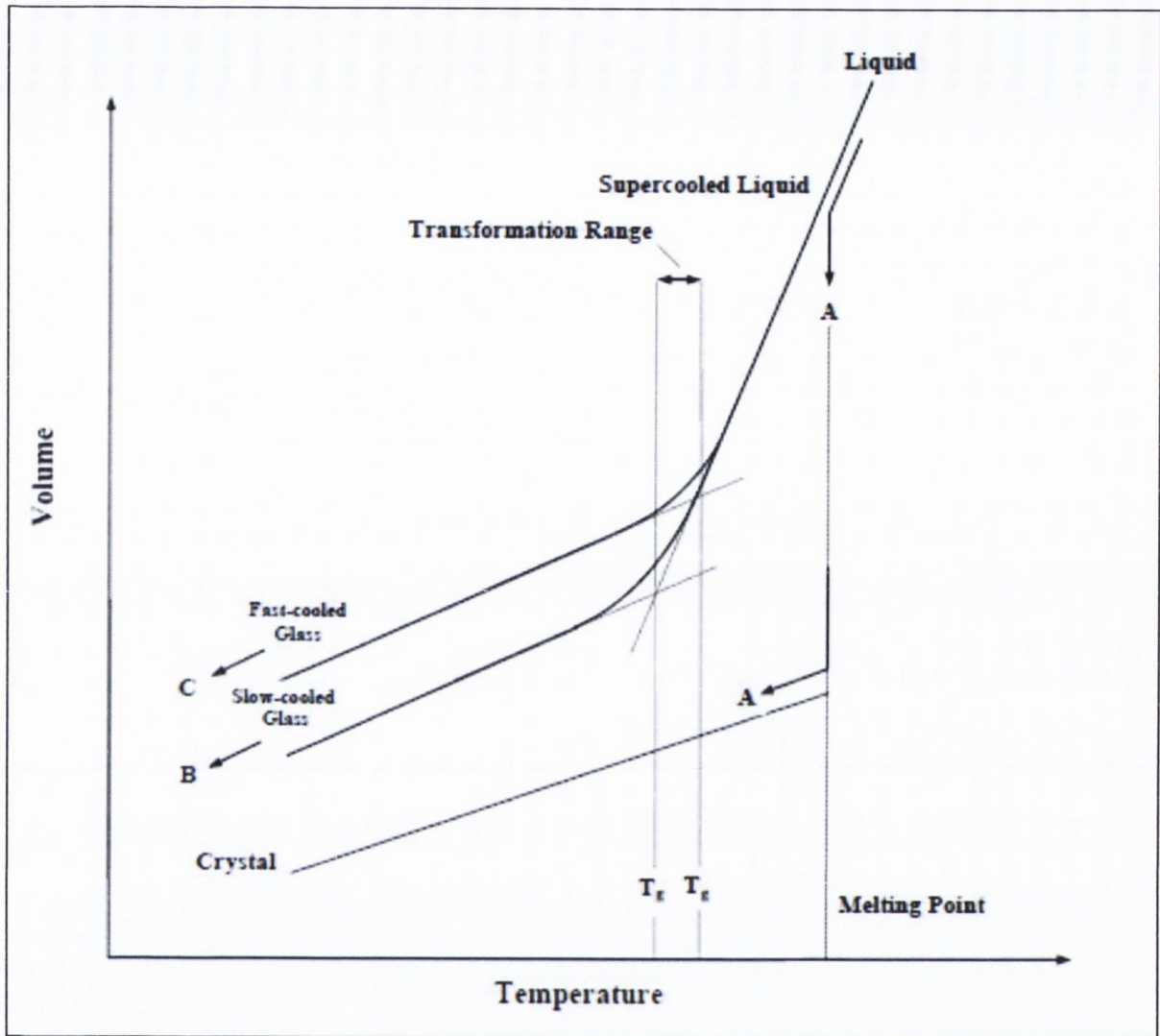
McLean and Hughes (1965) reported the biaxial flexure strength (BFS) of 'pure' low fusing dental porcelain disc-shaped specimens (n=6) to be 66 MPa with a reported minimum value of 57 MPa and a maximum value of 78 MPa. BFS testing of disc-shaped feldspathic porcelain specimens (Vitadur Alpha, Vita Zahnfabrik, Bad Säckingen, Germany) dispersion strengthened with up to 10% alumina was variously reported to be 72 (12) MPa (n=40) (Isgró et al., 2011) and 94 (10) MPa (n=30) (Addison et al., 2007a). Vitadur-N aluminous core porcelain (Vita Zahnfabrik, Bad Säckingen, Germany) is a dispersion strengthened aluminous core porcelain with up to 40 wt.% alumina. The mean BFS of Vitadur-N has been reported by Hornberger (1995) to be 134 (15) MPa which was similar to the 133 (6) MPa reported by Marquis and Fisher (1993).

The susceptibility of dental porcelain to fracture clinically under relatively low service loads continues to be the predominant criticism of the dental profession. In order to strengthen dental porcelain it could also be supported by a high strength ceramic, in a similar manner to PFM restorations, to form a hybrid layered ceramic structure (Jones, 1983). As a result, there has been a progressive commercial bias to further the hybrid layered (bilayered) concept by developing functionally higher strength core ceramic materials which are then veneered with a low strength aesthetic feldspathic porcelain. In attempting to enhance all-ceramic performance utilising the hybrid layered approach, the combined thickness needed for the high strength ceramic core layer fused to the veneering porcelain layer requires a more aggressive tooth preparation to avoid unaesthetic and over-contoured restorations. The dental profession has seen significant progress in the evolution of high strength core restorative materials that employ alumina or yttria-stabilised tetragonal polycrystalline zirconia (Y-TZP).

Alternatively, in contrast to the bilayered concept approach, a monolithic porcelain can itself be strengthened (Jones, 1983), whereby the structure continues to provide optimal aesthetics with translucency similar to enamel and is more biologically conservative because the tooth preparation required involves a minimally invasive approach. In addition, it has been reported that increasing the flexural modulus of a dental ceramic provides an increase in strength because dental ceramics generally fail at a critical strain of approximately 0.1% (Jones, 1983). There is also significant long term clinical evidence to support the theory that monolithic glass ceramic restorations perform remarkably well, particularly when acid-etched and luted with resin-based composite (RBC) materials (Malament and Socransky, 2001, 1999a,b). Therefore the monolithic restorative concept remains commercially viable. Minimally invasive treatment modalities with an optimum clinical outcome define the standard of care which should be aspired to in a patient-centred approach to dental care. The patient benefits to be gained with monolithic restorations are significant, namely, the conservation of tooth structure through a minimally invasive approach combined with optimal aesthetics which can be supported by long term clinical evidence (Malament and Socransky, 1999a,b, 2001). To enable the author to potentially improve the clinical longevity of monolithic restorations it is necessary to appreciate the developments in dental porcelain fabrication processes from their beginning. Furthermore, since glass is a significant component of dental porcelain, an understanding of the nature of glass itself is essential if an understanding of the formulation of dental porcelains is to be achieved (McLean, 1979a).

1.2 WHAT IS GLASS?

The structure of most substances in the molten or liquid state is disarranged with the atoms in continuous motion. If cooling occurs at a precisely defined melting temperature, namely the melting point, it results in the formation of a structure where the atoms are arranged into a regular network (Kingery et al., 1976b). Glass is traditionally formed by rapid cooling from a melt and has been commonly described as a supercooled liquid in terms of its physical chemistry, with the mechanical characteristics of a solid, based on Hookean elasticity theory. Rapid cooling of the glass melt at a predefined melting temperature increases the viscosity resulting in a kinetic barrier such that the atomic structure remains discontinuous and lacks any long range periodicity, with a gradual reduction in enthalpy. However, controlled cooling at or below the crystal melting temperature ensures a slower rate of change of viscosity which permits atomic restructuring and results in crystallisation as the enthalpy reduces abruptly (Kingery et al., 1976b). The viscosity of the melt at the crystallisation temperature is high, however, if cooling is sufficiently rapid, the atoms are unable to arrange themselves into a crystalline network and a supercooled liquid is produced (Kingery et al., 1976b). The temperature at which the glassy liquid phase changes to the supercooled liquid phase is called the glass transition temperature and glass is described as a meta-stable amorphous material (Figure 1.2).



crystalline silica (Figure 1.3a) and silica-based glass (Figure 1.3b). The ions in the silica-based glass are arranged with short-range regularity and the lack of long-range order results in increased enthalpy when compared with the corresponding crystalline phase. A glass is therefore a state of matter and may be defined as an amorphous solid without any long range structural periodicity, which therefore demonstrates time-dependent glass transformation behaviour (Shelby, 2005).

It was the seminal work on the atomic structure of glass by Zachariasen (1932) that provided a defining framework for glasses and has evolved into what has become known as the random network model. The model random network model describes any network structure in general and specifically glass in terms of four rules which describe a three-dimensional matrix lacking symmetry, periodicity, and regular unit repetition. For oxide glass formation to occur, the model advocates that: each oxygen ion should not be linked to more than two multivalent cations, the number of oxygen ions surrounding the central cation must be four or less, and the oxygen polyhedra must share corners, not edges or faces (so that in a three dimensional network), with at least three corners in each oxygen polyhedron shared. The cation component in the glass structure is described as a network former and the network structure is modified by the addition of randomly distributed alkali oxides which provide additional oxygen ions (Figure 1.3c). Connectivity within the network is determined by the bridging oxygens (Figure 1.3d) which link the Si-O tetrahedra and non-bridging oxygens which do not. In the case of soda lime silicate glass, one of the most industrially important glasses, the silicon cation functions as the network former with a typical oxygen to network former ratio of 2.4 (Kingery et al., 1976a), while the alkali oxides of sodium, or soda (Na_2O) and calcium (CaO), serve as the network modifiers. The alkali oxides disrupt and reduce network connectivity by adding more non-bridging oxygens and reducing bridging oxygens,

respectively. In practice, the Na_2O acts as a flux to reduce the melting temperature and the CaO confers stability to the material once formed.

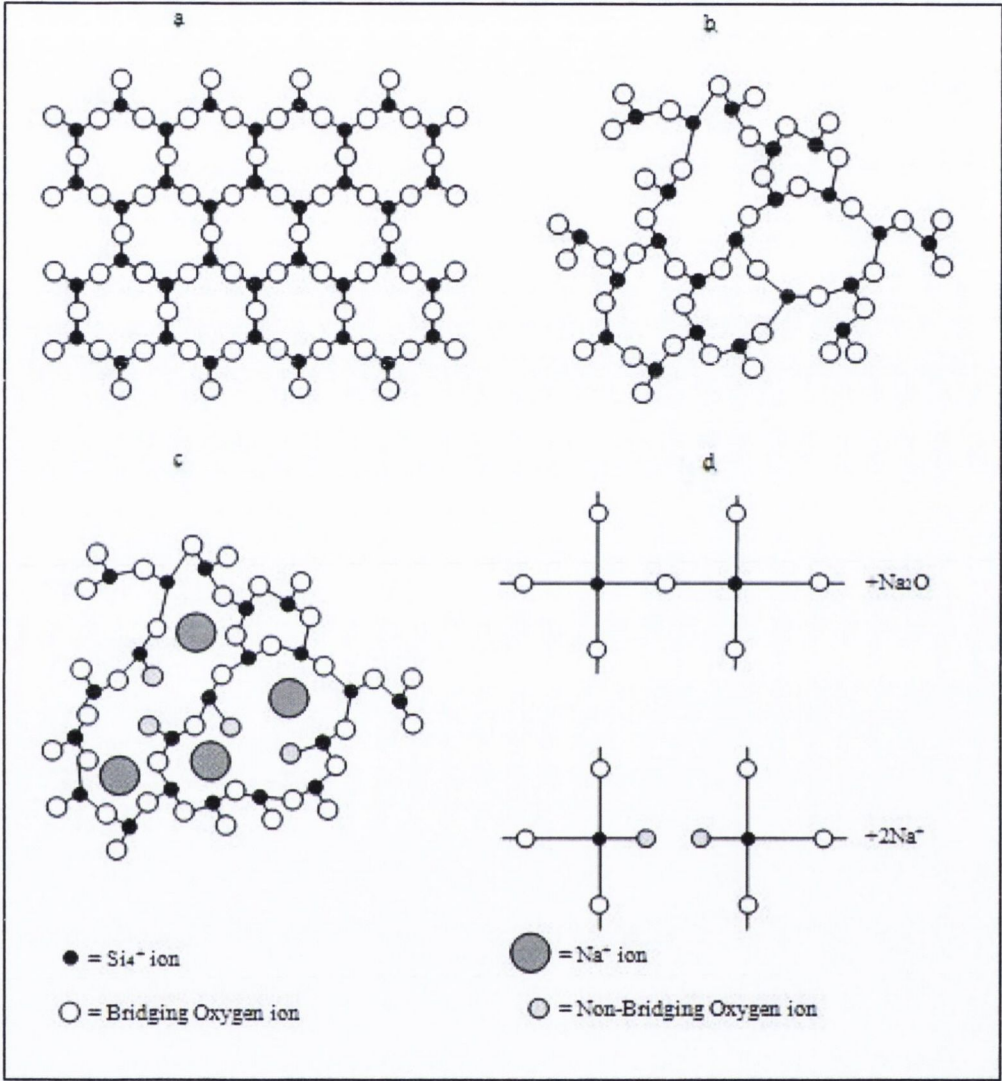


Figure 1.3: A two-dimensional schematic representation of silica glass in (a) the crystalline form and (b) the glassy form illustrating the basic SiO_4 tetrahedron structure of each form. The addition of Na^{+} ions into the silica network leads to the formation of a sodium silicate glass so that instead of a single bridging oxygen ion as before there are now two non-bridging oxygens. Adapted from McMillan (1964).

Strength of glass

The resistance of glass to fracture is characterised by brittle behaviour where no plastic deformation occurs prior to fracture. The actual glass strength is predominantly influenced by both surface condition and environmental factors, but is also dependent on the strength testing methodology employed. The actual glass strength is usually determined to be orders of magnitude less than the theoretical bulk strength and the difference has been explained by the presence of surface defects. These defects are susceptible to concentrated tensile stresses, where crack initiation and propagation results when the energy associated with crack extension exceeds the surface energy associated with the formation of new surfaces (Griffith, 1921). Previously, Ingles (1913) described crack behaviour in terms of stress at the crack tip which propagates once it exceeds the theoretical strength. A minimum radius of curvature of the crack tip was identified by Orowan (1934) which accounts for the geometric contribution in terms of the interatomic distance. The observed scatter in the experimental strength data of brittle materials is largely dependent on the presence of a critical flaw when exposed to an applied tensile stress (Kingery et al., 1976). Therefore the distributed nature of surface flaws and crack tip fracture behaviour explains why the strength of ceramics can never be reliably determined from 'average' fracture strengths (McLean and Kedge, 1987).

The dependence of brittle fracture strength on the surface defect configuration and their distribution implies that the surface area and volume of the specimen are also influential when considering the domain over which the surface flaw population exists. Various statistical methods have been developed to characterise the fracture strength of brittle materials based on the assumption that the quantity of defects is associated with the specimen surface area or volume (Ritter, 1995a,b). The most common statistical theory employed is the Weibull distribution (Weibull, 1951) which determines the probability with which failure

occurs in a material at a specified stress for an applied load. The Weibull modulus is the distribution parameter which indicates the flaw severity and distribution, where a low Weibull modulus indicates a highly variable defect population and a high Weibull modulus points towards a more uniform or narrow defect distribution. Furthermore, the probability with which failure occurs in a glass at a particular level of applied load can be employed as a design parameter for brittle materials by determining a characteristic strength described as the stress at which the probability of failure is 63% (Weibull, 1951).

The influence of moisture

Glass is a significant component of dental porcelain and the outer surfaces of restorations are readily exposed to water from saliva. Water is known to compromise the strength of silicate glasses and oxide ceramics due to static fatigue effects (Kingery, 1976) and has been demonstrated as being the 'primary rate-dependent mechanism' in the development of cone cracks in monolithic ceramic dental crowns with a significant glass phase (Lee et al., 2002).

The rate of moisture assisted crack growth in soda lime glass bend specimens has been demonstrated to depend on the rate of reaction and external exposure to moisture at the crack tip (Charles, 1958a,b,c). Ionic exchange between the H^+ ions in solution and the mobile alkali (M^+) ions in the glass produces OH^- anions at the crack-tip and exposes the Si-O-Si bonds at the crack tip to attack from external moisture (Douglas & Isard, 1949; Budd, 1962). The external moisture breaks the Si-O-Si bonds at the crack tip to form a hydroxide which further increases corrosion rate and specific volume of the crack tip leading to a more open structure, compared with an annealed glass with a denser structure of similar composition. Wiederhorn (1967) reported the process by demonstrating that the crack growth velocity was reaction rate controlled where an increase in the availability of external water vapour enhanced static

fatigue failure under an applied load. In a study of crack growth rate as a function of glass composition, it was determined that sodium was deleterious to the corrosion resistance of glass (Wiederhorn and Bolz, 1970), which lends further support for soda lime silicates as an eminently suitable material to investigate the role of moisture-assisted slow crack growth in a rate dependent setting.

Ambient water vapour may be present during glass formation leading to the inclusion of hydroxyl bonds or less commonly, distributed molecular water within the bulk glass but is more commonly encountered during subsequent environmental exposure to moisture (Shelby, 2005). The external surfaces of a glass exposed to tensile stress in the presence of moisture accelerate flaw growth until the conditions within the defect result in failure by catastrophic fracture consistent with the Griffiths criterion. Studies on the effect of static fatigue on the loading of dental porcelain rods to fracture in ambient air for times from 0.01 to 86400 s (24 h) identified that dental porcelain was susceptible to static fatigue failure - the ratio of the average breaking stress from testing at 0.01 compared with 86400 s, varied from 1.73-1.85 for the three different porcelain types investigated (Baker and Preston 1946a,b).

Whilst there is general agreement in the ceramic, engineering and dental literature that slow crack growth reduces the strength of glasses (Charles, 1958a,b,c; Wiederhorn, 1967; Cesar et al., 2008; Pinto et al., 2008; Gonzaga et al., 2011), there is much confusion as to who was the author(s) that first reported the discovery. Much of the dental publications attribute slow crack growth in glasses to Sheldon Wiederhorn in 1967 (560 citations) but this article was published almost a decade after the three Charles publications of 1958 (339,^a 130^b and 148^c citations, respectively). Wiederhorn and Bolz in 1970 suggested Louis Geret observed a loading rate dependence on glass strength in 1899 although it was not until 1929 that Lowell

Milligan published an article demonstrating the slow crack growth process in glass. Despite these early observations, Baker and Preston (1946a,b) reported dental porcelain suffered from slow crack growth which was hardly surprising since dental porcelain consists largely of a continuous glass matrix. What is surprising, however, was that the two papers by Baker and Preston (1946a,b), although cited 71^a and 45^b times, respectively in the ceramic and engineering literature, have not been cited at all in the dental literature, notwithstanding the subject matter. Instead researchers in dentistry appear to favour attributing slow crack growth in to Sheldon Wiederhorn in 1967.

1.3 DEVELOPMENT OF DENTAL PORCELAIN

Dental porcelains use the basic Si-O network as the glass forming matrix (McLean, 1979). Additional properties including aesthetics, thermal expansion coefficient, fusion temperature and viscosity (resistance to pyroplastic flow) have to be incorporated into the glass utilising additives (McLean, 1979a). Dental porcelains are formed into simple shapes which require high translucency so that high-feldspar/low-clay compositions are most effective (Kingery et al., 1976c). The routine dental porcelain composition is made up of a minimum of 60 wt.% SiO₂ and the remaining oxides are carefully controlled so that properties required for clinical dentistry such as aesthetics, fusing temperature and viscosity are readily achieved. Feldspar is an anhydrous aluminosilicate containing K⁺ (potash feldspar, K(AlSi₃)O₈), Na⁺ (soda feldspar, Na(AlSi₃)O₈) or Ca²⁺ (lime feldspar, Ca(Al₂Si₂)O₈) cations which serve as a flux to aid the formation of the glass phase (McLean, 1979). The function of a flux is to reduce the softening temperature of the glass by reducing the crosslinking between the oxygen and silicon and the formation of non-bridging oxygens (Figures 1.3c and 1.3d). In the fluxing process, the addition of alkali ions (Na⁺, Ca²⁺ and K⁺) into the random silica network

produces structural changes, namely, the formation of non-bridging oxygens and the addition of Na^+ ions leads to the formation of a sodium silicate glass. By adding an intermediate oxide (such as Al_2O_3), the SiO_4 tetrahedra are replaced by AlO_4 tetrahedra which is the structure commonly identified in aluminosilicates such as feldspars. The resultant glass has a higher viscosity and therefore the porcelain has an increased resistance to slumping (pyroplastic flow) preventing a loss of contour during firing (McLean, 1979). Small additions (<12 wt.%) of boric acid (B_2O_3) to aluminosilicates such as feldspars can also result in a porcelain with a lower thermal expansion coefficient and a more stable structure through the introduction of BO_4 tetrahedra into the glass (McLean, 1979).

Traditional dental porcelains have been classified according to the fusing temperature (Jones, 1985) or the translucency appropriate to the clinical indication. The fusing temperature may be low (850-1060°C), medium (1090-1260°C) or high (1315-1380°C) while the translucency can be described as opaque, core, body dentine or enamel. Opaque porcelain is used in PFM systems to mask the effect of the underlying metal and should have a thermal expansion coefficient similar to the metal so that tensile stresses are not induced in the porcelain during cooling (McLean, 1979a,b). Core porcelain is reinforced with alumina (Al_2O_3), however, translucency is lost because of the high (40 wt.%) Al_2O_3 content (McLean and Hughes, 1965). Body dentine and enamel porcelains are similar to core porcelain but the alumina content is reduced to around 15 and 5 wt.%, respectively, to achieve a semi-translucent effect (McLean and Hughes, 1965).

Marquis and Fisher (1993) identified that surface flaws introduced during fabrication were strength limiting and further refinement in operator technique to improve surface quality of the ceramic by employing platinum foil burnishing and pre-heating led to an increase in mean

BFS. In addition, Fleming et al. (1999) demonstrated that the reliability of Vitadur-N aluminous core porcelain disc-shaped specimens was significantly influenced by the porcelain powder to modelling liquid mixing ratio. In a dental laboratory setting, a dental technicians preferred slurry consistency is typically dictated by a subjective porcelain powder to modelling liquid ratio routinely employed during condensation of the porcelain restoration. An optimum mixing ratio of 1.4 g of Vitadur-335N aluminous core porcelain powder with 0.4 g of modelling liquid was identified, which resulted in a significant increase in reliability (Weibull modulus) for the biaxial flexural strength testing of 30 disc-shaped specimens ($m=12.3$ (2.2)) when compared with a porcelain powder content of 1.2 g ($m=9.7$ (1.8)) which was 'too fluid' (Fleming et al., 1999), or 1.6 g ($m=11.7$ (2.1)) which was 'too clumpy' (Fleming et al., 1999). Interestingly, there was no significant statistical difference ($p<0.01$) in the mean biaxial flexure strengths reported of 145 (17) MPa, 155 (14) MPa and 142 (14) MPa, for the powder contents of 1.2, 1.4 and 1.6 g, respectively (Fleming et al., 1999). Similarly, for disc-shaped specimens fabricated with a dentine porcelain (Vitadur Alpha, Vita Zahnfabrik, Bad Säckingen, Germany) an optimum mixing ratio of 0.91 g porcelain powder to 0.33 ml of modelling liquid was identified. The disc-shaped specimens prepared with the optimum mixing ratio were observed to have an increased mean biaxial flexure strength of 87.3 (11.9) MPa and Weibull modulus of 8.0 (1.5) with minimum porosity when compared with the specimens fabricated with 0.82 g or 1.0 g porcelain powder to 0.33 ml of modelling liquid (Fleming et al., 2000). The importance of employing an optimum porcelain powder to modelling liquid ratio was further emphasised when considering the influence of the defect population (Bhamra et al., 2002) and pre-cementation surface treatments including alumina particle air abrasion (Fleming et al., 2004) or hydrofluoric acid-etching (Fleming et al., 2006) on the observed biaxial flexure strength of aluminous core disc-shaped specimens.

The main clinical indications of PLV restorations are to disguise labial staining, correct form discrepancies or minor misalignment. The conservative labial tooth reduction of 0.5 mm (Rosenstiel et al., 2006) permits sufficient space to contour the PLV which is adhesively bonded to the prepared tooth with a RBC luting material. A variation of the PLV technique is the dentine bonded crown (DBC) (Sutton and McCord, 2001) originally conceptualised by Resinkor (1987) and involved full or partial crown coverage with the biological benefit of minimal tooth reduction. Several studies have reported the favourable clinical performance of PLVs manufactured from feldspathic porcelain (Shaini et al., 1997; Dumfahrt and Schaffer, 2000; Burke, 2012), yet manufacturers and researchers alike have focused on castable glass ceramics and pressable ceramics as possible candidate materials for PLVs and DBCs.

Dicor was a castable fluorine-containing tetra silicic glass-ceramic developed in 1978 by Adair and Grossman (1982) which was introduced commercially in 1982 (Dentsply/York Division Lab Products, York, PA, USA). The Dicor restoration was formed by a 'ceraming' process which involved heating the glass to initiate seeding and subsequent crystal nucleation (MacCulloch, 1968). The main components of the system were oxides of SiO_2 , K_2O and MgO along with MgF_2 and minor additions of Al_2O_3 , ZrO_2 to increase restoration durability (Grossman, 1972; Adair, 1984). The fluoride was essential to the process as it acted as a nucleating agent for devitrification to facilitate the transformation to a semi-crystalline state and also helped to provide sufficient fluidity when the ingot melted to cast the monolithic restoration. Although discontinued, the clinical success of Dicor was remarkable. In a clinical study of 1444 Dicor restorations over 16 years, Malament and Socransky (2001, 1999a,b) estimated an overall annual failure rate of 13% although that failure rate was significantly influenced by surface condition and luting material. Surface pre-treatment of Dicor restorations with hydrofluoric acid prior to luting resulted in an estimated annual risk of

failure of circa 5% for restorations luted with zinc phosphate (n=25), circa 3% for restorations luted with glass ionomer cement (n=192) and circa 2% for restorations luted with an RBC material (n=1066). It is important to note that there were no significant differences in the estimated annual risk of failure between restorations luted with RBC, glass ionomer or zinc phosphate, for non-acid-etched intaglio surfaces of the Dicor restorations in the study (Malament and Socransky, 2001). This result led Kelly (2012) to interpret the 'key' clinical finding as a roughened intaglio surface from acid-etching was essential in obtaining reinforcement from the RBC luting materials. However, the lustre of Dicor restorations was problematic resulting in the material being discontinued despite the remarkable performance of acid-etched resin-luted restorations.

1.4 HIGH STRENGTH DENTAL CERAMICS

The IPS Empress system (Ivoclar, Schaan, Liechtenstein) was a dispersion strengthened glass ceramic designed for injection moulding, or pressing, which was pre-cerammed feldspathic porcelain reinforced with leucite crystals (40-50 wt.%) (Gorman et al., 2000). The IPS Empress restoration was injection moulded directly to full contour prior to glazing and surface characterisation, providing aesthetic restorations with a translucency particularly suited to the anterior region of the mouth (Sorenson et al., 1998). Disadvantages with the IPS Empress included moderate flexural strengths and fracture resistance which contraindicated its use in the posterior region of the mouth (Heintze and Rousson, 2010). The mean BFS of IPS Empress has been variously reported for disc-shaped specimens to be 120 (21) MPa (n=21) (Cattell et al., 2002), 134 (12) MPa (n=6) (Gorman et al., 2000) and 175 (32) MPa (n=20) (Albakry et al., 2003). Further increasing the leucite crystal content to improve the flexural strength was limited by compromising the aesthetics which resulted in clinically

unacceptable opaque restorations. IPS Empress was superseded by IPS Empress 2 (Ivoclar, Schaan, Liechtenstein) which incorporated up to 60% lithium disilicate crystals to strengthen the feldspathic porcelain matrix. Since the refractive index of the crystalline phase was similar to the glass matrix, translucency was controllable even with the increased crystalline content. Albakry et al. (2003) and Chen et al. (2008) reported the mean BFS for IPS Empress 2 disc-shaped specimens as 407 (45) MPa (n=20) and 355 (26) MPa (n=8), however, Cattell et al. (2002) reported a much reduced BFS of 266 (26) MPa (n=20). The current IPS e.max Press system incorporates up to 70% lithium disilicate crystals with a reported BFS for disc-shaped specimens (n=20) of 440 (45) MPa (Albakry et al., 2003).

Despite the inherent advantages of a reduction in sound tooth structure lost to the bur during tooth preparation and the remarkable aesthetics of monolayer restorations, manufacturers and materials scientists alike have been determined to pursue hybrid layered (bilayered) restorations for DBCs and FPDs. The pursuit of the bilayered approach has been fruitful in producing higher moduli core materials or frameworks that do not fracture, however, the result is at the expense of sound tooth structure. Some of the major advancements in the field of high strength core materials for use as substructures which are then veneered with the lower flexural modulus and lower strength aesthetic porcelains are outlined below.

Injection moulding of a high strength core material contoured by the lost wax technique was reported by Sozio and Riley in 1984. Cerestore (Coors Biomedical, Lakewood, CO, USA) was marketed as a non-shrink alumina-based castable ceramic which was reported to overcome some of the problems associated with shrinkage on sintering (Sozio and Riley, 1984) since the MgO_2 and Al_2O_3 , when sintered at 1370°C formed a spinel phase (MgAl_2O_4) which occupied a larger volume than the constituent oxides. The core was then layered to a

full contour conventionally with the lower flexural modulus and lower strength aesthetic feldspathic veneering porcelain. Although scientifically clever in terms of the non-shrink aspect, the clinical success of the system was compromised by increased levels of porosity which reduced the structural properties of the core (Marquis and Wilson, 1986). In addition, the porosity was most noticeable at the inner surface of the crown where the pores acted as crack initiation sites (Kelly et al., 1996) which resulted in an increase in the clinical failure rate (Anusavice and Hojjatie, 1992) compared with aluminous core porcelain crowns despite the increased modulus and strength of the core material.

Further scientific advances were made with the introduction of In-Ceram (Vita Zahnfabrik, Bad Säckingen, Germany) which was a novel interpenetrating phase composite (IPC) material (Hornberger, 1995). In-Ceram consisted of a porous alumina substructure which was subsequently infiltrated and densified with a lanthanum glass to produce the IPC. The production route, however, was expensive in terms of the time taken to produce the dense IPC substructure and the technique was notoriously operator sensitive. Unpublished data by Hornberger showed that for disc-shaped specimens tested in biaxial flexure the mean strengths of the discs increased with operator experience. Hornberger consistently produced discs with a mean BFS of 310 (30) MPa in the first 12 months of her studies, this increased to 470 (48) MPa during the second year and in her final year of her PhD she could consistently produce samples with a mean BFS of 680 (67) MPa (Hornberger, 1995). As a result, the BFS values reported in the literature should be viewed with extreme caution. In addition, the clinical performance was compromised by the increased time required by the dental technician to master the technique notwithstanding the poor uptake of the material by practitioners owing to the increased associated cost of the crowns.

It was inevitable that the high purity alumina investigated by McLean and Hughes (1965) would be further investigated as a potential core material and was subsequently marketed as Procera Allceram (Nobel Biocare, Göteborg, Sweden). Interestingly the company had to wait until McLeans original patent expired before the material was marketed to the dental profession. Procera incorporates the high strength core, composed of 99.9% aluminum oxide, which was subsequently veneered with aesthetic porcelain. The mean BFS of disc-shaped specimens (n=10) fabricated with polycrystalline high alumina content Procera AllCeram was determined to be 687 (58) MPa (Wagner and Chu, 1996), while Itinoche et al. (2006) reported a BFS of 647 (48) MPa for Procera AllCeram disc-shaped specimens (n=10).

Zirconia which was trialled by Binns (1962) as a crystal phase to dispersion strengthen feldspathic porcelain, has also been considered as a core material for use beneath an aesthetic veneering porcelain. Zirconia is a high strength polycrystalline ceramic that is polymorphic and ordinarily exists below 2370°C in the tetragonal phase. A tetragonal-to-monoclinic phase transformation occurs below 1170°C and is accompanied by a 3-5% volumetric expansion which causes high internal stresses (Kosmac et al., 1999). Yttrium oxide (Y_2O_3) is added to partially stabilise the zirconia in the tetragonal phase at room temperature by controlling volumetric expansion (Garvie and Nicholson, 1972), forming what is known as yttria-stabilised tetragonal polycrystalline zirconia (Y-TZP). In a process called transformation toughening, tensile stresses that occur within the crack tips effect a local phase transformation from martensitic tetragonal to monoclinic with a respective increase in volume resulting in a local compressive stress due to dilatational strains within the crack tip to retard crack propagation by reducing the crack tip stress intensity (Heuer et al., 1986). The highly crystalline nature of zirconia gives an unacceptable opaque appearance which contraindicates its application as a monolithic restoration, but the material is typically indicated to function as

a core substructure with additional layers of translucent veneering porcelain applied to improve the aesthetics. Curtis et al. (2006a,b) reported a mean BFS of 1267 (161) MPa for Y-TZP (Lava, 3M ESPE, St. Paul, MN, USA) disc-shaped specimens (n=30), while Pittayachawan et al. (2007) reported a mean BFS of 1152 (138) MPa for the same Y-TZP disc-shaped specimens (n=30). Although the zirconia core offers a higher flexural modulus (234 GPa) which is comparable with metals, the full-contour bilayered crown structure is susceptible to porcelain veneer chipping fracture (Al-Amleh et al., 2010; Cehreli et al., 2009; Ortorp et al., 2009; Groten and Huttig, 2010).

1.5 MECHANICAL PROPERTIES OF DENTAL CERAMICS

The mechanical properties of dental ceramics are variously reported in the literature and the reasons for the inconsistencies are explained largely by the dominant brittle nature of ceramic materials. In addition, the range of test methodologies employed has been demonstrated to provide significantly different strength values (Jin et al., 2004). Uniaxial flexure testing is sensitive to edge effects normally present in the ceramic samples and the loading during testing is not uniform when three or four point flexural loading is employed. The BFS test (Ban and Anusavice, 1990; Ban et al., 1992), where a disc-shaped specimen is loaded centrally so that the maximum tensile stresses occur at the centre of the specimen and decrease rapidly with increased radial distance from the centre of the disc (Ban et al., 1992) offers a convenient alternative to three or four point flexural loading. The BFS technique eliminates spurious edge failures, offers controllable specimen geometry, simple sample preparation and specimens which have a surface to volume ratio more closely related to that of actual crowns than conventional three or four point flexural samples (Piddock et al., 1987). Several variations of the BFS test method exist (Ritter et al., 1980; Shetty et al., 1980),

however, the ball-on-ring or shell test was advocated by de With and Wagemans (1989) as being the most reliable BFS test method. More importantly, the BFS test induces fracture patterns which reflect clinical failure modes evident in retrieved fracture fragments of failed ceramic restorations (Kelly, 1999), namely, tensile failure from the cementation surface. As a result, the flexure strength results reported in the current review are limited to BFS for the reasons outlined above.

The difficulty in predetermining the size, location and distribution of the most critical defects in nominally identical ceramic specimens (Davies, 1973; Stanley et al., 1973; Evans, 1982; Ritter, 1995a,b) emphasised that the 'average' fracture stress that traditionally had been taken as the strength of a material could not easily be used as a design parameter for ceramic materials (Stanley et al., 1973; Creyke et al., 1979; Ibrahim & McCabe, 1993). Methods such as Weibull statistics (Weibull, 1951) assess the probability at which failure occurs in a material for a particular applied load and can be employed as a design parameter for structures made with brittle materials including dental porcelain restorations. From a general design perspective, the distributed nature of the strength data for ceramics dictates that a higher factor of safety should be adopted in selecting a material for service. Furthermore, since the mean fracture strength data for ceramic materials has been observed with standard deviations up to 25% (Ritter, 1995a,b), fabricating nominally identical specimens with a minimal fault population is highly important when examining determinants which modify strength.

Whilst dental ceramics have been continuously evolving since the development of the aluminous core porcelain by McLean and Hughes (1965), several highly innovative and technologically advanced high flexural moduli substrates have been developed in the past 20

years although the handling and performance characteristics vary between materials. It can be difficult to predict the relative success of each system when used clinically owing to the fact that the high flexural moduli substrate materials have to be veneered with an aesthetic porcelain. The rapid commercial evolution in dental ceramics presents a practical challenge to both materials scientists and general dental practitioners alike in estimating the clinical efficacy and reliability through independent laboratory and pre-clinical investigations. The challenge is further complicated by the fact that dental ceramics are brittle and the strengths are variously reported in the literature with the variation largely attributed to differences in testing conditions such as testing configuration, loading rate, specimen preparation techniques and specimen geometry (Hornberger, 1995).

Various test methodologies have been adopted to investigate the performance of hybrid layered dental ceramics, such as the crunch-the-crown, uniaxial three-point flexure and biaxial flexure testing protocols. The clinical relevance of crunch-the-crown test methodologies has been challenged (Kelly, 1999; Isgró et al., 2011; Kelly et al., 2012). The crunch-the-crown test produces multiple fracture fragments and the failure origin is induced on the external surface of the restoration due to the contact stresses. Therefore, the failure mode and fracture origin do not replicate the failure pattern evident in the clinical situation where fracture typically initiates on the intaglio ceramic surface in tension (Anusavice and Hojjate, 1992) and generally results in two fracture fragments (Kelly et al., 2010). In terms of comparing the performance of crown specimens, the crunch-the-crown test only returns a failure load which is substantially less meaningful than failure stress and in no way accounts for the complex geometry and dimensions of the crown specimen (Kelly, 1999; Kelly et al., 2012). From an analytical perspective, the biaxial flexure strength test method involving multi-layered disc-shaped specimens loaded in a ball-on-ring configuration provides a

simplified analytical model which facilitates the determination of a clinically representative failure stress under static loading. Together with advanced closed-form analytical solutions (Hsueh, 2005) to quantify the biaxial flexure strength as a function of failure load, specimen geometry, material properties and test configuration, performance can be determined and compared in a way that is both experimentally robust and clinically meaningful.

1.6 CLINICAL PERFORMANCE OF DENTAL PORCELAIN

The clinical performance of dental porcelain is variable and influenced by patient and operator factors, in addition to the specific restorative material properties. Reported patient factors such as tooth position, tooth history and condition such as caries, periodontal disease, occlusal schema, treatment history (Malament and Socransky, 2010) and factors associated with the operator such as previous training, age and gender (Burke and Lucarotti, 2009) can have an impact on the associated survivability of dental ceramic restorations.

Cost effective and predictable dental treatment is central to the achievement of patient satisfaction regardless of whether the treatment is paid privately by the patient or provided by a third party similar to the General Dental Services (GDS) in England and Wales (Lucarotti et al., 2005a-c; Burke et al., 2005a,b). In attempting to understand the clinical performance of dental ceramics in the dental literature, it is important to remain aware of the limitations incurred by inconsistencies in the study methodologies, definitions of clinical success and clinical failure and the determinants of the perceived outcomes. Although there are several parameters by which the clinical performance of dental ceramics have been characterised, in terms of patient satisfaction with aesthetics, PLVs generally provide optimal results. PLVs require minimal intervention involving approximately 0.5-1.0 mm enamel reduction to

maintain the PLV thickness to within the restored anatomical contour of the tooth. The primary indication for PLVs is often to electively address the aesthetic concerns of the patient, such that it is important to identify the clinical performance of PLVs although the evidence on the longevity widely varies (Shetty et al., 2011). An estimated survival probability of 91% was reported in a 10 year study of 191 PLVs (Dumfahrt and Schaffer, 2000). Interestingly, when the PLVs were adhesively cemented with RBC luting agents and the tooth preparation was confined to enamel, only one PLV failure was observed (Dumfahrt and Schaffer, 2000). The promising observations from Dumfahrt and Schaffer (2000) are in contrast to the survival rate of 53% identified in a 10 year study of 2562 PLVs by Burke and Lucarotti (2009). The recently published studies of an age-stratified sample of GDS patients clustered by year of birth, were extensive in that they included over eighty thousand adult patients (Lucarotti et al., 2005a-c; Burke et al., 2005 a,b). The data consisted of items gleaned from the payment claims submitted by GDS dentists to the Dental Practice Board (DPB) in Eastbourne, Sussex, England from a representative sample of patients treated in the GDS of England and Wales between 1991 and 2001. The patients accounted for a total of 719,009 claims sent to the DPB within the timeframe investigated to determine the dental factors associated with the restoration, including restoration type and size of the cavity. Therefore, the poor survival rate of 53% could be an indication of how PLV restorations perform in 'real' clinical practice rather than in controlled patient group as is routinely reported in the dental literature when studies are conducted in a controlled environment such as a University Dental School or Hospital. This fact is further emphasised by studies where the 10 year success rate of 87 PLVs was reported to be 64% (Peumans et al., 2004) and the 6.5 year success rate of 372 PLVs was 67% (Shaini et al., 1997). The Shaini et al. (1997) study included PLV restorations fitted by students (n=235) and staff (n=137) with significantly increased PLV failures occurring amongst the student (n=91 (39%)) compared with the staff

cohort (n=31 (23%)). Therefore it is difficult to assess the actual performance of PLV restorations from the dental literature.

Dicor DBCs had an estimated 5 year cumulative survival rate of 87.5% (n=1066) when luted with RBC materials which equated to circa 2% annual failure rate for DBC restorations which was remarkable given that the annual failure rate for the higher strength systems is remarkably consistent at circa 3% (Burke et al., 2002) although this figure does not include chipping fractures (Anusavice, 2012). Anusavice (2012) appropriately highlighted the importance of standardising how technical complications such as chipping fracture are defined, particularly when evaluating meta-data included in systematic reviews which attempt to further our understanding of how all-ceramic restorations fail to perform. Additionally, the relevance of standardising definitions for clinical success and clinical failure and study design has been described by Anusavice (2012) where deficiencies in standardisation are particularly pronounced when attempting to compile a systematic review to analyse data. Regarding hybrid layered (bilayered) restorations, Pjetursson et al. (2007) in a systematic review of all-ceramic restorations fabricated from various ceramic materials identified that for all positions in the mouth, Empress - a leucite-reinforced glass ceramic, InCeram - an infiltrated glass ceramic and Procera - a densely sintered alumina, had estimated 5 year crown survival rates of 95.4% (n=1683), 94.5% (n=1915) and 96.4% (n=729), respectively. Additionally, Pjetursson et al. (2007) identified the 5 year survival rate of anterior ceramic crowns to be higher than posterior crowns with a clinical success rate of 91.4% compared with 84.4% for Empress (n=590; p=0.009) and 96.7% compared with 90.4% for InCeram crowns (n=533; p=0.028). Therefore, in line with the comments of Anusavice (2012) regarding the interpretation of data from systematic reviews, it can be challenging to understand the clinical performance of restorations without a detailed

classification system for identifying adverse clinical outcomes such as technical or biological failures. Adherence to a classification system alone is not sufficient and the details which indicate the causative nature of the reported failures are factors often omitted from the subsequent analysis. For example, the treatment and pathological history of a tooth which was subsequently restored with a hybrid layered crown or a FPD and selected for inclusion in a clinical investigation can have a prognostic influence on the clinical outcome of the restoration (Anusavice et al., 2007).

There also exists a significant potential for investigator bias in the over-reporting of successful treatment outcomes and the under-reporting of adverse events, particularly where studies have been sponsored directly or indirectly by industry (Anusavice, 2012). One could critique studies confined to an idealised and controlled study population in an academic context which may not accurately reflect the ‘real’ world of clinical practice (Balevi, 2011). However, nobody can account for investigator bias whereby there is no requirement for the treating dentist to be excluded as an evaluator (Anusavice, 2012) which is a variant of the ‘fox in the chicken coop’ scenario. Evidence to support this contentious eventuality was described in a systematic review of the dental implant literature conducted by Popelut et al. (2010) which identified the annual failure rate of dental implants reported by industry supported investigations to be significantly lower than studies not in receipt of industrial support.

Conformation to a prescribed investigative protocol leads to the potential for consistent and comparative analysis of restorative treatment modalities, however, nowhere is this more appropriate and unfortunately lacking, than in the area of hybrid layered zirconia crowns and FPDs. Adhesive bonding of zirconia is problematic since the Y-TZP ceramics used in

dentistry lack a significant glass phase which would allow for an acid-etching surface pretreatment prior to adhesive bonding. In addition, the performance of hybrid layered zirconia restorations are not comparable with metal-ceramic restorations as the failure of zirconia systems by chipping fracture of the veneering porcelain has been variously reported in the literature to occur in 8-50% of restorations with the onset of crazing or fracture and minor loss of material after 1-2 years observation (Von Steyern, 2005; Von Steyern et al., 2005; Larsson et al., 2006; Raigrodski et al., 2006).

In view of the clinical success of PLVs and DBCs manufactured from thin (0.4-1.0 mm), low strength materials and luted with RBC materials, notwithstanding the significant saving of sound tooth structure, it is the opinion of the author that improving the clinical performance of PLVs and DBCs through modification of the precementation operative variables would be more beneficial than employing or developing further high flexural moduli hybrid layered restorations that 'butcher' sound tooth structure. Evidence for this ethos can be seen in the clinical context of veneering porcelain fracture on high flexural moduli ceramic cores. It is remarkable that the short-term clinical data for full-contour monolithic lithium disilicate crowns is promising (Fasbinder et al., 2010; Reich et al., 2010) and more recently, in-vitro sliding contact fatigue testing on the occlusal surface of these crowns (n=18) determined that thin (<1 mm) monolithic lithium disilicate restorations resin luted to RBC dies had increased reliability compared with Y-TZP veneered hybrid layered restorations but comparable reliability with metal ceramic systems (Silva et al., 2012).

The superior aesthetic qualities of monolithic feldspathic porcelain type materials over the higher flexural moduli crystalline ceramic hybrid layered restorations currently available will also ensure their continued clinical use particularly in the anterior aesthetic region of the oral

environment as PLVs and posteriorly as DBCs. The relatively low strength of feldspathic porcelain can lead to premature clinical failure due to fracture which may manifest as unaesthetic fracture lines, stain acquisition, microleakage and ultimately partial or complete loss of the restoration (Kelly, 1999). However, the clinical advantage is that despite a partial or complete loss of the restoration the conservative preparation with minimal tooth reduction offers the operator the potential for re-intervention replacing like with like to achieve the optimum biological outcome. Due to the anatomical characteristics of dentine, deeper tooth preparation exposes wider dentinal tubules which are more permeable and therefore more susceptible to physiological insult (Pashley, 1990). The incidence of pulpal devitalisation due to crown preparation has been variously reported from 4-13% (Abou-Rass, 1982; Bergenholtz and Nyman, 1984; Felton et al., 1989; Reichen-Graden and Lang, 1989; Gonzalez et al., 1991). The potential clinical benefits of minimal reduction during tooth preparation are clear and conservative intervention with aesthetic feldspathic porcelain reinforced by and bonded to the tooth substrate with an adhesive RBC luting material is logically more preferable to an aggressive tooth reduction protocol as would be necessitated by high flexural moduli hybrid layered restorations.

Operative variables

The adhesive cementation of all-ceramic PLV or DBC restorations with RBC luting agents has been shown, in laboratory (Marquis, 1992; Rosenstiel et al., 1993; Pagniano et al., 2005; Fleming et al., 2006) and clinical (Malament and Socransky, 1999a,b) studies to improve performance. Burke et al. (2002) reported that improved retention was provided by RBC luting materials in mediating a reliable bond with the restoration and tooth. Several studies have reported increased fracture resistance of all-ceramic restorative materials adhesively cemented with an RBC (Bernal et al., 1993; Scherrer et al, 1994; Burke et al., 1999; Behr et

al., 2003) and a significant increase in flexure strength has been reported for ceramic specimens coated with an RBC (Rosenstiel, 1993; Addison et al., 2007d).

All-ceramic restorations are adhesively cemented in the clinical setting by applying a thin layer of RBC luting material to the intaglio surface of the restoration and seating the restoration on the supporting tooth. The determinants which can influence performance of the all-ceramic restoration have been reported to include the flexural modulus of the RBC luting material (Addison et al, 2008) and the precementation surface condition of the all-ceramic restoration (Addison et al., 2007a,b). In addition, the load at which the restoration is seated has been observed to modify the flexure strength of dental porcelain (Addison et al., 2010) although the influence of the resulting RBC film thickness in the context of RBC flexural modulus and seating load has not been reported.

The intaglio surface of an all-ceramic restoration is routinely modified to improve adhesion with the RBC luting material and optimise retention of the restoration (Lacy et al., 1988; Burke et al., 1998). The integrity of the bond between the dissimilar restorative materials is dependent on micromechanical interlocking and the chemical interaction of the RBC luting material with the ceramic substrate (Ozcan and Vallittu, 2003). Surface roughening of dental porcelain was originally proposed by Rochette (1975) as a technique to promote bonding. Prior to resin-cementation, an increase in the available ceramic surface area for enhanced adhesion may be achieved by creating surface asperities with interlocking micromechanical features by acid-etching (Calamia, 1985) or alumina particle air abrasion (Wolf et al., 1993). The development of silane technology to enhance the chemical interaction between dissimilar materials was pioneered commercially by Dow Corning over fifty years ago. Although the bonding mechanism of silane coupling agents is not fully understood, the application of a

silane coupling agent to a dental porcelain substrate was initially proposed to improve the chemical bond of resin-based composite employed for the intra-oral repair of chipped dental porcelain (Newburg and Pameijer, 1978). Ceramic surface modification influences the physical and chemical components which characterise adhesion with a resin-based composite luting material and the increase in surface roughness also increases the surface energy which promotes bonding due to increased wettability.

The surface treatment of glass with hydrofluoric (HF) acid is known to influence the actual strength observed by modifying the surface defect population (Proctor, 1962). Preferential surface dissolution by HF acid leads to the formation of hexafluorosilicic acid or tetrafluorosilicic acid, resulting in crack length reduction, crack tip blunting or ultimately complete removal of the defect population with an associated increase in actual strength (Dabbs and Lawn, 1983; Saha and Cooper, 1984; Sglavo et al., 1993). The application of 10% HF acid to the intaglio surface of feldspathic dental porcelain restorations was proposed to roughen the exposed surface (Horn, 1983) and the observed preferential dissolution of different porcelain phases results in a topography more favourable for bonding (Stangel et al., 1987). The susceptibility of a dental ceramic to HF acid-etching is predominantly dependent on the presence of a glass phase for dissolution since the crystalline component is resistant to degradation by HF acid (Della Bona and Anusavice, 2002). More recently the HF acid-etching of feldspathic dental porcelain has been reported to depend on substrate composition, surface topography, acid concentration and etching time (Addison et al., 2007b).

Alumina particle air abrasion is commonly employed to promote bonding by roughening the intaglio surface of an all-ceramic restoration. The technique employs Bernoulli's principal to invert pressurised air at low velocity through a small diameter nozzle at high velocity

entrained with aluminium oxide particles. The aluminium oxide particles which are suspended in the high velocity air stream and directed at the ceramic surface, impart a localised abrasive effect as the kinetic energy is resolved on contact with the substrate (Addison et al., 2007a). The alumina particle air abraded surface is characterised by an effective increase in surface area with undercut features which enhance micromechanical retention. The surface topography resulting from alumina particle air abrasion pre-treatment is dependent on the particle size, distance of delivery nozzle from surface, air stream delivery pressure and velocity including the direction and duration of particle delivery (Darvell, 2002; Addison et al., 2007a). As a safer alternative to acid-etching, the alumina particle air abrasion technique has been advocated to increase surface roughness (Wolf et al., 1993) although alumina particle air abrasion may also be combined with acid-etching to further promote bonding (Ozcan and Vallittu, 2003).

Silanes are silicon-based molecules with reactive organic and non-organic components which provide a coupling function to promote adhesion between different materials. The silane coupling agent acts as a primer to modify the inorganic substrate surface and enhance the chemical bond between the dissimilar materials. In dentistry, silane coupling agents are routinely employed to mediate chemical adhesion between the inorganic silica-based all-ceramic restoration and the organic resin-based composite luting material (Lung and Matinlinna, 2012). Without silane priming, the chemical interaction between the silica based all-ceramic restoration and the resin-based composite luting material is dominated by weak Van der Waal forces and hydrogen bonding. The hydrolysable inorganic functional alkoxy (methoxy- and ethoxy-) groups of the silane coupling agent react with the hydroxyl groups of inorganic silica-based ceramic substrate, while the organic functional groups (vinyl, amino) react with the methacrylate groups in the organic resin-based composite luting material

(Matinlinna et al., 2004). A silane coupling agent commonly utilised in the clinical setting is 3-Methacryloxypropyltrimethoxysilane (MPS), which is applied to the cementation surface of an all-ceramic restoration in an aqueous alcohol solution. The silane solution may be prehydrolysed in a single bottle with water and alcohol or as a two bottle system with the unhydrolysed silane in an alcohol solution and separate water-based acetic acid solution. The solutions in two bottle system are dosed and mixed to hydrolyse the silane immediately prior to surface treatment, overcoming the limited shelf life of the prehydrolysed single bottle system. Condensation of the silane coupling agent by drying leaves a layer of silane primer on the ceramic surface which is approximately 50-100 nm thick, prior to cementation with the resin-based composite luting material. The coupling functionality of the silane agent is theoretically achieved with a molecular monolayer on the ceramic surface but final thickness of the silane layer is influenced by the random molecular orientation (van Ooij and Sabata, 1992), activated silane concentration and drying regimen (Shen et al., 2004).

Resin-cementation

Commercially available RBC luting materials are routinely formulated to optimise operator handling for the intended clinical indications. A reduced viscosity RBC luting material enables the resin to flow during seating of the restoration which is convenient to the operator. However, in order to achieve the reduced viscosity, the filler content of the RBC luting material is reduced which results in a reduction in the flexural modulus of the lute. Although evidence indicates that the adhesive cementation of all-ceramic restorations improves performance in the clinical setting, the strengthening mechanism conferred by resin cementation is not fully understood (Addison et al., 2007d).

Various theories have been reported to describe the resin-strengthening mechanism, including crack closure stresses (Roach et al., 1988; Nathanson, 1994), full or partial crack-healing (Fabes and Uhlmann, 1990; Marquis, 1992), and Poisson constraint effects (Wang et al., 1995). None of the theories described fully explain the resin-strengthening mechanism because all theories are implicit in that the strengthening mechanism is dependent on the severity of the defects present on the ceramic surface. Fleming et al. (2006) challenged this view by demonstrating that the degree of resin-strengthening conferred on a Vitadur Alpha dental porcelain substrate was independent of defect severity suggesting the previously reported theories were incorrect. Addison et al. (2007c) identified that strengthening of resin-coated Vitadur Alpha dentin porcelain disc-shaped specimens was sensitive to the flexural modulus of the RBC luting agent and the author introduced the concept of a resin-ceramic hybrid layer. The existence of a resin-ceramic hybrid layer was validated by Addison et al. (2008) with scanning electron microscopy and by identifying the increase in resin-ceramic strengthening at a theoretical 'zero' resin-lute thickness. The pattern of strengthening observed in the feldspathic porcelain substrate was attributed to the combined influence of the RBC flexural modulus and Poisson constraint on the interaction within the resin-ceramic hybrid layer (Addison et al., 2008).

2. MATERIALS AND METHODS SUPPLEMENTAL MATERIAL

2.1 RAW MATERIALS

2.1.1 Dentine porcelain

The dentine porcelain investigated in the current study was Vitadur-Alpha dentine powder (Shade A2, Lot #62167) and Vitadur Modelling fluid (Lot #14209R) which were supplied by Vita Zahnfabrik H. Rauter GmbH & Co. KG, Bad Säckingen, Germany.

2.1.2 Soda lime glass analogue

The dentine porcelain analogue material selected for the current study was a soda lime glass. The transparent soda lime glass (Glass 8337B) was received in rod form (1.5 m length, 11.4 mm diameter) and supplied by SCHOTT-Rohrglas GmbH, Mitterteich, Germany. The curved soda lime glass disc-shaped specimens were fabricated from transparent flat soda lime glass stock supplied by AGC Glass Europe, Brussels, Belgium.

2.1.3 Alumina particles for air abrasion

Aluminium oxide with an average particle size of 50 μm (Metrodent, Huddersfield, UK) was employed in the current study to produce a surface which was conducive to the surface texture routinely observed on the fit surface of PLV and DBC restorations prior to cementation clinically. The dentine porcelain disc-shaped specimens were alumina particle air abraded with an Eco Dry Oxide System (Dentalarm, Torino, Italy) operating at a pressure of 4 bar. Alumina particle air abrasion was performed at a distance of 2 cm from the nozzle of the abrader to the fit surface of the dentine porcelain disc-shaped specimen. The alumina particle air abrasion technique was performed orthogonal to the fit surface of the dentine

porcelain disc-shaped specimen in a smooth linear sweeping motion for a maximum of 5 s to prevent excessive localized abrasion of the disc surface. The apparatus employed to alumina particle air abrade the soda lime glass disc-shaped specimens was a GOBI-2 (Wassermann Dental-Maschinen GmbH, Hamburg, Germany) in accordance with the protocol described for the dentine porcelain disc-shaped specimens. The alumina particle air abraded disc-shaped specimens were rinsed in distilled water for 10 s prior to drying completely with oil free compressed air. The disc-shaped specimens were stored in a desiccator at ambient temperature to prevent hydrolysis of the silicate bonds (Budd, 1961) until required for further testing and analysis.

2.1.4 Hydrofluoric acid etchant

The acid-etchant employed in this study to chemically roughen the specimen surface was 9.6% hydrofluoric (HF) acid (Porcelain Etch Gel, Pulpdent Corporation, Watertown, MA, USA). The HF acid-etchant was applied to the alumina particle air abraded surface of the disc-shaped specimen for 120 s and rinsed with distilled water for 10 s prior to drying with oil free compressed air. The disc-shaped specimens were then stored in a desiccator at ambient temperature until required for further testing and analysis.

2.1.5 Silane coupling agent

The silane coupling agent utilised in this study was a 3-methacryloxypropyl-trimethoxysilane based ceramic primer (Rely-X Ceramic Primer, 3M ESPE, St. Paul, MN, USA). Immediately prior to resin-cementation, the silane coupling agent was applied to the alumina particle air abraded and HF acid-etched surface of the disc-shaped specimen with an applicator brush and allowed to dry in ambient air.

2.1.6 Resin-based composite materials

The resin-based composite (RBC) materials employed in this study were selected to provide a range of flexural moduli and included a flowable RBC (Venus Flow) reported by the manufacturer (Heraeus, Hanau, Germany) to have a filler content of 60 wt.%, a luting RBC (Rely-X Veneer) with a reported filler content of 66 wt.% (3M ESPE, St. Paul, MN, USA) and two different hybrid RBCs. The first hybrid RBC employed (Clearfil AP-X) had a reported filler content of 82 wt.% (Kuraray, Kurashiki, Japan). The manufacturer discontinued Clearfil AP-X during the study and a replacement hybrid RBC (Clearfil Majesty Posterior) was selected with a reported filler content of 92 wt.% (Kuraray, Kurashiki, Japan).

The flexural moduli of each RBC material employed in the current investigation were established using a three-point flexure testing assembly (ISO 4049:2000). An open-ended aluminium split-mould (25 mm length, 2 mm width, 2 mm thickness) was used to fabricate nominally identical bar-shaped specimens for each RBC investigated (Bhamra et al., 2010). To ensure consistent specimen packing following resin placement, the aluminium split-mould was covered with a polyester film and a flat glass slide with a 1 kg weight was applied. The bar-shaped specimens were light irradiated at an output intensity of $380 \pm 21 \text{ mW/cm}^2$ with an Optilux 501 (SDS Kerr, Danbury, CT, USA) quartz tungsten halogen (QTH) light curing unit (LCU). The length of the bar-shaped specimens (25 mm) exceeded the 13 mm light tip diameter and as a result the bar-shaped specimens were irradiated at successive overlapping incremental sites along the length of the bar-shaped specimens (ISO 4049:2000). The light tip diameter was placed in contact with the polyester film at the centre of the bar-shaped specimen and irradiated for 20 s. The light tip diameter was incremented longitudinally along the bar-shaped specimen such that a quarter of the light tip diameter overlapped with the previously exposed central area and were light irradiated for a further 20 s (Bhamra et al.,

2008) such that the bar-shaped specimens were irradiated in three steps. The aluminium split-mould was disassembled and the irradiation protocol repeated on the opposing surface of the bar-shaped specimen. Any excess residual flash remaining on the bar-shaped specimen after removal from the aluminium split-mould was carefully removed and bar-shaped specimens with visually apparent surface defects or porosity inclusions were discarded. The bar-shaped specimens were stored dry in a dark incubator at 37°C for 24 h prior to testing in three-point flexure at a loading rate of 1 mm/min (Bhamra et al., 2008) to determine the flexural moduli of each RBC system employed.

2.2 PORCELAIN SPECIMEN MANUFACTURING ROUTE

The actual strength of dental ceramic materials is predominantly influenced by both surface condition (Kelly et al., 1989; Kelly et al., 1990; Zhang et al., 2004; Quinn et al., 2005; Anusavice and Hojjatie, 1992) and environmental factors (Kim et al., 1999; Yeo et al., 2000; Lee et al., 2002; Gonzaga et al., 2011), but is also dependent on the strength testing methodology employed (Ban and Anusavice, 1990). Therefore the actual strength of a dental ceramic material is routinely determined to be orders of magnitude less than the theoretical bulk strength and differences have been explained by the presence of surface defects (Lawn, 1993; Ritter, 1995). These defects are susceptible to concentrated tensile stresses, where crack initiation and propagation results when the energy associated with crack extension exceeds the surface energy associated with the formation of new surfaces (Griffith, 1921). The observed scatter in the strength data for dental ceramic specimens is reported to be largely dependent on the presence of a critical flaw when exposed to an applied tensile stress (Kingery et al., 1976). Therefore, the distributed nature of surface flaws and crack tip fracture behaviour explains why the strength of dental ceramics can never be reliably determined

from 'average' fracture strengths (McLean and Kedge, 1987). The dependence of brittle fracture strength on the surface defect configuration and distribution implies that the surface area and volume of the specimen are also influential when considering the domain over which the surface flaw population exists (Jayatilaka and Trustum, 1977; Quinn and Quinn, 2010). While the surface area and volume can be negated by using a single specimen geometry and testing methodology, one limitation of all strength testing methodologies is in the dental ceramic sample preparation. Variability in the measured strength can be introduced during fabrication (Gill, 1932; Fleming et al., 2000), through sintering or heat-pressing (Isgro et al., 2010) and even CAD/CAM routes (Addison et al., 2012) which is further influenced by the technical skill of the operator (Piddock et al., 1984; Fleming et al., 2000). Variability introduced by the operator, during specimen preparation, was observed by Marquis and Fisher (1993) who identified that surface flaws introduced during dental porcelain condensation were strength limiting. Further refinement in operator technique to improve the surface quality of the ceramic by employing platinum foil burnishing and pre-heating led to an increase in the biaxial flexure strength (BFS). In addition, Fleming et al. (2000) demonstrated that the reliability of the dentine porcelain used in the current study was significantly influenced by the porcelain powder to modelling liquid mixing ratio. In a dental laboratory setting, the preferred slurry consistency determined by dental technicians is typically dictated by the subjective nature of the porcelain powder to modelling liquid ratio routinely employed during condensation of porcelain restorations. An optimum of porcelain powder to modelling liquid mixing ratio was identified and disc-shaped specimens prepared with the optimum mixing ratio were observed to have an increased mean BFS of 87.3 (11.9) MPa and Weibull modulus of 8.0 (1.5) with minimum apparent porosity (3.3-4.1%). Specimens fabricated with reduced (85.1 (12.9) MPa, 7.2 (1.3) and 4.8-5.3%, respectively) or increased (81.9 (13.2) MPa, 6.7 (1.2) and 5.6-6.2%, respectively) porcelain powder contents,

for a constant volume of modelling liquid had inferior properties (Fleming et al., 2000). Consequently, the optimum porcelain powder to modelling liquid mixing ratio identified by Fleming et al. (2000) was employed in the current investigation to reproducibly prepare nominally identical dentine porcelain disc-shaped specimens.

Nominally identical Vitadur Alpha dentine porcelain disc-shaped specimens (13.0 (0.2) mm diameter, 1.90 (0.06) mm thickness) were formed using a Perspex ring-mould burnished with aluminium foil to produce a consistent surface texture. The condensation method outlined by Fleming et al. (2000) was employed whereby an optimum powder to liquid slurry consistency was obtained by manipulating 1.05 g of powder (Shade A2, Lot #62167, Vita, Bad Säckingen, Germany) weighed using an electronic precision balance (Sartorius Expert, Sartorius AG, Goettingen, Germany) accurate to 1 µg. The porcelain powder was placed on a porcelain mixing tray with 0.3 ml of Vitadur Modeling Fluid (Lot #14209R, Vita, Bad Säckingen, Germany) delivered using a calibrated 200 µl micropipette (Gilson, Villiers-le-Bel, France). The porcelain slurry was formed with minimal movement using a paintbrush, prior to being transferred into a secured Perspex ring-mould. The condensation technique involved vibration for 90 s on an ultrasonic condenser (Ceramasonic II, Shofu Dental Corporation, Kyoto, Japan), to displace the excess modeling fluid to the surface, which was manually blotted dry with tissue paper. The overfilled condensed porcelain was then incrementally removed with a sharp razor blade, maintained flush with the surface of the Perspex mould, to level the surface of the specimen. The condensed porcelain disc-shaped specimen was carefully transferred to a silicon nitride firing slab which was then placed in a vacuum furnace (Vita Vacumat 40T, Bad Säckingen, Germany) to facilitate sintering in accordance with manufacturer's guidelines. The specimens were pre-dried at 600°C for 6 mins, fired under vacuum from 600 to 960°C at a heating rate of 60°C/min, air fired at 960°C

for a further 60 s and cooled at 20°C/min to room temperature. After cooling, the specimens were examined visually to ensure no surface imperfections were evident. The specimens were labelled to differentiate the glazed surface which was in contact with the silicon nitride firing slab during sintering and the fit surface which was levelled with the sharp razor blade. The disc-shaped specimens were stored in a desiccator at ambient temperature to prevent hydrolysis of the silicate bonds (Budd, 1961) until required for further testing and analysis.

2.3 SODA LIME GLASS ANALOGUE SPECIMEN PREPARATION

It was considered that it would be useful to identify a substrate from which reliable, bulk-defect-free nominally identical samples could be produced. As the important strengthening effect to be observed are surface dominated (Addison et al., 2007a-c), the fracture toughness of the material is less important, however, the dependence of resin-strengthening on surface defects must be analogous to the previously reported data (Addison et al., 2007d; 2008). There are several potential benefits of using soda lime glass disc-shaped specimens cut from a prefabricated round section rod stock instead of manually condensing porcelain disc-shaped specimens (Gill, 1932; Fleming et al., 2000) most notably the inclusion of sub-surface porosity which may not be apparent on visual inspection (Fleming et al., 2000). Additionally, the sintering process itself can induce residual thermal stresses (Isgro et al., 2010; Isgro et al., 2011) which may cause geometric deformation and effectively reduce the reliability of the BFS data (Isgro et al., 2011). The disc-shaped specimens prepared by cutting a soda lime glass rod may be controlled such that nominally identical specimens can be reproduced consistently with a minimal surface flaw and bulk defect population.

The flexural modulus of the soda lime glass was firstly determined to ensure it was within the range of flexural moduli routinely observed for dentine porcelains (35-64 GPa) employed to manufacture PLV and DBC restorations. The transparent soda lime glass rod stock was visually inspected for surface and bulk defects prior to sectioning. The soda lime glass bar-shaped specimens were prepared by cutting the supplied soda lime glass rod stock into 25 mm length sections using a precision circular saw (Isomet1 Low Speed Saw, Buehler, Lake Bluff, IL, USA) coupled with a diamond wafering blade (15 HC Diamond, Buehler, Lake Bluff, IL, USA) rotating at 170 rpm under water irrigation. The 25 mm length sections of soda lime glass rod stock were then sectioned into rectangular bar-shaped specimens (25 mm length, 2 mm width, 2 mm thickness) which were visually inspected for surface and bulk defects. Any residual flash was carefully removed by hand-lapping using P400 silicon carbide abrasive paper (Buehler, Lake Bluff, IL, USA) under water lubrication and light finger pressure. The soda lime glass bar-shaped specimens were dried immediately with oil-free compressed air and stored in a desiccator at ambient temperature to prevent hydrolysis of the silicate bonds (Budd, 1961) prior to further testing and analysis in a standard three-point flexure test assembly (ISO 4049:2000).

The flat nominally identical soda lime glass disc-shaped specimens (11.4 mm diameter) were prepared by transversely sectioning the soda lime glass rod at 1 mm intervals using the precision circular saw coupled with the diamond wafering blade operating at 170 rpm with water irrigation. The transverse sectioning with the rotating diamond wafering blade occasionally resulted in the formation of a minor lip on the surface of the removed disc-shaped specimen which was facing the rotating diamond wafering blade due to the lateral deflection of the wafering blade at the end of the sectioning cycle caused by the release of the disc-shaped specimen. Residual flash and lipping were carefully removed by hand-lapping

using P400 silicon carbide abrasive paper and the soda lime glass disc-shaped specimens were visually inspected for surface and bulk defects. The soda lime glass disc-shaped specimens were stored in a desiccator at ambient temperature until required for further preparation, testing and analysis.

The range of single curved soda lime glass disc-shaped specimens selected for the current study was representative of the curvatures evident in PLV and DBC dental restorations. The generic three-dimensional geometries of the four single curved disc-shaped specimens chosen, each with different radii of curvature, were created with a parametric three-dimensional CAD system (Pro/Engineer, Wildfire 2.0, Parametric Technology Corporation, Needham, MA, USA). The three-dimensional geometry was generated by sketching an axis of revolution and creating a parametric two-dimensional hemi-section of the curved disc-shaped specimen which was then revolved 360° about the axis of revolution to form a solid feature of the curved disc-shaped specimen. The nominal thickness of each curved disc-shaped specimen was specified at 1.0 mm with the diameter specified at 12 mm and the curved disc-shaped specimen geometries specified with radii of curvature at 40, 30, 20 and 10 mm. Detailed drawings of the four curved disc-shaped specimen CAD geometries generated from the three-dimensional geometries are shown in Figure (2.1) and were employed to specify the design and manufacture of the furnace mould tools used to fabricate the curved disc-shaped specimens by slumping (Advanced Glass Industries, Rochester, NY, USA) flat soda lime glass stock (AGC Glass Europe, Brussels, Belgium).

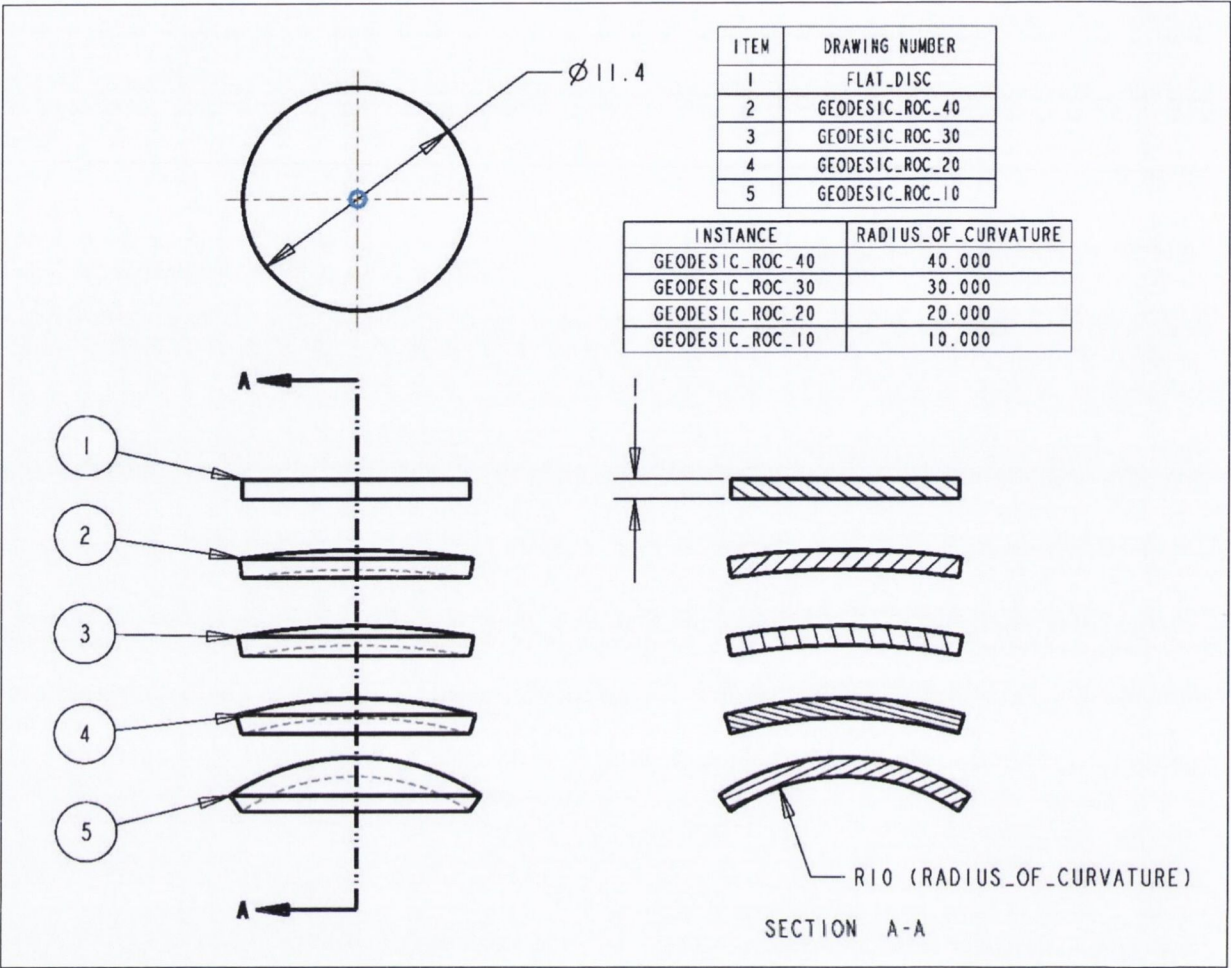


Figure 2.1: Two-dimensional schematic of the flat and curved disc-shaped specimens

Furnace mould tools were fabricated according to the dimensions specified in the detailed drawing of the each of the four curved disc-shaped specimens (Figure 2.1), with compensated specimen radii to achieve each of the specified radii of curvature to slump the flat soda lime glass disc-shaped specimens. The flat soda lime disc-shaped specimens were scribe-cut from the flat soda lime glass stock and then edged by chamfering. A quantity of flat soda lime glass disc-shaped specimens were designated as the flat disc-shaped control specimens for BFS testing to compare with the BFS test data for the curved disc-shaped specimens. The soda lime glass disc-shaped specimens to be formed were heated to 575°C in the furnace mould which was rotated four times through 90° every 60 s to complete the slumping process

and achieve the specified curvature. The curved soda lime glass disc-shaped specimens were then allowed to cool to ambient temperature at a rate of 1°C/s.

The central thickness of each flat and curved soda lime glass disc-shaped specimen was measured with a screw gauge micrometer fitted with a ball-nose adapter on the spindle tip which was accurate to 1 µm (Mitutoyo Corporation, Tokyo, Japan). The ball-nose adapter of the screw gauge micrometer was placed in contact with the pre-cementation treated flat or concave surface of the disc-shaped specimen during assessment of disc thicknesses. The flat and curved soda lime glass disc-shaped specimens were individually placed into separate plate wells and stored in a desiccator prior to testing and analysis.

2.4 MECHANICAL TESTING

When assessing the suitability of in vitro research methodologies to provide meaningful clinical guidance, the fundamental questions that need to be asked are: What do you really want to know? What do you want to measure? and What are you going to measure? (Darvell, 1995). In examining the failure mechanisms in dental ceramics, the selected laboratory test configuration should identify the determinants of failure which result in the failure modes routinely observed in the clinical setting for the restoration type under investigation. The abundantly prevalent load to failure data of dental ceramics in the literature (Burke and Watts, 1994; Ferro et al., 1994; Pallis et al., 1994; Yoshinari et al., 1994; Evans and O'Brien, 1999; Burke, 1999; Behr et al., 2003; Bindl et al., 2006a,b; Aboushelib et al., 2007; Blatz et al., 2008; Borges et al., 2009) include the “crunch-the-crown” test method which attempts to replicate clinical failure with anatomically correct test specimens. The clinical relevance of the “crunch-the-crown” test has been challenged in the dental literature for several reasons

(Fleming et al., 2006; Isgro et al., 2010; Kelly, 2012) Although the geometry of the test specimen is realistic, the failure mode involves indenter contact surface damage and does not replicate the failure mechanism of sub-surface radial fracture which is evident in the clinical setting (Kelly et al., 2012). The forces identified to initiate failure have been demonstrated to result in excessive masticatory and parafunctional loads (Gibbs et al., 1981; Josephson et al., 1985; Josephson et al., 1991; Smith et al., 1994; Carrier and Kelly 1995) and contact stress (Lawn, 1993; Kikuchi et al., 1997). Clinical failure of all-ceramic restorations often results in a low number of fracture fragments, usually two, while the “crunch-the-crown” test method usually results in multiple fracture fragments indicative of the release of excessive stored internal energy (Fleming et al., 2006; Isgro et al., 2010; Kelly et al., 2010). The method is also limited in that it reports only the load at which failure occurs without describing the stressing patterns within the complex geometry of the specimen and therefore comparison between studies is problematic.

All-ceramic restorations are known from axisymmetric finite element analysis studies (Anusavice and Hojjatie, 1992; Kelly, 1999; Dong and Darvell, 2003; de Jager et al., 2006) to fail due to the tensile extension of pre-existing flaws on the intaglio surface resulting in sub-surface radial fracture. Therefore the clinical relevance of testing modalities which result in contact failure at the loaded surface have been challenged (Kelly, 1999; Kelly et al., 2012). The BFS test has been advocated as a method which reliably reproduces the failure mode of sub-surface radial fracture observed in the clinical setting (Ban and Anusavice, 1990). A variation on the standardised piston-on-three-ball BFS test (ISO 6872:1995) which results in failure due to radial fracture is the ball-on-ring test. The ball-on-ring test has been advocated as the most reliable method of assessing the BFS (de With and Wagemans, 1989) and has been widely employed to evaluate the strength of dental ceramics (Ban et al., 1992). The

coaxial loading and deflection of a disc-shaped specimen which is supported at its periphery concentrates the stress field towards the centre of the specimen and reduces edge effects (Bernebaum and Brodie, 1959). The existence of support friction can influence the stress distribution within a specimen and result in an overestimation of strength. As friction is increased, the stress in the central region reduces although the magnitude of the reduction in the stress is reduced for thicker specimens (Morrell, 2007).

The current investigation employed BFS testing of the disc-shaped specimens in a ball-on-ring configuration. The disc-shaped specimens were loaded coaxially with a 4 mm diameter stainless steel ball indenter while supported on a 10 mm diameter knife edge ring support in a universal testing machine (Instron Model 5565, Instron Ltd., High Wycombe, UK). The analytical solutions and finite element models employed in the current study assumed frictionless supports and consistent boundary conditions in the experimental setting were maintained with a thin latex sheet (0.2 mm thickness) between the disc-shaped specimens and the ring support. The compliance of the thin latex sheet minimised friction with the ring support, permitting small radial displacement of the disc-shaped specimen during loading and stabilised the disc-shaped specimen axially where any out of plane geometric irregularities existed across the specimen surface.

2.5 ANALYTICAL SOLUTION

In bending theory, the concept of a neutral plane is defined by zero longitudinal deformation or strain in flexure and coincides with the geometric centroid for a homogenous symmetrical structure (Benham and Crawford, 1989). The neutral plane describes the location within a beam cross-section placed under flexure where an overall compressive stress is transitioned

to a net tensile stress. The analysis of bending stress and deflection for a composite beam with asymmetrical geometry about the geometric centroid with dissimilar material properties (flexural modulus and Poisson's ratio), will result in the establishment of a neutral plane position which is not coincident with the geometric centroid.

The analytical solutions developed by Hsueh et al. (2005) to calculate the axial stress distribution within a flat bilayered disc-shaped specimen were employed in the current investigation to determine the biaxial flexure stress at failure. The analytical equations involve the calculation of a modified neutral plane which is a function of the asymmetric layer thicknesses, in addition to the dissimilar flexural moduli and Poisson's ratios of the bilayer components. The material discontinuity and layer thickness asymmetry are determinants in the calculation of the position of the neutral plane (t_n) which is calculated as a function of the individual layer thicknesses (t_1 and t_2) and flexural moduli (E_1 and E_2) from Equation 2.1.

$$t_n = \frac{E_1^*(t_1)^2 - E_2^*(t_2)^2}{2(E_1^*t_1 + E_2^*t_2)} \quad \text{Equation 2.1}$$

$$\text{where } E_1^* = \frac{E_1}{1-\nu_1^2} \quad \text{Equation 2.2(a)}$$

$$\text{and } E_2^* = \frac{E_2}{1-\nu_2^2} \quad \text{Equation 2.2(b)}$$

The average Poisson's ratio (ν) of a bilayered disc is determined as a function of the individual layer Poisson's ratios and layer thicknesses using Equation 2.3

$$\nu = \frac{(\nu_1 t_1 + \nu_2 t_2)}{t_1 + t_2} \quad \text{Equation 2.3}$$

where ν_1 and ν_2 are the Poisson's ratios of each layer, respectively. The biaxial flexure stress is calculated coaxially ($r=0$) at the bonded interface ($z=0$) with Equation's 2.4 and 2.5.

$$\sigma = \frac{-3P(1+\nu)(z-t_1)}{2\pi(t_1+t_2)^3} \left[1 + 2\ln\left(\frac{a}{b}\right) + \frac{1-\nu}{1+\nu} \left(1 - \frac{b^2}{2a^2} \right) \frac{a^2}{R^2} \right] \left[\frac{E_1^*(E_1^*t_1 + E_2^*t_2)(t_1+t_2)^3}{(E_1^*t_1^2)^2 + (E_2^*t_2^2)^2 + 2E_1^*E_2^*t_1t_2(2t_1^2 + 2t_2^2 + 3t_1t_2)} \right]$$

Equation 2.4

for $(0 \leq z \leq t_1)$

$$\text{where } b = \frac{(t_1 + t_2)}{3}$$

Equation 2.5

and where z is the axial position, P the measured load at failure (N) and a is the radius of the knife-edge support (mm). The radii of the loaded region and the disc-shaped specimen are b and R , respectively. Therefore, the biaxial flexure stress for monolayer disc-shaped specimens are determined coaxially ($r=0$) on the surface in tension ($z=0$) by setting $t_2=0$ using Equations 2.2-2.5.

To enable accurate quantification of the axial stress distribution within a flat bilayered (resin-coated) disc-shaped specimen, the flexural moduli of the RBC and soda lime glass materials employed in the current study were determined by loading bar-shaped specimens to failure with a three-point flexure testing assembly (ISO 4049:2000). The bar-shaped specimens were placed on a 20 mm span support and loaded centrally to failure at a crosshead speed of 1 mm/min with a universal testing machine (Instron Model 5565, Instron Ltd., High Wycombe, UK). The cross-sectional dimensions of each fractured bar-shaped specimen fragment were measured at the fractured surface using a screw gauge micrometer accurate to 1 μm (Mitutoyo Corp., Tokyo, Japan). The slope of the linear elastic region of the load-deflection curves were determined and flexural moduli calculated using Equation 2.6.

$$E = \frac{L^3}{4bd^3} \cdot \frac{\Delta P}{\Delta D}$$

Equation 2.6

where E is the flexural modulus, L the support span (20 mm), b the specimen width (mm), d the specimen thickness (mm) and $\Delta P/\Delta D$ is the slope of the linear elastic region of the load-deflection curve.

2.6 NUMERICAL SIMULATION

The application of closed-form analytical solutions to determine the stress and strain distribution within a structure is usually limited to simple geometries. Understanding the influence of composite structures, such as resin-coated bilayered disc-shaped specimens, on the resultant stresses imposed by an applied load is often not possible when employing analytical solutions in isolation. Finite element analysis (FEA) is a numerical technique commonly employed by engineers to simulate the mechanical behaviour of complex geometries in a virtual environment. The FEA method originated in the early 1940's with the seminal work of Hrennikoff (1941) and Courant (1943) and involved the numerical approximation to boundary value problems by mesh discretisation of a continuous domain into smaller parts, or elements. By subdividing the global geometry into a mesh of small elements, the differential equations or shape functions which defined the response of each small element can be solved iteratively to estimate the global response of the structure.

Commercially available FEA software packages employ either the classical H- or P-type element to mesh the geometry and achieve iterative convergence of the numerical solutions which describe the stress and strain distribution within the structure. The methods which employ the H-type elements achieve geometric approximation by adaptive refinement in mesh size and distribution to obtain solution convergence while the polynomial order of the shape function defining the element boundaries remains constant. The less commonly

employed approaches which utilise the P-type elements, achieve convergence by increasing the polynomial order of the shape function which defines the element boundaries without increasing the number or reducing the size of the elements to achieve discretisation of the global geometry. The main advantage of employing P-type elements is the efficient and flexible modelling of complex geometric shapes without adaptive mesh refinement. Therefore, employing the P-type elements to model the behaviour of the system requires less user input when generating the mesh for the simulated geometric domain. Furthermore, the technique of incrementing the order of the polynomial which defines the shape function to achieve accuracy and minimise error without modifying the mesh, is highly conducive to parametric design optimisation and sensitivity studies which interrogate the model for geometric effects on the stress distribution within a structure.

In general, the FEA method is a fundamental design and analysis tool for engineering applications and the technique has been widely reported in the dental literature (Geng et al., 2008; Shetty et al., 2010; May et al., 2010) since the early implementation of FEA in dentistry by Ledley and Huang (1968) to analyse tooth displacements under an applied load. The FEA method was employed to verify the analytical solution determined by Hsueh et al. (2005) to calculate the biaxial flexure stress in the bilayered disc-shaped specimen. Similarly, in the current study, the three-dimensional CAD geometry of the flat disc-shaped specimens was examined using the FEA method to verify the analytical solution employed to calculate the biaxial flexure stress in the bilayered disc-shaped specimens, to validate the experimentally determined findings.

In addition, the verified FEA model was adapted to simulate the stress distribution within single curved disc-shaped specimens where no analytical solution existed to determine the

BFS. The parametric three-dimensional CAD geometries of the nominally dimensioned flat and curved disc-shaped specimens were sectioned through the axis of revolution to create the two-dimensional hemi-surface which was imported into a linear P-code FEA software, Pro/MECHANICA (Wildfire 2.0, Parametric Technology Corporation, Needham, MA, USA). The model type was defined as two-dimensional axisymmetric using triangular solid P-type elements. The mesh was automatically generated with the number of nodes assigned to the edge at the symmetry axis set to 20 and the number of nodes assigned to the top and bottom edges set to 60, resulting in a mesh with 196 elements. The symmetry axis of revolution prevented rigid body motion in the radial direction by applying a zero displacement radial constraint. The boundary displacement constraints reflected the ball-on-ring experimental test configuration whereby a frictionless ring support constraint was simulated by fixing the radial support nodes axially (vertical displacement) at the ring support radius while allowing free radial (horizontal) displacement. The flexural modulus and Poisson's ratio were specified for the material assigned to each component layer and the interface between the layers was assumed to be an ideal bond. The parametric three-dimensional model was manually specified for each disc-shaped specimen tested. An equivalent ball indenter load was applied as a uniformly distributed load to the outer edge of the two-dimensional axisymmetric model of the disc-shaped specimen over a length from the symmetry axis which was equal to the radius determined by the equation for estimating the load contact area (Equation 2.5) in the analytical solution. The measured central thicknesses of each component layer, radius of curvature and failure load determined from the BFS testing were specified in the model to simulate the actual disc-shaped specimen under investigation. The FEA solver was configured to run a static analysis with a maximum polynomial value of nine and a multi-pass adaptive convergence criteria of 3% for a user-defined measure parameter of maximum principal stress at an axial position ($r=0$) in the disc-

shaped specimen on the lower surface in tension ($z=0$), in addition to the local strain energy. The runtime log was checked to confirm the convergence criteria were satisfied. The user-defined measure parameter of maximum principal stress at an axial position ($r=0$) in the disc-shaped specimen on the lower surface in tension ($z=0$) was recorded for the simulated disc-shaped specimen. The measure parameter of maximum principal stress determined with the FEA simulation corresponded to the resultant BFS due to the applied load at failure which was measured experimentally.

2.7 STATISTICAL ANALYSIS

In general, the correlation coefficient will increase as sample size increases and groups of 10 specimens are often employed by investigators in the dental literature although data obtained with this sample size is unlikely to give reliable results (McCabe and Carrick, 1986). By increasing the sample size and therefore the power of a study, more dependable results can be obtained (Swinscow, 1996). The determination of sample size is dependent on the type one and type two error rates. The variability of the data and the effect size and the sample size employed in a study should be justified for the data to be meaningful (Hannigan and Lynch, 2013). Type one error is to reject the null hypothesis when it is true while a type two error is to accept the null hypothesis where there is a difference between group data (Swinscow, 1996). The variability of the data is described by the standard deviation and the effect size is the magnitude of the difference to be detected. The conventional default values for type one and type two error rates are 5 and 20%, respectively, while the appropriate effect size for a study can be estimated if the examined data is well understood or where precedent data exists.

In the current study, the dependent variable to be examined is the BFS with factors including specimen thickness, pre-cementation surface treatment and RBC material flexural modulus. The BFS testing of dental ceramics can lead to standard deviations up to 25%, largely due to the statistical nature of brittle fracture (Addison, 2007). There exists precedent BFS data for disc-shaped specimens fabricated with the same dentine porcelain employed in the current investigation (Vitadur Alpha, Vita Zahnfabrik H. Rauter GmbH & Co. KG, Bad Säckingen, Germany) which demonstrated standard deviations of up to 13.6 MPa, equivalent to 13.3% of group mean BFS data (Bhamra et al., 2002). A further study which reported the BFS data for disc-shaped specimens fabricated with the Vitadur Alpha dentine porcelain identified standard deviations up to 17.1 MPa, equivalent to 15.4% of the group mean BFS data (Addison et al., 2007c). Based on the reported precedent data, the sample size n in the current investigation was determined with Equation 2.7 (Swinscow, 1996).

$$n = 16 \cdot \frac{\sigma^2}{d^2} \quad \text{Equation 2.7}$$

where σ was the standard deviation conservatively assumed to be 10 MPa for the uncoated control disc-shaped specimens with a minimum group mean BFS of 58 MPa (Addison et al., 2007d) and d was the effect size specified to be 15% of the group mean BFS which resulted in a sample size of $n=21$. It is important to note that the group mean BFS of 58 MPa was reported with an associated standard deviation of 4.7 MPa (Addison et al., 2007d), however, the assumed standard deviation of 10 MPa for the uncoated control disc-shaped specimens provides a factor of safety of two in the determination of sample size. Furthermore, a sample size of $n=24$ was employed in the current study to exceed the minimum sample size required to identify the prescribed effect size.

Group BFS data were tested for normality with the Shapiro-Wilk test with a critical alpha value of 0.05. The analysis of group mean BFS data was performed utilising a generalised

linear model univariate analysis with multiple factors. Post hoc all paired Tukey tests were employed at a significance level of $P < 0.05$ (SPSS for Windows 14, SPSS Inc., Chicago, USA) to test for differences between individual group mean BFS data. Group mean data were plotted and fitted with linear regression curves to identify the correlation coefficient from the coefficient of determination (R^2 value). The linear regression curves were extrapolated to a theoretical 'zero' resin-coating thickness to estimate the 'actual' magnitude of strengthening compared with the uncoated control.

2.8 SURFACE CHARACTERISATION

2.8.1 Surface topography

The topographical features which characterise a surface area are generally described by the measured parameters which define the surface topography. The surface topography parameters which are universally employed to characterise a surface include form, waviness and roughness (ISO 4287:1998+A1:2009). The form describes the general shape of a profile or surface area and is either the result of deliberate design intent or a manifestation of non-cyclic or long wavelength deviation from the nominal form. Waviness (Wa-value) is the widely spaced periodic deviation normally introduced due to machining vibration and is the standardised international parameter of waviness. The roughness (Ra-value) is the standardised international parameter described by the arithmetic mean of the absolute departures of the roughness profile from a mean line (Bhamra et al., 2002) and is the periodic deviation of short wavelength which is superimposed on the waviness. Determinants of roughness include machining tool aberrations and material composition factors. The primary trace is processed with a high pass filter to extract or 'cut-off' the Ra-value and a low pass filter to identify the Wa-value.

2.8.2 Profilometry techniques

A profile may be visualised as the planar intersection with a surface. Profilometry is a two-dimensional measurement technique employed to identify the characteristics of a profile by scanning a surface along a fixed trajectory while measuring the point by point variation in height. Profile waviness (Wa-value) and profile roughness (Ra-value) parameters describe the profile characteristics along the scanned trajectory. By incrementing the fixed trajectory and scanning repeated profiles, the surface area characteristics are established by reconstituting the scanned two-dimensional profiles. Recent developments in three-dimensional surface metrology have led to the introduction of updated standardised terminology which more comprehensively describes three-dimensional surface characteristics (ISO 25178-2:2012) to expand on the two-dimensional determination of surface characteristics.

The measurement of surface form, waviness and roughness is performed with a non-contact or contact type of profilometer. Non-contact profilometry employs optical techniques such as laser triangulation, confocal microscopy or low coherence interferometry to scan and characterise the surface. Non-contact profilometry is indicated where there is a need to avoid surface contact damage and the reflective properties of the surface are compatible with the selected optical scanning method. Transparent and hard surfaces are appropriately scanned with contact profilometry which involves a mechanical apparatus to record the vertical deflection of a sharp stylus tip in physical contact with the examined surface which translates along a straight trajectory to report a traced primary profile over the scanned length. The performance of contact and non-contact profilometry techniques were compared and validated (Whitehead et al., 1999) when employed to assess monolithic dental glass-ceramic blocks (Dicor, Corning, NY, USA). Contact profilometry has been successfully employed to

assess the surface characteristics of similarly fabricated dentine porcelain disc-shaped specimens (Addison et al., 2007; Isgro et al. 2010, Isgro et al., 2011).

2.8.3 Profilometry of the flat disc-shaped specimens

In the current investigation, the flat disc-shaped specimens were placed on a stable custom-made multi-axial levelling device during profilometric analysis performed with an inductive gauge conisphere diamond stylus tip (2 μm radius) using a Talysurf CLI 2000 (Taylor Hobson Ltd., Leicester, UK). The displacement measurements were performed at a measurement velocity of 3 mm/s and an applied load of 0.75 mN across a rectangular area of 10 mm² (10 mm length and 1 mm width) that included the centre and extending to the periphery of the disc-shaped specimen. The scan resolution was 3334 points (x-direction) and 334 traces (y-direction) using a 3 mm step size (x- and y-direction) and vertical resolution of 40 nm (z-direction). The mean Ra-value and mean Wa-value were determined by extracting and averaging 20 traces in the y-direction.

2.8.4 Profilometry of the curved disc-shaped specimens

Profilometry of the curved disc-shaped specimens was performed to verify that the curvatures of the disc-shaped specimens manufactured by slumping and received by AGI were as specified in Figure (2.1). Random selections of three specimens from each batch of the flat and curved disc-shaped specimens were examined. The curved disc-shaped specimens were placed securely on a bespoke platform while profilometric scanning was completed on the concave surface with the inductive gauge conisphere diamond stylus tip using the Talysurf CLI 2000. The displacement measurements were obtained at a measurement velocity of 1 mm/s and an applied load of 0.75 mN across a rectangular area of 18 mm² (9 mm length and 2 mm width) that included the centre and extended radially to the periphery of the soda lime

glass disc-shaped specimen. The scan resolution was 901 points (x-direction) and 201 traces (y-direction) with a resolution of 40 nm in the z-direction.

A random sample set of three curved soda lime glass disc-shaped specimens for each radius of curvature investigated were profilometrically evaluated to determine the radius of curvature obtained by slumping for each curved soda lime disc-shaped specimen. In addition, the surface roughness (Ra-value) and surface waviness (Wa-value) were established to assess the surface quality. The scan area was determined diametrically in the x-direction and also the y-direction to calculate an average radius of curvature for each soda lime glass disc-shaped specimen for comparison with the respective specified values. Three scan profiles were extracted from the scan area determined in the x-direction and three scan profiles were extracted from the scan area determined in the y-direction. The extracted scan profile was assumed to be circular and the radius of curvature (R) was estimated in the x-direction and y-direction using an equation based on Heron's formula (Equation 2.8) by calculating the distances between three points identified on the trace and an average radius of curvature was determined.

$$R = \frac{abc}{4 \cdot \sqrt{s(s-a)(s-b)(s-c)}} \quad \text{Equation 2.8}$$

where a , b and c are the distances between the three points, s is the semi-perimeter defined by the three points and calculated using Equation 2.9.

$$s = \frac{a+b+c}{2} \quad \text{Equation 2.9}$$

The radii of curvature of the curved disc-shaped specimens identified with profilometry were employed to define the geometries modelled in the FEA study to reflect the experimental BFS test configuration.

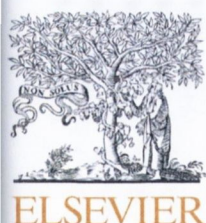
3. SELECTED MANUSCRIPTS

The current investigation is presented as a series of manuscripts which have been published (Manuscripts 3.1 - 3.3) or submitted for publication (Manuscripts 3.4 - 3.5) in relevant peer-reviewed dental journals, namely 'Dental Materials' (Manuscript 3.1), 'Journal of Dentistry' (Manuscripts 3.2 - 3.4) and Acta Biomaterialia (Manuscript 3.5). The manuscripts are therefore presented in the style and format appropriate for each respective journal and comprehensively summarised to describe the sequence of work in the context of the overall investigation.

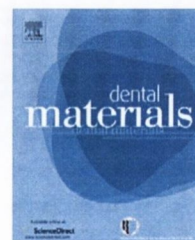
3.1 THE INFLUENCE OF RESIN FLEXURAL MODULUS ON THE MAGNITUDE OF CERAMIC STRENGTHENING

GJP Fleming, P Hooi, O Addison (2012). The influence of resin flexural modulus on the magnitude of ceramic strengthening. *Dental Materials*, 28:769-776.

Improved clinical performance of PLV and DBC restorations has been reported when RBC luting materials are employed to adhesively bond the restorations to the prepared tooth (Malament and Socransky, 1999, 2001, 2010). Previous research has indicated that the adhesive cementation of dentine porcelain disc-shaped specimens with RBC materials with different flexural modulus (Fleming et al., 2006; Addison et al., 2007d) and resin layer thickness (Addison et al., 2008) influence the observed magnitude and pattern of resin-strengthening, but the strengthening mechanism is not fully understood (Addison et al., 2007d, 2008). In the context of the overall investigation, the current study examined the influence of both the RBC flexural modulus and the resin layer thickness on the BFS of dentine porcelain disc-shaped specimens. Further evidence was identified to demonstrate the presence and function of the resin-ceramic hybrid layer. The hypothesis tested was that increasing the resin flexural modulus would result in increased resin-strengthening observed at a theoretical 'zero' resin-coating thickness. The RBC materials were selected with different flexural moduli and the dentine porcelain disc-shaped specimens were resin-cemented at clinically relevant layer thicknesses to emulate the RBC cementation of PLV and DBC restorations in the clinical setting. The analytical solutions utilised to determine the maximum tensile stress in the disc-shaped specimen tested in biaxial flexure were verified with FEA to validate the experimental results. Linear regression curves were fitted to the mean BFS data for each RBC material through the discrete resin-coating thicknesses.

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The influence of resin flexural modulus on the magnitude of ceramic strengthening

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ABSTRACT

Introduction. The aim was to determine the magnitude of ceramic resin-strengthening with resin-based materials with varying flexural moduli using a regression technique to assess the theoretical strengthening at a 'zero' resin-coating thickness. The hypothesis tested was that experimentally, increasing resin flexural modulus results in increased resin-strengthening observed at a theoretical 'zero' resin-coating thickness.

Method. Vitadur Alpha dentin porcelain disk ($n=250$) were condensed, fired, alumina particle air abraded and randomly assigned into ten groups. Groups were resin-coated at 50, 100 and 150 μm with Venus Flow, Rely-X Veneer and Clearfil AP-X before biaxial flexure testing at 24 h and the stress at failure calculated using a multilayer analysis. An analytical methodological approach was undertaken to predict the biaxial flexure stresses under boundary conditions that reflected the experimental test and a finite element model was used to verify the analytical prediction.

Results. The magnitude of resin-reinforcement was significantly influenced by resin-coating type ($P < 0.001$) and resin-coating thickness ($P = 0.013$), however, a significant interaction was observed between resin-coating type and thickness ($P = 0.048$). Linear regression identified a 17, 38 and 47% biaxial flexure strength contribution when Venus Flow, Rely-X Veneer and Clearfil AP-X were used, respectively. The finite element model determined the maximum principal stress was within 3.3% of the predicted analytical solution.

Significance. Experimentally, the flexural modulus and thickness of resin-based material used to cement DBC or PLV restorations have a significant impact on the magnitude of resin-strengthening observed. However, for resin-based materials with different flexural moduli the variability in the relationships between thickness and observed increases in biaxial flexure strength of the ceramic requires careful characterization to optimize clinical performance.

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1. Introduction

In 1999 and now more than a decade ago, the 14 year findings of a comprehensive prospective clinical study into the

variables determining the survival of 1444 restorations fabricated from a single glass-ceramic substrate were reported [1,2]. The authors demonstrated the glass-ceramic restorations were significantly more resistant to fracture when adhesively bonded to the prepared tooth structure using

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resin-based materials compared with when luted with acid–base cements [2]. The clinical outcome data [1,2] supported the proposals made previously by Marquis [3] and Nathanson [4] that adhesive cementation strengthens feldspathic porcelain and dental glass-ceramic restorations [3,4]. Although the two independent investigators provided different hypotheses for the exact resin-strengthening mechanism, both authors agreed the interaction of the resin-based material with the critical flaws on the ceramic surface conferred the magnitude of observed strengthening [3,4]. Over the last decade there has been a significant increase in the use of porcelain laminate veneer (PLV) and dentin bonded crown (DBC) restorations [5] which combine conservative tooth preparation ideals with adhesive bonding strategies to esthetic but relatively weak dental ceramic materials [6]. The clinical success of these restorations fabricated from feldspathic porcelains or glass-ceramics is remarkable given that failure rates in the anterior dentition do not differ dramatically from restorations constructed from ceramic substrates with up to ten-fold increased flexural strengths [7].

In attempts to identify operative strategies available to practitioners' to maximize the magnitude of resin-strengthening, investigators [8–10] have demonstrated the reinforcement achieved was significantly modified by the choice of resin-based material. Resin-based materials have evolved considerably since Bowens' resin-based composite (RBC) formulation [11] and in recent years non-methacrylate monomeric resins [12] and filler technologies encompassing nanotechnology [13] have been developed. Manufacturers have increased the filler volume fraction, thereby increasing the mechanical properties (flexural strength, flexural modulus and wear resistance) [14,15] since in terms of mechanical properties the filler is dominant over the resin [16]. Today, the performance of RBCs which has been reported in a prospective clinical study to be superior to dental amalgam restorations, albeit when placed by an experienced operator [17]. However, while the promotion of superior mechanical properties appears to be the goal in RBC development for restorative applications, the improvement of resin-based luting materials appears to be governed by optimization of the resin viscosity and resin film thickness in efforts to simplify restoration seating and improve marginal adaptation. As a result, the resin-based materials routinely employed for luting feldspathic porcelain and dental glass-ceramic restorations (PLVs or DBCs) are limited to flowable and luting resin formulations which have significantly reduced filler loading, lower flexural moduli [9] and increased viscoelasticity when compared with commercial RBC restorative formulations [14,15].

The magnitude of ceramic strengthening following resin-coating has been demonstrated to be a function of the resin's ability to interpenetrate surface defects forming a 'hybrid-layer' [9]. The reinforcement achievable is therefore dependent on the ceramic surface texture, but is also influenced by intrinsic and mechanical properties of the resin-based material and the ultimate resin-coating thickness [18,19]. A near-linear relationship between the flexural modulus of the resin-coating and ceramic reinforcement has been reported [9]. Consideration of the transient and residual stresses in a dental ceramic before and after resin-coating has shown that resin-based materials exert a compressive stress state across the

entire ceramic surface defect integral due to the volumetric shrinkage associated with polymerization [20,21]. Although the shrinkage stress generated during polymerization of resin-based materials is a function of flexural modulus, the relationship is complicated by differing shrinkage volumes in materials with similar flexural moduli due to their different formulations. The near-linear relationship between resin flexural modulus and ceramic reinforcement is therefore under scrutiny. In a simple bilayered structure such as a dental ceramic disk coated on one surface with resin, the relative stiffness of the variable layer (the resin), alters the position of the neutral plane of the specimen in bending and therefore alters the stress gradient between the neutral plane and the ceramic surface. As the critical flaw is likely to vary in its pre-loaded length, location and orientation the gradient of an increasing tensile stress within the loaded structure is an important consideration alongside the terminal stress value at failure [22,23]. To isolate the absolute reinforcement conferred by resin-based materials with different flexural moduli, the creation of a thin resin-coating ($<10\text{ }\mu\text{m}$) is required, however, this is impracticable [24]. As an alternative the authors have developed a regression technique to approximate the effect of a theoretical 'zero' resin-coating thickness [10].

The aim of the current study was to determine the magnitude of ceramic resin-strengthening with resin-based materials with varying flexural moduli using a regression technique to assess the theoretical strengthening at a 'zero' resin-coating thickness. The hypothesis tested was that experimentally, increasing resin flexural modulus results in increased resin-strengthening observed at a theoretical 'zero' resin-coating thickness.

2. Materials and methods

2.1. Experimental approach

Two hundred and fifty nominally identical Vitadur Alpha dentin porcelain disk-shaped specimens (13.0 (0.2) mm diameter, 1.90 (0.06) mm thickness) were formed using the condensation method outlined by Fleming et al. [25]. An optimum powder to liquid slurry consistency [25] was obtained by manipulating 1.05 g of powder (Shade A2, Lot #62167, Vita, Bad Säckingen, Germany) with 0.3 mL of Vita Modeling Fluid (Lot #14209R, Vita, Bad Säckingen, Germany) in a porcelain mixing tray. The porcelain slurry was condensed into a Perspex mold (15.0 mm diameter, 2.0 mm thickness) secured to a glass slide using clamps. The Perspex mold was vibrated for 90 s, on an ultrasonic condenser (Ceramosonic II, Shofu Dental Corporation, Kyoto, Japan), to displace the excess modeling fluid, which was manually blotted dry with tissue paper. The excess condensed porcelain slurry was removed from the top of the specimen using a razor blade and this was termed the leveled surface [25].

The specimen was removed from the mold and the leveled surface placed in contact with a silicon nitride firing slab. The specimens were fired in a vacuum furnace (Vita Vacumat 40T, Bad Säckingen, Germany) in accordance with manufacturer's guidelines. The specimens were pre-dried at 600 °C for 6 min, fired under vacuum from 600 to 960 °C at a heating rate

of 60 °C/min, air fired at 960 °C for a further 60 s and cooled at 20 °C/min to room temperature. After cooling, the leveled surface was marked and the specimens were stored at 22 °C in a desiccator to prevent hydrolysis [26]. The leveled surface of each specimen was alumina particle air abraded (Eco Dry Oxide System, Dentalarm, Torino, Italy) with 50 µm alumina particles, at a distance of 2 cm from the nozzle of the abrader under a pressure of 2 bar [27]. The alumina particle air abrasion technique was performed orthogonal to the disk surface in a smooth linear sweeping motion for a maximum of 5 s to prevent excessive localized abrasion of the disk surface. The specimens were randomly assigned into ten groups ($n=25$) and the central thicknesses were measured using a screw gauge micrometer accurate to 10 µm (Mitutoyo Corp., Tokyo, Japan). One specimen group (Group A) was stored in a desiccator prior to testing and analysis while the remaining nine groups (Groups B–J) were prepared for resin-coating.

A bespoke Perspex jig used a fixed displacement offset, governed by feeler gauges at the three prescribed thicknesses to enable the application of discrete resin-coating thicknesses (50, 100 and 150 µm) to the alumina particle air abraded disk-surface. The Perspex base plate was covered with a polyester film (Mylar™, DuPont Teijinn Films, Middlesbrough, UK) to prevent resin-adhesion to the base plate. Prior to resin-coating, the alumina particle air abraded surface was silanated with a 3-methacryloxypropyl-trimethoxysilane (Rely-X™ Ceramic Primer, 3M ESPE, St. Paul, MN, USA) and allowed to air dry, in accordance with the manufacturers recommendations. Three different resins were used to coat the disk-shaped specimens. Specimens were resin-coated with Venus Flow (Heraeus, Hanau, Germany) at resin-coating thicknesses of 50 ± 25 µm (Group B), 100 ± 25 µm (Group C) and 150 ± 25 µm (Group D), Rely-X Veneer (3M ESPE, St. Paul, MN, USA) at 50 ± 25 µm (Group E), 100 ± 25 µm (Group F) and 150 ± 25 µm (Group G) and Clearfil AP-X (Kuraray, Kurashiki, Japan) at 50 ± 25 µm (Group H), 100 ± 25 µm (Group I) and 150 ± 25 µm (Group J). For the Clearfil AP-X resin-coated specimens (Groups H–J), the resin was first placed on a glass-slide and heated to 50 °C prior to resin-coating to reduce viscosity of the resin [9] thereby enabling the achievement of the required discrete resin thicknesses. Following resin-coating with Clearfil AP-X, the specimens were allowed to cool to ambient room temperature prior to light irradiation through the Perspex base plate. The specimens were light irradiated using an Optilux 501 (SDS Kerr, Danbury, CT, USA) through the Perspex base plate for 20 s with a 13 mm light tip diameter and the output intensity at the resin surface was 380 ± 21 mW/cm² – determined using a radiometer.

The failure loads (N) of the control (Group A) and the resin-coated (Groups B–J) porcelain disk-shaped specimens were determined using a ball-on-ring test configuration in a universal testing machine (Instron Model 5565, Instron Ltd., High Wycombe, UK) employing a 2 kN load cell following 24 h dry storage. The specimens were tested with the alumina particle air abraded surface (Group A) or the resin-coated surface (Groups B–J) in contact with a flat sheet of latex (0.2 mm thickness) which covered the 10 mm diameter knife-edge ring support. The latex compensated for variations in geometric flatness and allowed small lateral displacements, thereby, minimizing friction and shear [28]. The disk-shaped

specimens were centrally loaded with a 4 mm diameter ball indenter at 1 mm/min. The central thickness of each fracture fragment was measured and the average resin thickness was determined for each disk.

The stress at failure was calculated using a multilayer analysis [22]. The position of the neutral plane (t_n) was determined using the flexural modulus of the dentin porcelain (E_1) – 64 GPa [29], the resin flexural moduli (E_2 – assessed experimentally below) and the thicknesses of the porcelain and resin layers (t_1 and t_2 , respectively) from Eq. (1).

$$t_n = \frac{E_1^*(t_1)^2 - E_2^*(t_2)^2}{2(E_1^*t_1 + E_2^*t_2)} \quad (1)$$

where

$$E_1^* = \frac{E_1}{1 - \nu_1} \quad (2a)$$

and

$$E_2^* = \frac{E_2}{1 - \nu_2} \quad (2b)$$

The Poisson's ratio (ν) of the bi-layered disk was determined from Eq. (3)

$$\nu = \frac{(\nu_1 t_1 + \nu_2 t_2)}{t_1 + t_2} \quad (3)$$

where ν_1 (0.25 [29]) and ν_2 (0.27 [30]) were the Poisson's ratios of the ceramic and resin. The biaxial flexure stress was calculated in the ceramic, for an axial position at the bonded interface ($z=0$) in the center of the bilayered specimens ($r=0$), the ceramic surface ($z=t_1$) and the resin surface ($z=-t_2$). The biaxial flexure stress for the monolayer disk-shaped specimens (Group A) was determined assuming $t_2=0$ using Eqs. (1)–(6).

$$\sigma = \frac{-3P(1+\nu)(z-t_n)}{2\pi(t_1+t_2)^3} \left[1 + 2 \ln \left(\frac{a}{b} \right) + \frac{1-\nu}{1+\nu} \left(1 - \frac{b^2}{2a^2} \right) \frac{a^2}{R^2} \right] \times \left[\frac{E_1^*(E_1^*t_1 + E_2^*t_2)(t_1+t_2)^3}{(E_1^*t_1^2)^2 + (E_2^*t_2^2)^2 + 2E_1^*E_2^*t_1t_2(2t_1^2 + 2t_2^2 + 3t_1t_2)} \right] \quad (4)$$

($0 \leq z \leq t_1$) and

$$\sigma = \frac{-3P(1+\nu)(z-t_n)}{2\pi(t_1+t_2)^3} \left[1 + 2 \ln \left(\frac{a}{b} \right) + \frac{1-\nu}{1+\nu} \left(1 - \frac{b^2}{2a^2} \right) \frac{a^2}{R^2} \right] \times \left[\frac{E_2^*(E_1^*t_1 + E_2^*t_2)(t_1+t_2)^3}{(E_1^*t_1^2)^2 + (E_2^*t_2^2)^2 + 2E_1^*E_2^*t_1t_2(2t_1^2 + 2t_2^2 + 3t_1t_2)} \right] \quad (5)$$

($-t_2 \leq z \leq 0$). P was the load at fracture, a and R were the radii of the supporting ring (5 mm) and specimen (6.1 mm), respectively. b was the radius of loading contact area determined using Eq. (6)

$$b = \frac{t_1 + t_2}{3} \quad (6)$$

The flexural moduli of the three resin-based materials (Venus Flow, Rely-X Veneer and Clearfil AP-X) were established using a three-point flexure testing assembly [31]. An open-ended aluminum split-mold (25 mm length, 2 mm width, 2 mm thickness) [32] was used to fabricate five nominally identical bar-shaped specimens for each resin. To ensure consistent specimen packing following resin placement, the mold was covered with polyester film and a glass slide with a 1 kg load was applied. The specimens were light irradiated through the Perspex base plate, at an output intensity of $380 \pm 21 \text{ mW/cm}^2$.

The length of the bar-shaped specimens (25 mm) exceeded the 13 mm light tip diameter and as a result the specimens were irradiated at successive overlapping incremental sites along the length of the bar-shaped specimens [32]. The light tip diameter was placed in contact with the polyester film at the center of the bar-shaped specimen and irradiated for 20 s. The light tip diameter was moved axially (both left and right) along the bar-shaped specimen such that a quarter of the light tip diameter overlapped with the previously exposed central area and were light irradiated for a further 20 s [33] such that the specimens were irradiated in three steps. The polyester strip was removed, the split-mold disassembled and the irradiation protocol repeated on the opposing surface of the bar-shaped specimen. Any excess residual flash was carefully removed and specimens with visually apparent surface defects or porosity inclusions were discarded. The bar-shaped specimens were stored dry in a dark incubator at 37°C for 24 h prior to testing in three-point flexure [33]. The specimens were placed on a support span (20 mm) and loaded centrally using the universal testing machine at 1 mm/min.

The flexural moduli were calculated from the linear elastic region of the stress–strain curve using Eq. (7).

$$E_B = \frac{PL^3}{4bd^3D} \quad (7)$$

where E_B is the flexural modulus, P is the load at failure, L is the support span (20 mm), b is the specimen width (mm), d is the specimen thickness (mm) and D is the deflection (mm) [31].

2.2. Analytical solution

An analytical methodological approach was undertaken to predict the biaxial flexure stresses determined by the closed form solutions (Eqs. (1)–(6)) under specific boundary conditions that reflected the experimental test parameters, namely the flexural modulus, resin-coating thickness and Poisson's ratio of the ceramic and resin-based materials employed along with the geometric dimensions of the test configuration. The biaxial flexure stress was predicted at an axial position ($r=0$) at the ceramic–resin interface ($z=0$) for an experimentally representative fixed load of 200 N. A cross-tabulation of the predicted biaxial flexure stresses was performed to demonstrate the effect of resin flexural modulus (varying from 4 to 18 GPa) and resin-coating thickness (10–150 μm).

2.3. Finite element analysis

The experimental ball-on-ring test was simulated using Finite Element Analysis (FEA) to verify the analytical prediction for biaxial flexure stress, thereby validating the experimental result. A two-dimensional axisymmetric finite element model of the resin-coated bilayered disk, in a ball-on-ring test configuration, was generated using a linear P-code FEA software, Pro/MECHANICA (Wildfire 2.0, Parametric Technology Corporation, Needham, MA, USA). The specific applied load in the finite element model (200 N) was consistent with the load used with the analytical solution, to determine the maximum principal stress in the center of the ceramic ($r=0$) at the ceramic–resin interface ($z=0$). Additionally, the boundary displacement constraints reflected the experimental test configuration whereby a frictionless ring support constraint was simulated by fixing the nodal axial (vertical displacement) at the ring support radius while allowing free transverse (horizontal) displacement. The ball load was applied to the outer porcelain surface over a central area equivalent to that determined by the equation for estimating the load contact area (Eq. (6)) with the contact regions excluded from the analysis. The resin–ceramic interface was assumed to be an idealized intimate bond throughout.

2.4. Statistical analysis

Analysis of group means was performed utilizing a two-way analysis of variance (ANOVA) ($P < 0.05$) where factors were resin-coating type at three levels (Clearfil AP-X, Rely-X Veneer, Venus Flow) and resin-coating thickness at three levels (50, 100 and 150 μm). Post hoc all paired Tukey tests were employed at a significance level of $P < 0.05$ (SPSS for Windows 12.0.1, SPSS Inc., Chicago, USA). A one-way ANOVA ($P < 0.05$) was used to determine the significance of reinforcement achieved by comparing the group means of specimens coated with 50 μm of different resin-based materials against the uncoated control. Subsequently the mean biaxial flexure strengths were plotted against the mean resin-coating thickness and a regression analysis to a theoretical 'zero' resin-coating thickness enabled estimation of the 'actual' magnitude of strengthening by comparison with the uncoated control.

3. Results

To facilitate the calculation of the stress at failure in the resin-coated specimens using the multilayer analysis [22], the flexural moduli and associated standard deviations of the resin-based materials investigated were 3.7 (0.2), 6.1 (0.3) and 12.6 (0.4) GPa for Venus Flow, Rely-X Veneer and Clearfil AP-X, respectively, under the experimental conditions investigated. A two-way ANOVA demonstrated that the magnitude of reinforcement achieved when ceramic disks were resin-coated was significantly influenced by resin-coating type ($P < 0.001$) and resin-coating thickness ($P = 0.013$), however, a significant interaction was observed between resin-coating type and thickness ($P = 0.048$). Post hoc analyses demonstrated a significant effect of resin-coating thickness on the observed biaxial flexure strength when Clearfil AP-X was used ($P = 0.001$) but

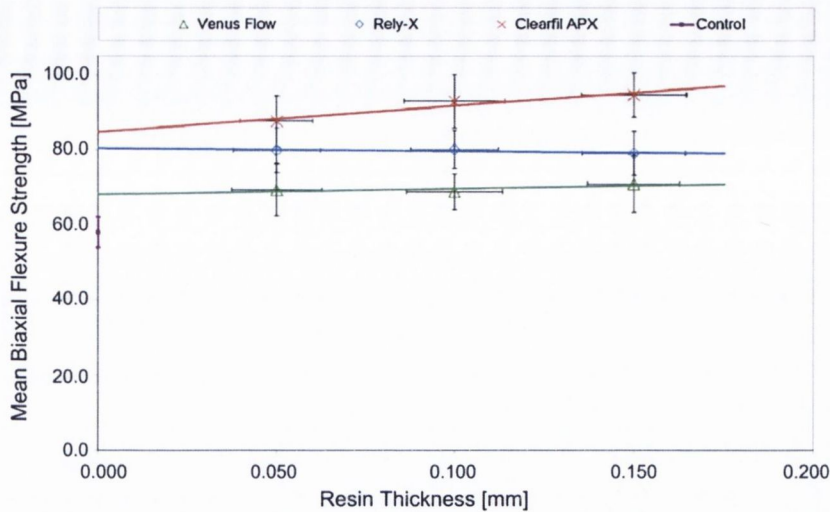


Fig. 1 – The plot of mean biaxial flexure strengths against resin-coating thickness for each resin-based material and respective standard deviations. Linear regression curves applied to the data and extrapolated to a ‘zero’ resin-coating thickness to allow for the determination of theoretical resin-strengthening relative to the un-coated control group (58 MPa) of 17% (68 MPa), 38% (80 MPa) and 47% (85 MPa) for the Venus Flow, Rely-X and Clearfil AP-X, respectively.

not when Rely-X Veneer ($P=0.881$) and Venus Flow ($P=0.338$) were employed.

The mean biaxial flexure strength and associated standard deviation of the alumina particle air abraded control (Group A) was 58.0 (4.0) MPa and a one-way ANOVA demonstrated that a 50 μm resin-coating thickness of all three resin-cements conferred significant reinforcement ($P<0.001$). Mean biaxial flexure strength was plotted against resin-coating thickness for each material (Fig. 1). Linear regression of the mean biaxial flexure strength values allowed extrapolation to a theoretical

‘zero’ resin-coating thickness and identified a 17, 38 and 47% biaxial flexure strength contribution when Venus Flow, Rely-X Veneer and Clearfil-AP-X were used, respectively.

3.1. Analytical solution

The cross-tabulation (Fig. 2) predicted, at the center-point radial position ($r=0$) and at the ceramic–resin interface ($z=0$) for an experimentally representative fixed load of 200 N, that increasing the flexural modulus of the resin-based material

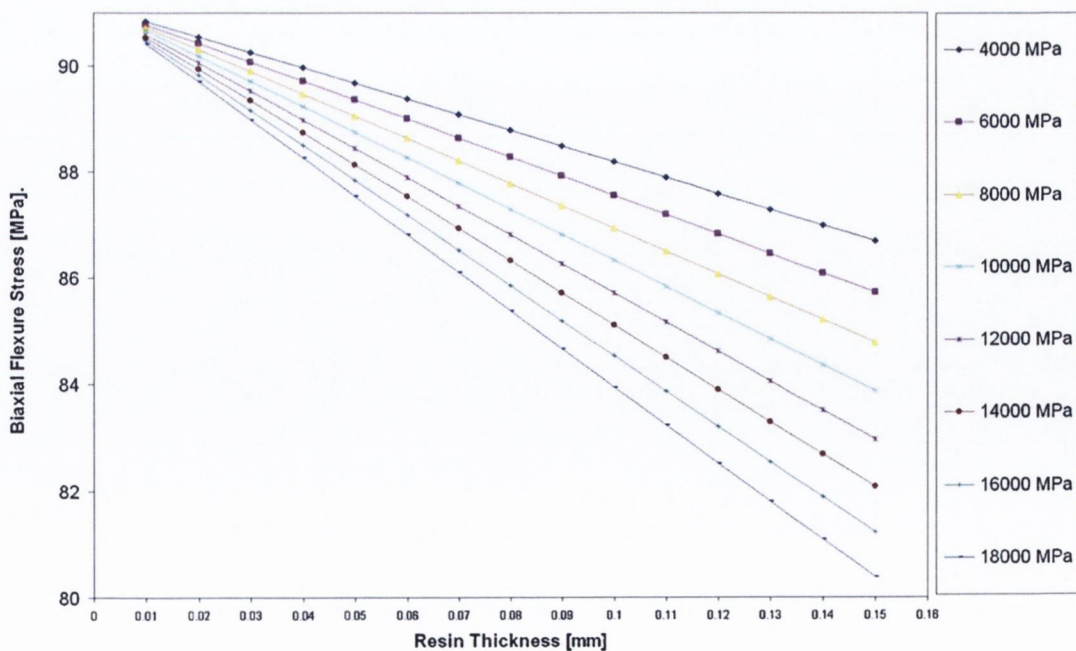


Fig. 2 – The cross-tabulation of biaxial flexure stress against thickness for varying resin-coating thickness (x -axis) and flexural moduli (legend), calculated in the ceramic disk-shaped specimen at the resin–ceramic interface ($z=0$) using the analytical solution. The applied load was 200 N, for a ceramic thickness of 1.895 mm and a ceramic flexural modulus of 64 GPa.

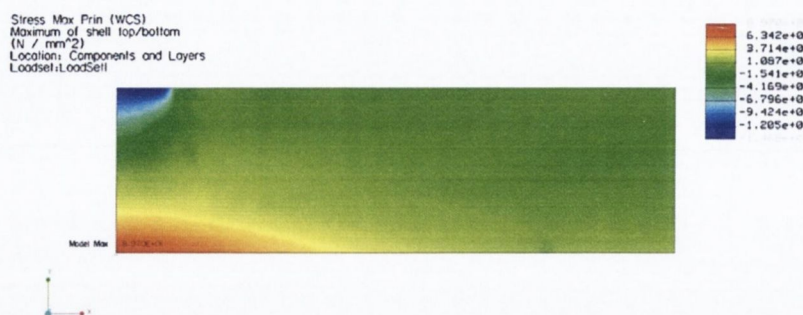


Fig. 3 – Two dimensional axisymmetric finite element analysis result showing a contour plot of the maximum principle stress in the ceramic disk-shaped specimen at the resin–ceramic interface ($z=0$), namely the bottom surface of the ceramic at the center of the specimen ($r=0$), for an applied load (200 N), a ceramic thickness of 1.895 mm and a ceramic flexural modulus of 64 GPa. The resin-coating thickness was 100 μm and a resin flexural modulus of 6.1 GPa (the resin-coating has been omitted from the contour plot of the maximum principle stress for clarity).

from 3.7 to 12.6 GPa reduces the resultant biaxial flexure stress. Additionally, increasing the resin-coating thickness from 10 to 150 μm results in the prediction that the biaxial flexure stress would also be reduced at $z=0$. The solution predicts that as the resin-coating thickness approaches a theoretical ‘zero’ thickness, the stress calculated at the resin–ceramic interface ($z=0$) for a fixed load is identical irrespective of resin-coating flexural modulus.

3.2. Finite element analysis

The finite element model predicted the stress generated in the ceramic at the center ($r=0$) of the resin-coated disk, at the resin–ceramic interface ($z=0$) under boundary conditions used in the analytical method, namely, a specific applied load (200 N), resin flexural modulus (12.6 GPa) and resin-coating thickness (100 μm). The maximum principal stress under these conditions was within 3.3% of the predicted analytical solution determined (by Eq. (4)) for the prescribed loading conditions (Fig. 3). The FEA was configured to use a maximum polynomial value of nine and convergence criteria of 2% for a specified measure parameter of maximum principal stress at an axial position ($r=0$) in the ceramic at the resin–ceramic interface ($z=0$).

4. Discussion

Clinical studies providing evidence for the use of adhesive technologies to support ceramics in the dental literature are limited [1,2,34] and the mechanical and rheological characteristics of the resin-coating have not been considered. Previously when testing resin-coated ceramic disks in biaxial flexure, the strength increase associated with resin-coating was identified to be linearly dependent on the flexural modulus of the selected resin-based material [9]. Tentatively it has been suggested that adhesive cementation of ceramic PLV or DBC restorations with higher flexural moduli resin-based materials could result in increased clinical performance [8,9].

Advanced analytical solutions derived by Hsueh et al. [22,23] allow the calculation of stress distributions at axial positions throughout biaxially loaded bilayered disk shaped

specimens [22]. Using the analytical solution to calculate the stress state in a resin-coated ceramic specimen predicts that a reduction in the biaxial flexure stress generated at the resin–ceramic interface on increasing the resin-coating thickness and/or increasing the flexural modulus of the resin-based material (Fig. 1). For the prescribed boundary conditions specified, the predicated principle stresses calculated using the analytical solution were confirmed within 3.3% by a two-dimensional axisymmetric finite element model. The experimental findings demonstrated significantly elevated biaxial flexure strengths for all three resin-based materials and therefore confirm that a resin-strengthening mechanism was operative in accordance with previous observations [8–10].

The flexural modulus of the resin-based materials was dependent on the resin filler content [14–16,35,36], as is generally accepted, that increasing the filler volume fraction of resin-based materials results in an increased in flexural modulus assuming integrity is maintained between the resin matrix and filler particles mediated through the silane coupling agent [11,37]. The flexural modulus values achieved in the current study were significantly reduced when compared with the flexural moduli reported previously, however, the proportional differences between the resin-based materials remained constant. The findings demonstrate the variability in the production and testing of bar-shaped resin specimens for the determination of flexural modulus. In the current study light irradiation through the Perspex base-plate produced an energy density of 7.6 J/cm² (380 mW/cm² for 20 s) compared with 14.8 J/cm² (740 mW/cm² for 20 s) used previously [10] and was responsible for the reduced values by influencing the degree of conversion [38].

The alumina particle air abraded dentin uncoated ceramic specimens had a mean biaxial flexure strength of 58.0 (4.0) MPa which is similar to values quoted in the literature (57.0 (4.0) [9], 58.0 (4.7) MPa [10] and 59 [4.2] MPa [18,27] when an identical alumina particle air abrasion regimen was used. The surface topography produced was conducive to adhesive bonding with resin-based materials and provided a consistent population of surface defects [27] reflected by a low standard deviation in the measured strength (<7%) [21]. The regression analyses of the resin-coating thickness data for the three resin-based materials was derived from unconstrained testing to estimate

the theoretical resin-strengthening when the resin bulk was eliminated. Extrapolating the linear regression curves thereby facilitated the quantification of the biaxial flexure strength at a theoretical 'zero' resin-coating thickness. Resin-coating significantly increased the mean biaxial flexure strength of the alumina particle air abraded dentin ceramic specimens by 17, 38 and 47% (Fig. 3) following coating with Venus Flow, Rely-X Veneer Cement and Clearfil AP-X, respectively. The increasing biaxial flexure strengths determined at a theoretical 'zero' resin-coating thickness with increasing resin flexural modulus suggests that magnitude of resin-strengthening was dependent on the flexural modulus of the resin-based material and the experimental hypothesis was accepted. While the mean biaxial flexure strengths were increased by 17, 38 and 47% on resin-coating with Venus Flow, Rely-X Veneer Cement and Clearfil AP-X, strength increases of 35, 62 and 107% were reported by Addison et al. [10] when employing the same resin-based materials. It should be noted that the bulk ceramic thickness was 1.9 mm in the current study compared with 0.9 mm reported by Addison et al. [10] which accounts for the variously reported strength increases on resin-coating when using Eqs. (1)–(6) but also highlights the significance of specimen rigidity on the magnitude of resin-reinforcement observed.

In the current study the resin flexural modulus had a variable influence on modulating the biaxial flexure strength when determined at different resin-coating thicknesses ranging from a theoretical 'zero' to 150 μm . The limited range of resin-coating thicknesses tested was clinically relevant and for Venus Flow and Rely-X veneer cement the magnitude of ceramic-reinforcement conferred was unaffected by increasing resin thickness. The observation suggests that the strengthening is mediated independently of resin bulk and is insensitive within the experimental conditions to the minor modifications of the tensile stress gradient from the neutral axis of bending to the resin–ceramic interface for the two materials. The proportional magnitude of strengthening between resin-based materials of different flexural moduli was variable with resin-coating thickness and suggests an over-emphasis previously of the importance of resin elasticity [9]. At a 150 μm resin-cement thickness the use of a material with a flexural modulus of 12.6 GPa compared with 6.1 GPa resulted in 18% additional strengthening of the ceramic, however, at a theoretical 'zero' resin-coating thickness the benefit was reduced to 6%. The interaction of resin-based materials possessing high filler loading with the ceramic surface will clearly differ from less viscous materials and the trend may potentially be the result of a lack of intimacy between the ceramic surface defects and the infiltrating resin-based material.

5. Conclusion

Experimentally, the flexural modulus and thickness of resin-based material used to cement DBC or PLV restorations have a significant impact on the magnitude of resin-strengthening observed. However, for resin-based materials with different flexural moduli the variability in the relationships between thickness and observed increases in the mean biaxial flexure

strength of the ceramic requires careful characterization in order to optimize clinical performance.

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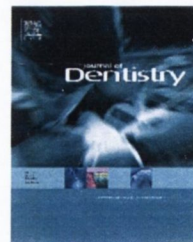
3.2 CAN A SODA LIME GLASS BE USED TO DEMONSTRATE HOW PATTERNS OF STRENGTH DEPENDENCE ARE INFLUENCED BY PRE-CEMENTATION AND RESIN-CEMENTATION VARIABLES?

P Hooi, O Addison, GJP Fleming (2013). Can a soda lime glass be used to demonstrate how patterns of strength dependence are influenced by pre-cementation and resin-cementation variables? *Journal of Dentistry*, 41:24-30.

The significant increase in BFS data determined at a theoretical ‘zero’ resin-coating thickness for RBC materials with different flexural moduli when compared with the uncoated controls further suggested that the resin-strengthening mechanism was mediated by a resin-ceramic hybrid layer (Manuscript 3.1). Furthermore, where the determinants on BFS to be examined are sensitive to surface and bulk defects, any strength test employed to examine dental ceramic materials is limited by the observed variability in measured strength which is introduced during specimen preparation due to the inclusion of surface flaws and bulk defects (Gill, 1932; Fleming et al., 2000). An analogue material for dentine porcelain was proposed in the current study to reduce the variability associated with specimen preparation. Soda lime glass was selected as the analogue material for dentine porcelain, from which reliable, bulk-defect-free and nominally identical samples were consistently produced. Visual inspection of the transparent soda lime glass to screen for surface and bulk defects was possible prior to preparation and testing. The current study investigated the influence of pre-cementation operative variables (alumina particle air abrasion and HF acid-etching) and resin-cement coating on the BFS of the soda lime glass analogue for dentine porcelain. Manuscript 3.2 represents a study to investigate the hypothesis that the soda lime glass analogue material would demonstrate the same patterns of strength dependence influenced by pre-cementation parameters and resin-cementation variables characterised previously (Addison et al., 2007c; Fleming and Addison, 2009).

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Can a soda-lime glass be used to demonstrate how patterns of strength dependence are influenced by pre-cementation and resin-cementation variables?

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ABSTRACT

Objectives: To determine how the variability in biaxial flexure strength of a soda-lime glass analogue for a PLV and DBC material was influenced by precementation operative variables and following resin-cement coating.

Methods: The flexural modulus of a transparent soda-lime glass was determined by longitudinally sectioning into rectangular bar-shaped specimens and the flexural moduli of three resin-based materials (Venus Flow, Rely-X Veneer and Clearfil Majesty Posterior) was also determined. Disc shaped soda-lime glass specimens ($n = 240$) were divided into ten groups and were alumina particle air abraded, hydrofluoric (HF) acid-etched and resin-cement coated prior to biaxial flexure strength testing. Sample sets were profilometrically evaluated to determine the surface texture. One-way analyses of variance (ANOVA) and post hoc all paired Tukey tests were performed at a significance level of $P < 0.05$. The mean biaxial flexure strengths were plotted against resin-coating thickness and a regression analysis enabled estimation of the 'actual' magnitude of strengthening.

Results: The mean three-point flexural modulus of the soda-lime glass was 40.0 (1.0) GPa and the Venus Flow, Rely-X Veneer and Clearfil Majesty Posterior were 3.0 (0.2) GPa, 6.0 (0.2) GPa and 14.8 (1.6) GPa, respectively. At a theoretical 'zero' resin-coating thickness an increase in biaxial flexure strength of 20.1% (63.2 MPa), 30.8% (68.8 MPa) and 36.3% (71.7 MPa), respectively was evident compared with the control (52.6 (5.5) MPa).

Conclusions: Disc-shaped specimens cut from round stock facilitated rapid fabrication of discs with uniform surface condition and demonstrated strength dependence was influenced by precementation parameters and resin-cementation variables.

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1. Introduction

The clinical success of resin-luted porcelain laminate veneer (PLV) or dentine bonded crown (DBC) restorations is remarkable¹ and is attributed to a synergism² between the tooth preparation and the low strength ceramic which is conferred

by the resin-based luting material. There is little doubt that the long-term comprehensive prospective clinical study of 1444 Dicor glass-ceramic crowns by Malament and Socransky¹ is the 'gold standard' in terms of identifying the effect of resin-cementation on improved clinical performance. Unfortunately, Dicor was obsolete when the 14-year results of the study were published in 1999. Given the number of new

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glass-ceramic and dispersion strengthened ceramics introduced to the dental market in the last 30 years, it would be unreasonable to perform similar long-term comprehensive prospective clinical studies on each ceramic type marketed. As a result, *in vitro* research methodologies have been devised to aid practitioners and materials scientists in assessing the appropriate ceramic material and cementation technique to improve clinical performance. When assessing the suitability of *in vitro* research methodologies a guest editorial in 1995 entitled 'The First Three Questions'³ suggested that 'in any discipline, for any project, whatever the motivation' the fundamental questions to be asked were: What do you really want to know? What do you want to measure? and What are you going to measure?³

To provide clinical guidance and maximise the clinical performance of resin-luted PLV or DBC restorations, axisymmetric finite-element analysis of molar crowns⁴ and quantitative fractography of failed restorations^{5–8} identified the failure mode to be tensile and the fracture origin to be controlled by a critical flaw on the inner surface of the crown.^{4–8} Therefore the suitability of an *in vitro* research methodology to investigate the underlying resin-strengthening mechanisms of PLV or DBC restorations is the replication of the clinically observed failure mode and the fracture origin.^{9–11} One test considered was the 'crunch-the-crown' testing methodology, however, failure was initiated at the loading contact zone on the external surface of the crown^{5,12} and therefore the clinically observed failure mode or fracture origin were not replicated in the testing protocol. In addition, the 'crunch-the-crown' test returns only the load at which failure is deemed to have occurred which is a scalar quantity that cannot be compared and does not inform the investigator about the resultant stress state within the complex geometry of the specimen which causes fracture. As a result of these inadequacies the 'crunch-the-crown' test has essentially been made redundant from the materials science testing armamentarium.¹³ Kelly et al.¹⁴ went further recently when after criticising the appropriateness of 'crunch-the-crown' protocols the authors stated they planned to engage editors to take an 'ethical stand against the publication of studies using "crunch-the-crown" protocols in particular'.¹⁴ Testing in biaxial flexure replicates the clinically observed failure mode and fracture origin of PLV or DBC restorations and the simplified disc-shaped specimen has a surface area to volume approximating such restorations. Using the biaxial flexure strength testing methodology, strengthening of PLV/DBC ceramic materials with resin-cement has been shown to be insensitive to defect size⁹ but sensitive to macroscopic surface roughness¹⁰ and resin-cement elasticity.¹¹ Therefore, the traditionally proposed theories of crack healing¹⁵ or the crack closure stresses secondary to polymerisation shrinkage¹⁶ have been demonstrated to incompletely explain the observed strengthening.^{9–11} One limitation of all strength testing methodologies is in ceramic sample preparation where variability in the measured strength can be introduced during fabrication^{17,18} through sintering or heat-pressing¹⁹ and even CAD-CAM routes²⁰ which is further influenced by the technical skill of the operator.^{18,21} To further understand and optimise the strengthening of PLV and DBC materials by resin-cements it would be useful to identify a substrate from

which reliable, bulk-defect-free nominally identical samples may be produced. As the important effect to be observed is surface dominated,^{22,23} the fracture toughness of the material is less important, however, the dependence of resin-strengthening on surface effects must be analogous to the previously reported data.^{24,25}

The aim of the current study was to determine how the variability in biaxial flexure strength of an analogue for a PLV and DBC material was influenced by pre-cementation operative variables (alumina particle air abrasion and hydrofluoric (HF) acid-etching) and following resin-cement coating. The hypothesis tested was that the analogue material would demonstrate the same patterns of strength dependence influenced by pre-cementation parameters and resin-cementation variables characterised previously with feldspathic and glass-ceramic materials.

2. Materials and methods

2.1. Soda-lime glass flexural modulus

A transparent soda-lime glass (Glass 8337B, SCHOTT-Rohr glas GmbH, Mitterteich, Germany) was received from the manufacturers in a rod form (1.5 m length, 11.6 mm diameter). The flexural modulus of the soda-lime glass was determined by longitudinally sectioning twelve rectangular bar-shaped specimens (25 mm length, 2 mm width, 2 mm thickness) using a precision circular saw (Isomet[®] Low Speed Saw, Buehler, Lake Bluff, IL, USA) coupled with a diamond wafering blade (15 HC Diamond, Buehler, Lake Bluff, IL, USA) rotating at 170 rpm under water irrigation. The bar-shaped specimens were visually inspected for surface defects, with any residual flash carefully removed by hand-lapping using P400 silicon carbide abrasive paper (Buehler, Lake Bluff, IL, USA) under water lubrication and light finger pressure. The bar-shaped specimens were air-dried and stored in a desiccator at $23 \pm 1^\circ\text{C}$ for 24 h prior to testing and analysis.

The flexural modulus of the soda-lime glass was determined by loading the twelve rectangular bar-shaped specimens to failure in a three-point flexure testing configuration. The specimens were placed individually on a 20 mm support span and loaded centrally at 1 mm/min using a universal testing machine (Instron Model 5565, Instron Ltd., High Wycombe, UK). The slope of the linear elastic region of the load-deflection curves were determined and flexural moduli calculated using Eq. (1).

$$E_B = \frac{L^3}{4bd^3} \frac{\Delta P}{\Delta D} \quad (1)$$

where E_B is the flexural modulus, L the support span (20 mm), b the specimen width (mm), d the specimen thickness (mm) and $\Delta P/\Delta D$ is the slope of the linear elastic region of the load-deflection curve.²⁶

2.2. Resin flexural modulus

The flexural moduli of the three resin-based materials (Venus Flow (Heraeus Kulzer GmbH, Hanau, Germany), Rely-X Veneer (3M ESPE, St. Paul, MN, USA) and Clearfil Majesty Posterior

(Kuraray, Kurashiki, Japan)) were also determined. An open-ended aluminium split-mould (25 mm length, 2 mm width, 2 mm thickness)^{27,28} was used to fabricate five nominally identical bar-shaped specimens for each of the three resin-based materials. A polyester film (Mylar™, DuPont Teijinn Films, Middlesbrough, UK) was placed in contact with the resin-based materials and a glass slide was then placed over the mould to ensure consistent specimen packing. The specimens were light irradiated (Optilux 501, SDS Kerr, Danbury, CT, USA) through the glass slide and polyester film, for 20 s with a 13 mm light tip diameter. The output intensity of the Optilux 501 was determined to be 480 ± 20 mW/cm² using the radiometer incorporated within the light curing unit.

The length of the bar-shaped specimens (25 mm) exceeded the light tip diameter (13 mm) and the specimens were irradiated at successive overlapping incremental locations along the length of the bar-shaped specimens.²⁶ The light tip diameter was placed in contact with the polyester film at the centre of the bar-shaped specimen and irradiated for 20 s. The light tip diameter was moved longitudinally (both left and right) along the bar-shaped specimen such that a quarter of the light tip diameter overlapped with the previously exposed central area and light irradiated for a further 20 s²⁸ such that the specimens were irradiated in three steps. The polyester strip was removed, the split-mould disassembled and the irradiation protocol repeated on the opposing surface of the bar-shaped specimen. Any residual excess residual flash was carefully removed and specimens with visually apparent surface defects or porosity inclusions were discarded. The bar-shaped specimens were stored dry in a dark incubator at 37 °C for 24 h prior to testing in three-point flexure.²⁸ The specimens were placed on a support span (20 mm) and loaded centrally using the universal testing machine at 1 mm/min and the flexural moduli calculated from the linear elastic region of the stress-strain curve using Eq. (1).²⁶

2.3. Specimen preparation

The glass rod was transversely sectioned (at 1 mm intervals) using the precision circular saw coupled with the diamond wafering blade operating at 170 rpm under water irrigation to produce 288 nominally identical disc-shaped specimens (11.6 mm diameter). Immediately after sectioning, the disc-shaped specimens were dried with oil free compressed air. The nominally identical disc-shaped specimens were randomly allocated into twelve groups (Groups A–L, respectively). Each specimen group contained 24 specimens and Group A specimens were stored in a desiccator prior to testing and analysis as the ‘as-cut’ control.

The remaining 264 disc-shaped specimens (Groups B–L) were alumina particle air abraded on one surface with 50 µm alumina particles using a pressure of 4 bar for 5 s (Eco Dry Oxide System, Dentalfarm, Torino, Italy). The nozzle of the alumina particle air abrader was maintained orthogonal to the disc-shaped specimen surface at a distance of 2 cm and passed over the surface in a smooth linear sweeping motion to minimise excessive localised abrasion.²² Twenty-four of the disc-shaped specimens (Groups B) were stored in the desiccator prior to testing and analysis to act as the ‘alumina particle air abraded’ control.

The 240 remaining disc-shaped specimens (Groups C–L) were acid-etched for 120 s using 9.6% HF acid (Porcelain Etch Gel (Lot 101025, Pulpdent Corporation, Watertown, MA, USA)). The specimens were rinsed for 10 s with distilled water to remove the acid completely, prior to drying immediately with oil free compressed air and 24 specimens (Group C) were stored in the desiccator prior to testing and analysis to act as the ‘acid-etched’ control.

2.4. Profilometric analysis

A random sample set of three disc-shaped specimens from each control group (Groups A–C) were profilometrically evaluated to determine the surface roughness (R_a -value) and surface waviness (W_a -value) of the ‘as cut’, alumina particle air abraded and acid-etched control group specimens. Each disc-shaped specimen was placed on a stable custom-made multi-axial levelling device during profilometric analysis.^{13,19} Profilometric scanning was performed with an inductive gauge conisphere diamond stylus tip (2 µm radius) using a Talysurf CLI 2000 (Taylor Hobson Ltd., Leicester, UK). The displacement measurements were performed at a measurement velocity of 3 mm/s and an applied load of 0.75 mN across a rectangular area of 10 mm² (10 mm length and 1 mm width) that included the centre and extending to the periphery of the disc-shaped specimen. The scan resolution was 3334 points (x-direction) and 334 traces (y-direction) using a 3 µm step size (x- and y-direction). The mean R_a -value and mean W_a -value were determined by extracting and averaging 20 traces in the y-direction.

2.5. Resin-coating

The central thickness of the remaining 216 disc-shaped specimens (Groups D–L) prior to preparation for resin-coating was measured using a screw gauge micrometre reading to 10 µm (Mitutoyo Corporation, Tokyo, Japan). The micrometre was fitted with a ball-nose adapter on the spindle tip which was placed in contact with specimen during assessment of the disc thickness. The alumina particle air abraded and HF acid-etched surface of the disc-shaped specimens were silane treated with 3-methacryloxypropyl-trimethoxysilane (Rely-X™ Ceramic Primer, 3M ESPE, St. Paul, MN, USA) and allowed to air dry, in accordance with the manufacturers recommendations. The disc-shaped specimens were resin-coated with Venus Flow at resin-coating thicknesses of 50 ± 25 µm (Group D), 100 ± 25 µm (Group E) and 150 ± 25 µm (Group F) and Rely-X Veneer at resin-coating thicknesses of 50 ± 25 µm (Group G), 100 ± 25 µm (Group H) and 150 ± 25 µm (Group I). The remaining specimens were resin-coated with Clearfil Majesty Posterior at thicknesses of 100 ± 25 µm (Group J), 150 ± 25 µm (Group K) and 200 ± 25 µm (Group L). It was not possible to achieve consistent thicknesses of Clearfil Majesty Posterior below 100 µm due to the increased filler loading and associated handling difficulties caused by the increased viscosity resin. The authors attempted to reduce the viscosity of the Clearfil Majesty Posterior by heating to 50 °C on a glass-slide which enabled resin-coating thicknesses in the range of 100–200 µm. The resin-coated disc-shaped specimens were allowed to cool to ambient temperature before light irradiation.

The resin-coated disc-shaped specimens were light irradiated using the Optilux 501 light curing unit through a 1 mm thick glass slide and polyester film, for 20 s with the 13 mm light tip diameter. The output intensity of the Optilux 501 was $480 \pm 20 \text{ mW/cm}^2$ at the resin surface when evaluated using the radiometer incorporated within the light curing unit. Following irradiation the central thickness of the resin-coated disc-shaped specimens were measured using the screw gauge micrometre and the resin-coating thicknesses were determined.

2.6. Biaxial flexure strength testing

A ball-on-ring test configuration was used to determine the failure loads (N) of the controls (Groups A–C) and the resin-coated (Groups D–L) disc-shaped specimens following 24 h storage in a desiccator. The control groups were tested with the ‘as cut’, alumina particle air abraded and acid-etched surfaces placed in contact with a flat sheet of latex (0.2 mm thickness) which covered the 10 mm diameter knife-edge ring support and centrally loaded with a 4 mm diameter ball indenter at 1 mm/min. The resin-coated surface of the disc-shaped specimens (Groups D–L) were placed in contact with the latex and knife-edge ring support and loaded centrally with the ball indenter at 1 mm/min. The latex compensated for geometric variation and permitted small lateral displacements to minimise friction and shear.

The biaxial flexure stress at failure was calculated using a bilayer analytical solution.²⁹ The position of the neutral plane (t_n) was calculated using the experimentally determined flexural modulus of the soda-lime glass (E_1), the resin flexural moduli (E_2) and the thicknesses of the glass and resin-coating (t_1 and t_2 , respectively) from Eq. (2).

$$t_n = \frac{E_1^*(t_1)^2 - E_2^*(t_2)^2}{2(E_1^*t_1 + E_2^*t_2)} \quad (2)$$

where

$$E_1^* = \frac{E_1}{1 - \nu_1} \quad (2a)$$

and

$$E_2^* = \frac{E_2}{1 - \nu_2} \quad (2b)$$

The Poisson's ratio (ν) of the bi-layered disc was determined from Eq. (3).

$$\nu = \frac{(\nu_1 t_1 + \nu_2 t_2)}{t_1 + t_2} \quad (3)$$

where ν_1 (0.22³⁰) and ν_2 (0.27³¹) were the Poisson's ratios of the glass and resins, respectively. The biaxial flexure stress was calculated in the soda-lime glass at the bonded interface ($z = 0$) in the centre of the bilayered specimens ($r = 0$). The biaxial flexure stress for the monolayer disc-shaped specimens (Group C) was determined by setting $t_2 = 0$ using Eqs. (2)–(6).

$$\sigma = \frac{-3P(1 + \nu)(z - t_n)}{2\pi(t_1 + t_2)^3} \left[1 + 2\ln\left(\frac{a}{b}\right) + \frac{1 - \nu}{1 + \nu} \left(1 - \frac{b^2}{2a^2} \right) \frac{a^2}{R^2} \right] \times \left[\frac{E_1^*(E_1^*t_1 + E_2^*t_2)(t_1 + t_2)^3}{(E_1^*t_1^2)^2 + (E_2^*t_2^2)^2 + 2E_1^*E_2^*t_1t_2(2t_1^2 + 2t_2^2 + 3t_1t_2)} \right] \times (0 \leq z \leq t_1) \quad (4)$$

and

$$\sigma = \frac{-3P(1 + \nu)(z - t_n)}{2\pi(t_1 + t_2)^3} \left[1 + 2\ln\left(\frac{a}{b}\right) + \frac{1 - \nu}{1 + \nu} \left(1 - \frac{b^2}{2a^2} \right) \frac{a^2}{R^2} \right] \times \left[\frac{E_2^*(E_1^*t_1 + E_2^*t_2)(t_1 + t_2)^3}{(E_1^*t_1^2)^2 + (E_2^*t_2^2)^2 + 2E_1^*E_2^*t_1t_2(2t_1^2 + 2t_2^2 + 3t_1t_2)} \right] (-t_2 \leq z \leq 0) \quad (5)$$

P was the load at fracture, a and R were the radii of the supporting ring (5 mm) and specimen (5.8 mm), respectively. b was the radius of loading contact area determined using Eq. (6).

$$b = \frac{t_1 + t_2}{3} \quad (6)$$

2.7. Statistical analysis

One-way analyses of variance (ANOVA) and *post hoc* all paired Tukey tests were performed at a significance level of $P < 0.05$ (SPSS for Windows 12.0.1, SPSS Inc., Chicago, USA) and used to compare the mean flexural moduli of the bar-shaped specimens for the three resin-based materials, the mean BFS, mean R_a -value and mean W_a -value of the control groups (Groups A–C). Analysis of the group biaxial flexure strength means (Groups C–L) was performed utilising a two-way ANOVA ($P < 0.05$) where factors were resin-coating material and resin-coating thickness. *Post hoc* all paired Tukey tests were performed at $P < 0.05$. The mean biaxial flexure strengths were plotted against the mean resin-coating thickness and a regression analysis to a theoretical ‘zero’ resin-coating thickness enabled estimation of the ‘actual’ magnitude of strengthening by comparison with the uncoated control.

3. Results

The mean three-point flexural modulus and associated standard deviation of the twelve bar-shaped soda-lime glass specimens was 40.0 (1.0) GPa. The mean three-point flexural moduli and associated standard deviations of the Venus Flow, Rely-X Veneer and Clearfil Majesty Posterior bar-shaped specimens were 3.0 (0.2) GPa, 6.0 (0.2) GPa and 14.8 (1.6) GPa, respectively. The one-way ANOVA and paired Tukey test comparisons identified significant differences ($P < 0.001$) between the mean elastic moduli of the three resin groups.

The mean biaxial flexure strength and associated standard deviation of the ‘as-cut’ (Group A) discs was 118.7 (13.8) MPa with a mean R_a -value and associated standard deviation of 0.68 (0.29) μm and a mean W_a -value and associated standard deviation of 0.76 (0.26) μm . Alumina particle air abrasion (Group B) resulted in a significant reduction ($P < 0.001$) in the mean BFS to 44.4 (4.8) MPa with a significant increase in the mean R_a -value to 3.26 (0.166) μm ($P < 0.001$) and the mean W_a -value to 5.726 (1.30) μm ($P < 0.001$). HF acid-etching of the alumina particle air abraded surface (Group C) resulted in a significant increase ($P = 0.006$) in the mean BFS to 52.6 (5.5) MPa compared with Group B specimens with no difference ($P = 1.000$) in mean R_a -value (3.26 (0.17) μm) but a significant increase ($P < 0.001$) in the mean W_a -value to 8.15 (2.93) μm was identified.

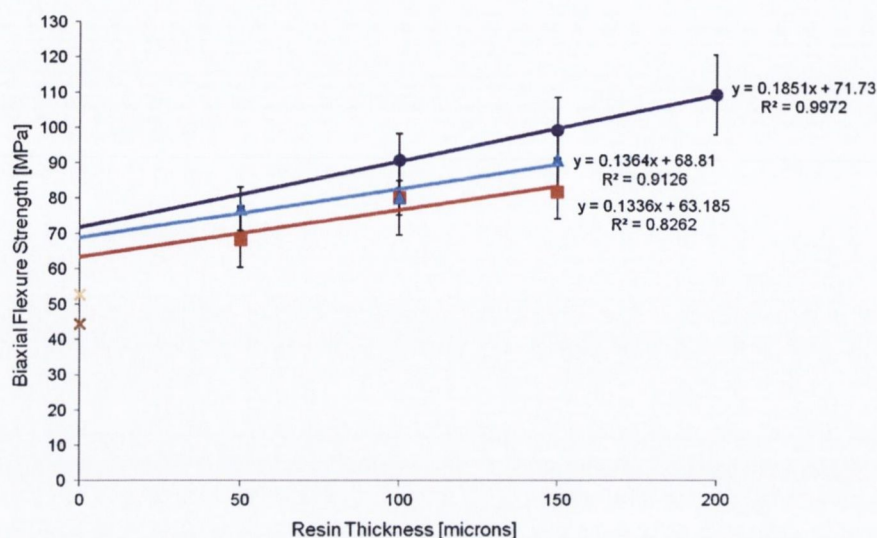


Fig. 1 – The plot of mean biaxial flexure strengths against resin-coating thickness for each resin-based material and respective standard deviations. Linear regression curves applied to the data and extrapolated to a ‘zero’ resin-coating thickness to allow for the determination of theoretical resin-strengthening relative to the uncoated control groups (44.4 MPa for alumina particle air abrasion; 52.6 MPa for alumina particle air abrasion and HF acid-etched). Theoretical resin-strengthening of 20% (63.2 MPa), 31% (68.8 MPa) and 36% (71.7 MPa) were evident for the Venus Flow, Rely-X Veneer and Clearfil Majesty Posterior, respectively.

The mean biaxial flexure strength of the uncoated (Group C) disc-shaped specimens was significantly increased (all $P < 0.001$) when resin-coated with Venus Flow ($50 \pm 25 \mu\text{m}$ (68.1 (7.9) MPa; Group D), $100 \pm 25 \mu\text{m}$ (80.1 (10.8) MPa; Group E) and $150 \pm 25 \mu\text{m}$ (81.5 (7.5) MPa; Group F)), Rely-X Veneer ($50 \pm 25 \mu\text{m}$ (76.8 (6.4) MPa; Group G), $100 \pm 25 \mu\text{m}$ (80.0 (5.0) MPa; Group H) and $150 \pm 25 \mu\text{m}$ (90.5 (9.1) MPa; Group I) and Clearfil Majesty Posterior ($100 \pm 25 \mu\text{m}$ (90.5 (7.7) MPa; Group J), $150 \pm 25 \mu\text{m}$ (98.9 (9.3) MPa; Group K) and $200 \pm 25 \mu\text{m}$ (109.0 (11.3) MPa; Group L).

Extrapolation of the linear regression curves (Fig. 1) applied to the mean biaxial flexure strength data for the individual resin-based materials investigated at a theoretical ‘zero’ resin-coating thickness provided an increase in biaxial flexure strength of 20.1% (63.2 MPa), 30.8% (68.8 MPa) and 36.3% (71.7 MPa) for the Venus Flow, Rely-X Veneer Cement and Clearfil Majesty Posterior, respectively compared with the uncoated (Group C) control specimens (52.6 (5.5) MPa).

4. Discussion

The three-point flexural modulus of the soda-lime glass was 40.0 (1.0) GPa which is close to the lower range limit for low strength PLV/DBC materials and suggested the possibility of employing it as an experimental analogue. The elastic moduli of the resin-based composite materials was correlated linearly ($R^2 = 0.9938$) with the filler loadings, namely 60, 66 and 92 wt% for Venus Flow (Groups D–F), Rely-X Veneer (Groups G–I) and Clearfil Majesty Posterior (Groups J–L), respectively.

The mean biaxial flexure strength of the ‘as-cut’ soda-lime glass discs represented the maximum strength through the

series of observations and accordingly the stress intensity of the surface defect was assumed to be at a minimum. Profilometric analyses of the surfaces (R_a -value of 0.68 (0.29) μm and W_a -value of 0.76 (0.26) μm) confirmed a lack of macroscopic surface texture and it is implied from the strength data that the surface defect size was therefore similarly small. The strength of dental ceramics used for PLV/DBC restorations is dependent on the presence of surface flaws which act as stress concentrators³² and clinically such restorations will be roughened through particle air abrasion during de-vestment and/or to increase the surface area available through micromechanical interlocking with the resin-cement.^{33,34} In the current study, surface modification of the ‘as-cut’ soda-lime glass disc-shaped specimens with alumina particle air abrasion modified the surface topography and introduced a different surface defect population manifesting a significant reduction in the mean biaxial flexure strength ($P < 0.001$). It has been previously proposed that the macroscopic surface roughness acts as a cumulative stress concentrator on critical flaws,³⁵ however, alumina particle air abrasion has been shown to not only modify the macroscopic surface of glassy dental ceramic materials (similarly observed in this study)²² but also create surface and sub-surface deformation³⁶ outside of the measurement scope of the metrological equipment used. Although the lack of explicit knowledge of the state of the critical defect is not ideal, the observations for the soda-lime glass investigated are, however, consistent with observations on feldspathic and glass ceramics where similar phenomena are likely.

Subsequent HF acid-etching of the particle air abraded surface, failed to modify the surface roughness (R_a -value) significantly, but a significant reduction in surface waviness (W_a -value) was demonstrated whereby the amplitude of the

larger surface features was reduced and superimposed over longer wavelengths. This finding is consistent with the investigation of Addison et al.²³ where a favourable HF acid-etching regime (concentration and etching time) was identified to enhance the mechanical performance of a PLV material. In addition, 'crack-blunting' was assumed to have occurred at the level of the critical crack tip as observed previously in the ceramic literature.^{37–39} As strengthening of soda-lime glass following HF-acid etching is well reported,^{37,38,40} we herein suggest that as an analogue material to evaluate resin-cement strengthening a surface state can be carefully introduced which closely matches that observed on PLV/DBC dental ceramic materials.

Resin-strengthening of PLV/DBC materials has been shown to be dependent upon the wetting of the resident surface texture by the resin,¹⁰ thereby creating an interpenetrated ceramic-resin composite hybrid layer.^{11,24} A significant observation has been that the strength in *in vitro* static tests can be correlated with the elastic modulus of resin-based composite luting material and has been translated into clinical practice by clinicians using conventional stiffer resin-based composite materials as PLV luting agents by modifying the resin viscosity through pre-warming.¹¹ In this study, when linear regression curves were applied through the mean biaxial flexure strengths of each resin group, across their respective resin-coating thicknesses, and extrapolated to a theoretical 'zero' resin-coating thickness the magnitude of resin-strengthening at a theoretical 'zero' resin thickness could be quantified (Fig. 1). The respective percentage increases in biaxial flexure strengths at a theoretical 'zero' resin-coating thickness were 20%, 31% and 36% for the Venus Flow, Rely-X Veneer and Clearfil Majesty Posterior, respectively. Interestingly, a similar pattern of strengthening was observed by Addison et al.¹¹ on a feldspathic ceramic following resin coating, namely 17%, 38% and 47% at a theoretical zero resin-thickness for the same three materials, respectively.¹¹ Therefore the tested hypothesis that the analogue material (soda-lime glass) would demonstrate the same patterns of strength dependence influenced by pre-cementation parameters and resin-cementation variables characterised previously with feldspathic and glass-ceramic materials was accepted.

There are several benefits of using soda-lime glass discs cut from a prefabricated round stock instead of manually condensing porcelain disc-shaped specimens^{17,18} most notably the inclusion of porosity which may not be apparent on visual inspection.¹⁸ Additionally, the sintering process itself can induce residual thermal stresses^{13,19} which may cause geometric deformation and effectively reduce the reliability of the biaxial flexure strength data.¹³

5. Conclusions

This study has demonstrated the suitability of soda-lime glass as a replacement substrate for porcelain disc-shaped specimens loaded in a ball-on-ring testing configuration. Additionally, precision cutting the disc-shaped specimens from round stock facilitated the rapid fabrication of disc-shaped specimens with consistently uniform surface condition, sample geometry and size.

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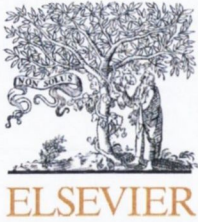
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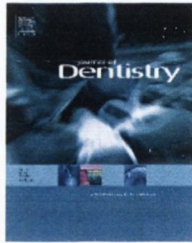
3.3 TESTING RATE AND CEMENTATION SEATING LOAD EFFECTS ON RESIN-STRENGTHENING OF A DENTAL PORCELAIN ANALOGUE.

P Hooi, O Addison, GJP Fleming (2013). Testing rate and cementation seating load effects on resin-strengthening of a dental porcelain analogue. *Journal of Dentistry*, 41:514-20.

The soda lime glass material validated as an analogue for dental porcelain in (Manuscript 3.2) was employed in the current study to investigate the influence of testing rate and cementation seating load on the magnitude and pattern of resin-strengthening. Load to failure testing of dental ceramic materials is typically performed at a constant standardised testing rate of 1 ± 0.5 mm/min (ISO 6872:2000) but a more appropriate loading regimen would employ different testing rates which are clinically representative of the human masticatory cycle. In the context of rate-dependent failure, water is known to be the primary determinant of rate-dependent fracture on the external surface of monolithic dental ceramic restorations (Lee et al., 2002) which is exposed to the moist oral environment. The susceptibility of soda lime glass to moisture-assisted slow crack growth makes this material eminently suitable in examining rate-dependent fracture. The influence of testing rate on the BFS of resin-coated soda lime glass disc-shaped specimens has not been reported in the literature. Furthermore, it has been demonstrated that seating load parameters employed during resin-coating significantly impacted on the BFS of a resin-coated leucite reinforced glass-ceramic material and the interaction between factors was complex (Addison et al., 2010). The influence of the seating load on resin-strengthening over a range of testing rates has not been examined. The current study was designed to investigate the influence of different testing rates on the BFS of soda lime glass disc-shaped specimens and the pattern of resin-strengthening where the resin-coating was applied with different seating loads in ambient air.

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Testing rate and cementation seating load effects on resin-strengthening of a dental porcelain analogue

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ABSTRACT

Objectives: To determine the resin-strengthening dependence of a soda-lime-glass analogue for dental porcelain as a function of biaxial flexure strength (BFS), test crosshead rate and cementation seating load.

Methods: Disc-shaped soda-lime glass specimens were divided into twelve groups ($n = 24$), alumina particle air abraded and hydrofluoric acid-etched. Specimens (Groups A–D) were stored in a desiccator prior to testing at crosshead rates of 0.01, 0.1, 1 and 10 mm/min, respectively. The remaining specimens were silane treated, Rely-X Veneer resin-coated with a seating load of 5 N (Groups E–H) and 30 N (Groups I–L) prior to light irradiation at 480 ± 20 mW/cm², 24 h dry storage and BFS testing at 0.01, 0.1, 1 and 10 mm/min, respectively. A linear logarithmic regression curve was fit to the raw data to elucidate static fatigue effects of the soda-lime-glass. Analysis of group means was performed utilising a general linear model univariate analysis and post hoc all paired Tukey tests ($P < 0.05$).

Results: The linear logarithmic regression curve demonstrated the static fatigue effects of the soda-lime-glass analogue. Rely-X Veneer resin-coating (Groups E–L) resulted in significant increases in the mean BFS data for all crosshead rates examined (all $P < 0.001$). However, the pattern of rate dependence effects on resin-cementation deviated from the log relationship observed with the uncoated controls.

Conclusion: This study further highlights that when slow crack growth is simulated during testing, valuable insights into the significant modification of a hereto well described phenomenon such as resin-strengthening mediated by the resin-ceramic hybrid layer is provided.

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1. Introduction

Failure of all-ceramic restorations due to fracture, leading to partial or ultimately total restoration loss, continues to compromise clinical treatment outcomes in dentistry.¹ The fracture origin for many classes of all-ceramic dental restorations was identified, using quantitative fractography in failed restorations^{2–4} and finite element⁵ approaches, to

initiate at the inner surface of the ceramic substrate¹ where the maximum tensile stresses occur in function.^{2–5} Tensile stresses and resultant strains can therefore lead to the extension of pre-existing flaws on the inner surface of the restoration^{2–4} whose crack length may be extended prior to fracture as a result of subcritical crack growth in service.^{6,7} Glasses,^{6–9} glass-ceramics¹⁰ and ‘porcelains’^{11,12} used in dentistry have been reported to be variously susceptible to slow crack growth when subjected to subcritical loads and

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exposed to moisture in the oral environment.^{6–12} However, much of the mechanical testing on dental ceramic systems is carried out under standardised conditions, often in a dry environment, and routinely at a fixed crosshead rate which is unrepresentative of the masticatory cycle.¹³ In the context of *in vitro* biaxial flexure strength (BFS) testing and the susceptibility of dental glass–ceramics to slow crack growth, the crosshead rate routinely adopted is 0.75 ± 0.5 mm/min.^{14,15} A more appropriate loading regimen would include both the simulation of stable crack growth at slow deformation rates and a range of clinically representative loading regimes which reflect typical biting rates observed during a normal masticatory cycle.¹³

Water is readily available to the external surfaces of all-ceramic restorations from saliva and has been demonstrated as being the ‘primary rate-dependent mechanism’ in the development of cone cracks in monolithic dental crowns.¹⁶ However, moisture at the inner surface of the crown can also potentiate crown fracture of the restoration with water provided from the dentine,¹⁷ formed in the setting reaction of acid/base cements^{18,19} or transported through water sorption into resin-based cements.²⁰ The rate of crack growth in a soda-lime-glass was identified by Charles,^{6,7} using direct observations on loaded four-point-bend specimens, as being reaction rate controlled and dependent on the rate of attack of environmental moisture at the crack tip. Consequently, Charles^{6–8} postulated that water reacted with the molecules at the crack tip, breaking the Si–O–Si bonds to form a hydroxide which resulted in increased corrosion. The proposed model^{6–8} was confirmed by Wiederhorn⁹ who employed the direct observation technique for glass rods tested in a nitrogen environment containing varying percentages of water vapour to show that the rate of crack growth was reaction rate controlled and limited by the availability of water vapour. Unfortunately the direct observation method could not be used for opaque materials like dental ceramics and consequently the majority of crack propagation data for these materials has been collected for transparent glasses where crack growth was easily monitored.^{6–9,16}

When tested at a crosshead rate of 1 mm/min, the dental literature is consistent in that adhesive resin-based composite luting materials increase the strength of a range of dental ceramic materials.^{21–35} However, a recent investigation of seating load effects on the resin-strengthening, for a leucite reinforced glass–ceramic, identified a significant dependence of mean BFS on cementation seating load but not cementation seating duration³⁶ suggesting the situation is much more complex than originally thought.^{21–35} Interestingly, clinically there is little doubt that improved performance has been demonstrated in long term studies of resin-luted monolithic glass–ceramic restorations.^{37–40} However, one of the major deficiencies in testing traditional dental ceramic materials such as dental ‘porcelains’ is in the ceramic sample preparation methodology. Variability can be introduced through the technical skill of the operator during fabrication,⁴¹ sintering or heat-pressing⁴² and even CAD–CAM routes⁴³ which can bias the measured strength reported and data interpretation.⁴⁴ Therefore the use of soda-lime-glass, which has been advocated for many years as a suitable model system that provides increased consistency in specimen fabrication can be

beneficial.^{6–9,16} Recently, it has been demonstrated that a similar pattern of resin-strengthening to dental ‘porcelain’ is observed for a soda-lime-glass tested at the routine (1 mm/min) crosshead rate.⁴⁴ However, no attempt has been made to identify the crosshead rate effects on the BFS of adhesive resin-cementation, especially over clinically relevant masticatory loading rates. The aim of this study was to investigate these effects for disc-shaped soda-lime-glass specimens with and without resin-coating. Within the context of the experimental setting, a further aim was to investigate the influence of seating load applied during resin-cementation on the mean BFS. The hypothesis tested was that the soda-lime-glass analogue would be susceptible to slow crack growth in air and the magnitude and pattern of resin-strengthening observed would be dependent upon the crosshead test rate and the resin-seating load.

2. Materials and methods

2.1. Specimen preparation

Nominally identical disc-shaped specimens (11.4 mm diameter, 1.0 mm thickness) were produced by transversely cutting from a soda-lime-glass rod (Glass 8337B, SCHOTT-Rohrglas GmbH, Mitterteich, Germany) with a precision circular saw (Isomet[®] Low Speed Saw, Buehler, Lake Bluff, IL, USA) operating at 170 rpm under water irrigation using a diamond wafering blade (Series 15HC Diamond No. 11-4244, Buehler, Lake Bluff, IL, USA).⁴⁴ The disc-shaped specimens were dried with oil free compressed air immediately after sectioning and stored in a desiccator to prevent hydrolysis. In total, 288 disc-shaped specimens were produced prior to being alumina particle air abraded with 50 μ m alumina particles using a pressure of 4 bar, for 5 s (GOBI-2, Wassermann Dental-Maschinen GmbH, Hamburg, Germany).⁴⁴ To minimise excessive localised abrasion, the nozzle of the abrader was maintained perpendicular to the surface of the disc-shaped specimen at a constant distance (2 cm) and passed over the surface in a smooth linear sweeping motion.²⁶ The disc-shaped specimens were acid-etched with 9.6% hydrofluoric (HF) acid (Porcelain Etch Gel, Pulpdent Corporation, Watertown, MA, USA) for 120 s.⁴⁴ Following acid exposure, the specimens were rinsed for 10 s with distilled water to remove the acid, prior to drying with oil free compressed air. The central thickness of each of the 288 disc-shaped specimens was measured with a screw gauge micrometer fitted with a ball-nose adapter on the spindle tip which was placed in contact with the surface specimen during assessment of the disc thickness which was accurate to 1 μ m (Mitutoyo Corporation, Tokyo, Japan). The disc-shaped specimens were randomly allocated into twelve groups (Groups A–L, respectively) each containing 24 specimens.

Group A–D specimens were placed individually into four 24-well plates and stored in a desiccator prior to testing and analysis to act as the ‘alumina particle air abraded and acid-etched’ control groups. The remaining 192 disc-shaped specimens (Groups E–L) were silane treated with 3-methacryloxypropyl-trimethoxysilane (Rely-X Ceramic Primer, 3M ESPE, St. Paul, MN, USA) and allowed to air dry. A constant mass (0.030 ± 0.005 g) of a resin-based luting cement (Rely-X

Veneer, 3 M ESPE, St. Paul, MN, USA)⁴⁵ was dispensed centrally on the silane treated surface of the disc, which was placed in contact with a polyester film located on a 1 mm thick glass slide. A flat-ended indenter (11 mm diameter) was placed in contact with the surface of disc-shaped specimen during Rely-X Veneer seating, thereby minimising stress localisation by evenly distributing the applied seating load across the entire surface of the disc. Ninety-six disc-shaped specimens (Groups E–H) were Rely-X Veneer resin-coated with a seating load of 5 N applied for 5 s,³⁶ with a universal testing machine (Instron 5565, Instron Ltd., High Wycombe, UK). When the load was released, the Rely-X Veneer resin-coated disc-shaped specimens were light irradiated using a quartz tungsten halogen (Optilux 501, DSD Kerr, Danbury, CT, USA) light curing unit (LCU) through the 1 mm glass slide and polyester film, for 20 s with a 13 mm light tip diameter. The output intensity of the Optilux 501 at the resin surface was $480 \pm 20 \text{ mW/cm}^2$ – determined using the radiometer incorporated within the LCU. Following irradiation the central thickness of the Rely-X Veneer resin-coated disc-shaped specimens were measured using the screw gauge micrometer and the Rely-X Veneer resin-coating thicknesses were determined. The remaining 96 disc-shaped specimens (Groups I–L) were Rely-X Veneer resin-coated with a seating load of 30 N for 5 s³⁶ and light irradiated according to the procedure described for Groups E–H.

2.2. Biaxial flexure strength (BFS) testing

A ball-on-ring test configuration was used in the universal testing machine to determine the failure loads (N) of the control groups (Groups A–D) and the Rely-X Veneer resin-coated (Groups E–L) disc-shaped specimens following 24 h storage in a desiccator.⁴⁶ The control specimens (Groups A–D) were tested with the control (alumina particle air abraded and acid-etched) surface placed in contact with a flat sheet of latex (0.2 mm thickness) which covered the 10 mm diameter knife-edge ring support. The Rely-X Veneer resin-coated specimens (Groups E–L) were tested with the resin placed in contact with the latex covered ring support. The latex compensated for geometric variation and permitted small lateral displacements to minimise friction and shear. The discs were centrally loaded with a 4 mm diameter ball indenter engaged at crosshead rates of 0.01 mm/min (Groups A, E and I), 0.1 mm/min (Groups B, F and J), 1 mm/min (Groups C, G and K) and 10 mm/min (Groups D, F and L) where the load data acquisition was set at 2 ms intervals for the universal testing machine.

The biaxial flexure stress at failure was calculated using a bilayer analytical solution^{47,48} where the position of the neutral plane (t_n) was calculated using the flexural modulus of the soda lime glass ($E_1 = 40.0 (1.0) \text{ GPa}^{44}$), the Rely-X Veneer flexural modulus ($E_2 = 6.0 (0.2) \text{ GPa}^{44}$) and the thicknesses of the soda-lime-glass and Rely-X Veneer resin-coating (t_1 and t_2 , respectively) from Eq. (2).

$$t_n = \frac{E_1^*(t_1)^2 - E_2^*(t_2)^2}{2(E_1^*t_1 + E_2^*t_2)} \quad (1)$$

where

$$E_1^* = \frac{E_1}{1 - \nu_1} \quad (1a)$$

and

$$E_2^* = \frac{E_2}{1 - \nu_2} \quad (1b)$$

The Poisson's ratio (ν) of the Rely-X Veneer resin-coated disc was determined from Eq. (2).

$$\nu = \frac{\nu_1 t_1 + \nu_2 t_2}{t_1 + t_2} \quad (2)$$

where ν_1 (0.22⁴⁹) and ν_2 (0.27⁵⁰) were the Poisson's ratios of the soda-lime-glass and Rely-X Veneer, respectively. The biaxial flexure stress was calculated in the soda-lime-glass at the bonded interface ($z = 0$) in the centre of the Rely-X Veneer resin-coated disc specimens ($r = 0$). The biaxial flexure stress for the monolayer disc-shaped specimens (Groups A–D) was determined by setting $t_2 = 0$ using Eqs. (2)–(6).

$$\sigma = \frac{-3P(1 + \nu)(z - t_n)}{2\pi(t_1 + t_2)^3} \left[1 + 2 \ln \left(\frac{a}{b} \right) + \frac{1 - \nu}{1 + \nu} \left(1 - \frac{b^2}{2a^2} \right) \frac{a^2}{R^2} \right] \left[\frac{E_1^*(E_1^*t_1 + E_2^*t_2)(t_1 + t_2)^3}{(E_1^*t_1^2)^2 + (E_2^*t_2^2)^2 + 2E_1^*E_2^*t_1t_2(2t_1^2 + 2t_2^2 + 3t_1t_2)} \right] \quad (3)$$

($0 \leq z \leq t_1$) and

$$\sigma = \frac{-3P(1 + \nu)(z - t_n)}{2\pi(t_1 + t_2)^3} \left[1 + 2 \ln \left(\frac{a}{b} \right) + \frac{1 - \nu}{1 + \nu} \left(1 - \frac{b^2}{2a^2} \right) \frac{a^2}{R^2} \right] \left[\frac{E_2^*(E_1^*t_1 + E_2^*t_2)(t_1 + t_2)^3}{(E_1^*t_1^2)^2 + (E_2^*t_2^2)^2 + 2E_1^*E_2^*t_1t_2(2t_1^2 + 2t_2^2 + 3t_1t_2)} \right] \quad (4)$$

($-t_2 \leq z \leq 0$).

P was the load at fracture, a and R were the radii of the supporting ring (5 mm) and specimen (5.8 mm), respectively. b was the radius of loading contact area determined using Eq. (5).

$$b = \frac{t_1 + t_2}{3} \quad (5)$$

2.3. Statistical analysis

Analysis of group means was performed utilising a general linear model univariate analysis where factors were crosshead rate; cementation seating load; and cementation. Post hoc all paired Tukey tests employed at a significance level of $P < 0.05$ (SPSS for Windows 12.0.1, SPSS Inc., Chicago, USA) to test for differences between individual means. Additionally, the mean BFS and associated standard deviation data were plotted against the crosshead rate and linear logarithmic regression curve fitted to the raw data for the uncoated control groups (A–D) to elucidate the static fatigue effects of the soda-lime-glass material.

3. Results

A general linear model univariate analysis demonstrated that BFS was significantly increased ($P < 0.001$) by resin cementation, by increasing crosshead rate ($P < 0.001$) but not by the seating load used during cementation ($P = 0.095$). However, a significant factorial interactions was observed between

Table 1 – Mean biaxial flexure strengths for specimen Groups A–L, where the superscript denominators denote significant differences ($P < 0.05$) between columns for the uncoated control (Groups A–D), resin-coated with a seating load of 5 N (Groups E–H) and resin-coated with a seating load of 30 N (Groups I–L).

Crosshead rate [mm/min]	Uncoated controls [MPa]	Resin-coated at 5 N [MPa]	Resin-coated at 30 N [MPa]
0.01	Group A 54.6 (5.7) ^a	Group E 83.4 (12.8) ^d	Group I 74.6 (6.2) ^g
0.1	Group B 64.1 (7.5) ^b	Group F 110.3 (17.7) ^e	Group J 96.1 (15.1) ^h
1	Group C 80.7 (10.5) ^c	Group G 118.7 (17.8) ^{ef}	Group K 121.6 (13.4) ⁱ
10	Group D 81.4 (8.5) ^c	Group H 123.6 (13.1) ^f	Group L 128.7 (22.6) ⁱ

cementation seating load crosshead rate ($P = 0.006$). The mean BFS and associated standard deviation of the control soda-lime-glass disc-shaped specimens (alumina particle air abraded and HF acid-etched) tested at crosshead rates of 0.01, 0.1, 1 and 10 mm/min were 54.6 (5.7) MPa (Group A), 64.1 (7.5) MPa (Group B), 80.7 (10.5) MPa (Group C), 81.4 (8.5) MPa (Group D), respectively (Table 1). Post hoc analyses demonstrated a significant reduction in mean BFS was identified for Group A specimens (Table 1) compared with Groups B–D (all $P < 0.001$). Further analysis identified a significantly reduced mean BFS for Group B specimens compared with Group C ($P < 0.001$) and Group D ($P < 0.001$) specimens (Table 1), however, no difference in mean BFS values were identified between Groups C and D ($P = 0.99$). The mean BFS and associated standard deviation data was plotted against the crosshead rate and linear logarithmic regression curve fitted to the raw data for the uncoated control groups (A–D) which demonstrated the static fatigue effects of the soda-lime-glass analogue (Fig. 1).

The mean BFS and associated standard deviation of the Rely-X Veneer resin-coated soda-lime-glass disc-shaped specimens cemented with a 5 N seating load and tested at crosshead rates of 0.01, 0.1, 1 and 10 mm/min were 83.4

(12.8) MPa (Group E), 110.3 (17.7) MPa (Group F), 118.7 (17.8) MPa (Group G) and 123.6 (13.1) MPa (Group H), respectively (Table 1). A significant decrease in mean BFS was identified for Group E specimens compared with Groups F–H (all $P < 0.001$). No significant differences in mean BFS was identified between Groups F and G ($P = 0.248$) although a significant decrease in mean BFS was identified for Group F specimens when compared with Group H specimens ($P = 0.019$). Additionally, no significant differences in mean BFS was identified between Groups G and H specimens ($P = 0.69$) (Table 1).

Increasing the seating load to 30 N prior to testing at crosshead rates of 0.01, 0.1, 1 and 10 mm/min resulted in mean BFS values and associated standard deviations of 74.6 (6.2) MPa (Group I), 96.1 (15.1) MPa (Group J), 121.6 (13.4) MPa (Group K) and 128.7 (22.6) MPa (Group L). Significant decreases in mean BFS were identified for Group I specimens when compared with Group J–L specimens (all $P < 0.001$) and Group J specimens when compared with Group K and L specimens (all $P < 0.001$) but no significant difference in mean BFS was determined between Group K and L ($P = 0.392$) specimens (Table 1).

Rely-X Veneer resin-coating (Groups E–L) resulted in significant increases (all $P < 0.001$) in the mean BFS data when compared with the uncoated control groups (A–D) at each of the crosshead rates examined (Table 2). Significant increases in mean BFS values were observed between the Rely-X Veneer resin-coated specimens prepared at seating loads of 5 and 30 N and tested at crosshead rates of 0.01 mm/min (Groups E and I; $P = 0.003$) and 0.1 mm/min (Groups F and J; $P = 0.003$). However, no significant differences in mean BFS values were observed at crosshead rates of 1 mm/min (Groups G and K; $P = 0.753$) and 10 mm/min (Groups H and L; $P = 0.51$) for the Rely-X Veneer resin-coated specimens prepared at seating loads of 5 and 30 N.

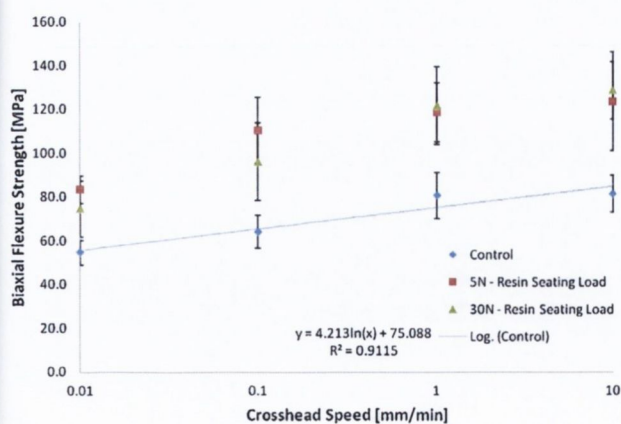


Fig. 1 – Biaxial flexure strength against crosshead rate for the uncoated disc-shaped controls (Groups A–D) and the resin-coated disc-shaped specimens (Groups E–H with resin-seating load of 5 N, Groups I–L with resin-seating load of 30 N). A linear logarithmic regression curve was fitted to the raw data for the uncoated control groups (A–D) which demonstrated the static fatigue effects of the soda-lime-glass analogue.

4. Discussion

There is agreement in the ceramic, engineering and dental literature that slow crack growth reduces the strength of glasses.^{6–12,16,51} For the soda-lime-glass dental ‘porcelain’ analogue investigated, when the mean BFS and associated standard deviation data was plotted against the crosshead rate and linear logarithmic regression curve fitted to the raw data for the uncoated controls (Groups A–D) a pattern of strength dependence consistent with the effects of static fatigue was

Table 2 – Significance levels identified when comparing the mean biaxial flexure strength data for the uncoated control (Groups A–D), resin-coated with a seating load of 5 N (Groups E–H) and resin-coated with a seating load of 30 N (Groups I–L) at respective crosshead rates. Analysis of group means was performed utilising a general linear model univariate analysis where factors were crosshead rate; cementation seating load; and cementation.

Crosshead rate [mm/min]	Uncoated vs resin-coated at 5 N	Uncoated vs resin-coated at 30 N	Coated at 5 N vs coated at 30 N
0.01	Groups A and E P < 0.001	Groups A and I P < 0.001	Groups E and I P = 0.003
0.1	Groups B and F P < 0.001	Groups B and J P < 0.001	Groups F and J P = 0.003
1	Groups C and G P < 0.001	Groups C and K P < 0.001	Groups G and K P = 0.753
10	Groups D and H P < 0.001	Groups D and L P < 0.001	Groups H and L P = 0.510

demonstrated (Fig. 1). This is consistent with previous investigations of the static fatigue of glasses,^{6–12,16} and dental ‘porcelains’ since Baker and Preston^{53,54} identified the static fatigue failure curves for dental ‘porcelains’ were similar to that for glass tested under identical conditions. Interestingly, the glass specimens exhibited a more pronounced susceptibility to static fatigue failure than the dental ‘porcelains’ when immersed in water which was associated with the decreased glassy phase present in the latter material.^{53–54} These findings were reinforced by Jones et al.⁵⁵ who tested three point flexure specimens (25 mm length, 4 mm width and 2 mm thickness) at strain rates of 0.01 and 800 cm/min and identified that the mean three point flexure strength of dental porcelain was increased from 43 to 97% when tested at the higher strain rate, thereby confirming the long term loading effect of dental porcelain. One problem, however, with all early studies of static fatigue failure and rate dependence in glassy materials was they were limited by deficiencies in universal testing devices available which produced unreliable data as a result of inertia effects.^{53,54}

All resin-coated disc-shaped specimens were significantly strengthened (at both the 5 N and 30 N seating loads, Groups E–L) when compared with the uncoated controls (Groups A–D), at their respective crosshead rates confirming previous in vitro findings.^{21–35} We have a similar pattern of rate dependence effects on resin-cementation but there is a clear deviation (Fig. 1) from the log relationship observed with the uncoated controls (Groups A–D). Therefore, while cementation seating load alters the interaction of the resin-based composite luting material with the resident ceramic flaw population, seating load variability results in differences in the interaction between the resin cement and surface defects modifying the resin–ceramic hybrid layer formed.^{22–27} The resin–ceramic hybrid layer has previously been demonstrated to consist of a multiplicity of defect sizes and resin interpenetration of the surface and associated stressing patterns of the resin within the region of surface defects is complex but results in clinically relevant reinforcement.^{24–26} The characteristics of the resin–ceramic hybrid layer have been reported to be ‘mediated by the complex interactions of various elements’²⁵ which include the seating load during resin-cementation. The impact of the variability in stressing rate on reinforcement conferred by differing resin–ceramic hybrid layers is not clear. In the current study, differences in resin-strengthening were evidently rate dependent (Fig. 1), exaggerated at slow (0.01 and

0.1 mm/min) crosshead rates but the differences were not rate dependent at high (1 and 10 mm/min) crosshead rates. These findings suggest competition between known phenomena at slow deformation rates, namely static fatigue extension of the resident glass flaw population (driven by water) and non-elastic (viscoelastic) behaviour of the resin within the resin–ceramic hybrid layer,^{24–26,52} a combination which is previously unreported in the literature.

Previous work on resin-based composite systems has shown viscoelastic¹³ and creep behaviour⁵⁶ in resins at slow testing rates and our data supports these observations. At high loading rates, the available time for viscoelastic deformation of the resin constituent within the resin–ceramic hybrid layer is limited such that viscoelastic effects do not dominate the resin-strengthening observed. Retrieved clinically failed all-ceramic crowns have been reported to break into two fragments, with little damage evident beneath the wear facet and patients reporting that failure occurred on chewing something ‘soft’.⁵⁷ This reinforces the importance of incorporating relevant crosshead test rates to simulate slow crack growth effects when testing all-ceramic materials routinely used in dentistry.

5. Conclusion

Crosshead test rate and resin-cementation load have been shown to significantly modify resin-strengthening mediated by the resin–ceramic hybrid layer in the presence of ambient moisture.

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3.4 THE INFLUENCE OF ATMOSPHERIC MOISTURE ON THE RESIN-STRENGTHENING OF A SODA LIME GLASS ANALOGUE FOR DENTAL PORCELAIN.

P Hooi, O Addison, GJP Fleming. The influence of atmospheric moisture on the resin-strengthening of a soda lime analogue for dental porcelain. Submitted to Journal of Dentistry, April 2013.

The investigation of testing rate and resin seating load on the pattern of resin-strengthening demonstrated that resin-coating the soda lime glass disc-shaped specimens significantly increased the BFS as testing rate was increased, when compared with the uncoated controls (Manuscript 3.3). The findings established the importance of employing clinically relevant testing rates. However, the environmental moisture was not a controlled factor during resin-cementation and therefore it was difficult to identify the contribution of moisture during the resin-cementation process on the observed rate-dependent pattern of reinforcement (Manuscript 3.3). While the influence of external moisture on the evolution of cone cracks on the outer surface of dental ceramic restorations has been reported (Kim et al., 1999; Yeo et al., 2000), the influence of moisture on the intaglio surface of the restoration is not fully understood. It was also postulated that the resin seating load may influence the characteristics of the resin-ceramic hybrid layer in the absence of moisture and was also examined. The current study investigated if resin-coating in the absence of environmental moisture would influence the resin-strengthening mechanism for the factors resin-cementation seating load and test crosshead speed.

ABSTRACT

Objectives: To investigate if resin-cementation of the soda lime glass dental analogue could further elucidate information regarding the pattern of resin-reinforcement when coated in an environment actively scavenged of moisture.

Methods: 192 soda lime disc-shaped specimens which were alumina particle air abraded, hydrofluoric acid-etched and silane coated were randomly assigned to eight groups (n=24 per group) prior to resin-coating at seating loads of 5 N (Groups A-D) and 30 N (Groups E-H) in an environment where moisture was actively scavenged and maintained below 15 ppm. Following one week storage the discs were tested in biaxial flexure at crosshead rates of 0.01, 0.1, 1 and 10 mm/min. Analysis of group means was performed utilising a general linear model univariate analysis and post-hoc all paired Tukey tests ($P<0.05$).

Results: The general linear model univariate analysis identified the mean biaxial flexure strength (BFS) was significantly influenced by the factors resin-cementation seating load ($P<0.001$) and crosshead speed of the applied load ($P<0.001$) with a significant interaction ($P=0.008$) between both factors. The linear logarithmic regression curves fitted to the group mean BFS data plotted against the crosshead speed highlighted significant differences between the pattern of resin-strengthening for the cementation loads and testing conditions.

Conclusions: The decrease in penetration expected from the resin in cracks within the 'resin-ceramic hybrid layer' following the removal of the higher seating load was proposed as the associated modifying parameter for the resin-strengthening behaviour. The response of the moisture, however, was difficult to interpret owing to the different behaviour of the 'resin-ceramic hybrid layer' with the two loading regimes examined.

Introduction

Clinical evidence to support the use of resin-based adhesive technologies for dental ceramics is evident in the findings of a comprehensive 14 year prospective study of 1444 Dicor restorations reported by Malament and Socransky [1-2] and the results of a 6 year follow-up of 115 fired ceramic inlays reported by van Dijken et al. [3]. The authors demonstrated ceramic restorations were significantly more resistant to fracture when adhesively bonded to the prepared tooth structure using resin-based materials compared with acid-base cements [1-3]. The clinical outcomes data reported [1-3] supported the theories in the dental literature of crack healing [4] and crack closure stresses secondary to polymerisation shrinkage [5]. Using the biaxial flexure strength (BFS) testing methodology, strengthening of PLV and DBC ceramic materials with resin-cement has been shown to be insensitive to defect size [6] but sensitive to macroscopic surface roughness [7] and resin-cement elasticity [8]. Therefore, the traditionally proposed theories of crack healing [4,9] or the crack closure stresses secondary to polymerisation shrinkage [5,10] have been demonstrated to incompletely explain the magnitude of observed strengthening since both theories specifically implied a sensitivity to individual defect size and geometry [6-8].

It is well known that glasses, glass-ceramics and dental porcelains are variously susceptible to the effects of moisture-assisted slow crack growth under subcritical loading [11-16]. In the clinical setting while moisture is available to the external surfaces of the crown, moisture is also readily available to the intaglio surface. The tooth structure is routinely prepared by the practitioner for PLV or DBC restorations such that moisture transport through the dental tubules to the luting cement is expected and RBC materials themselves absorb atmospheric moisture [17] during the cementation process. Using disc-shaped specimens in a laboratory setting, the influence of moisture-assisted slow crack growth was reported in cone cracks at

the exposed outer ceramic disc surface following Hertzian indentation [18-19]. Lee et al. [20] reported that the available external moisture did not appear to diffuse significantly through a soda lime glass substrate to the intaglio surface under tensile stress and the author raised a question about the viscoelastic component of the resin-coating and the interaction with the soda lime glass substrate [20]. In an attempt to understand this phenomenon the authors felt it was pertinent to investigate the influence of moisture availability during radial cracking of the intaglio surface, which during clinical function remains in contact with the resin-based composite (RBC) luting material.

Previously, the influence of testing rate effects was observed on the BFS data from disc-shaped soda lime glass specimens which were resin-coated at seating loads of 5 and 30 N in ambient air and tested at crosshead speeds ranging from 0.01 mm/min to 10 mm/min [21]. However, the specific contribution of ambient moisture during resin-coating of the disc-shaped specimens on the observed rate dependent and seating load pattern of resin-reinforcement was difficult to establish because ambient moisture was not a controlled factor in the study. The aim of this study was to investigate if resin-cementation of the soda lime glass dental analogue could further elucidate information regarding the pattern of resin-reinforcement when coated in an environment where moisture was actively scavenged and maintained below 15 ppm. The hypotheses tested were that resin-coating in the absence of environmental moisture would influence the resin-strengthening mechanism for the factors resin-cementation seating load and test crosshead speed.

Materials and Methods

Soda Lime Specimen Preparation

A soda lime glass rod (Glass 8337B, SCHOTT-Rohrglas GmbH, Mitterteich, Germany) was cut perpendicular to its longitudinal axis using a diamond wafering blade (Series 15HC Diamond No. 11-4244, Buehler, Lake Bluff, IL, USA) and a precision circular saw (Isomet[®] Low Speed Saw, Buehler, Lake Bluff, IL, USA). The diamond wafering blade rotated at 170 rpm under constant water irrigation to produce 192 nominally identical disc-shaped specimens (11.4 mm diameter, 1.0 mm thickness). The specimens were dried with oil free compressed air and stored in a desiccator to prevent hydrolysis of the silicate bonds [11-13]. The disc-shaped specimens were alumina particle air abraded with 50 μm alumina particles at a pressure of 4 bar for 5 s (GOBI-2, Wassermann Dental-Maschinen GmbH, Hamburg, Germany). The alumina particle air abrader nozzle was positioned 2 cm perpendicular to the surface of the specimen and traversed the surface in a smooth linear sweeping motion, thereby distributing uniform abrasion across the surface domain [21-23]. The specimens were acid-etched with 9.6% hydrofluoric (HF) acid (Porcelain Etch Gel, Pulpdent Corporation, Watertown, MA, USA) for 120 s, rinsed for 10 s with distilled water to remove the acid, prior to complete drying with oil free compressed air. The thickness of each disc was measured centrally ($\pm 1 \mu\text{m}$) with a screw gauge micrometer (Mitutoyo Corporation, Tokyo, Japan) where the ball-nose adapter on the spindle tip was placed in contact with the treated surface of the disc [21-22]. Randomisation of the soda lime disc-shaped specimens was performed by random allocation into eight groups of 24 specimens (Groups A-H, respectively), which were silane treated with 3-methacryloxypropyl-trimethoxysilane (Rely-X Ceramic Primer; 3M ESPE, St. Paul, MN, USA) and allowed to air dry prior to storage in a desiccator until required for further testing and analysis.

Resin-cementation

The disc-shaped specimens were placed in an environmental chamber (DAB01S Anaerobic Glove box; Saffron Scientific Equipment Ltd., Knaresborough, UK) containing 20% oxygen and 80% nitrogen in which moisture was actively scavenged and maintained below 15 ppm. A constant mass (0.030 ± 0.005 g) of the Rely-X Veneer (LOT N284083, Shade A3) RBC luting cement (3M ESPE, St. Paul, MN, USA) was dispensed centrally on the treated surface (alumina particle air abraded, HF acid-etched and silane coated) of the disc-shaped specimens and placed in contact with a polyester film located on a 1 mm thick glass slide. In total, 96 disc-shaped specimens (Groups A-D) were coated with the Rely-X Veneer luting cement at a cementation seating load of 5 N (equivalent mass of 500 g) applied for 5 s, with a flat-ended cylindrical silicone bung indenter (15 mm length, 10 mm diameter). The cylindrical silicone bung minimised stress localisation by evenly distributing the cementation seating load across the disc-shaped specimen. Following manual release of the applied load, the resin-coated disc-shaped specimens were light irradiated using a quartz tungsten halogen (QTH) (Optilux 501; DSD Kerr, Danbury, CT, USA) light curing unit (LCU) through the 1 mm glass slide and polyester film, for 20 s with a 13 mm light tip diameter. The output intensity of the QTH LCU at the resin surface was 480 ± 20 mW/cm² when determined using the radiometer incorporated within the LCU. The remaining 96 disc-shaped specimens (Groups E-H) were resin-coated with a cementation seating load of 30 N (equivalent mass of 3 Kg) for 5 s and light irradiated according to the procedure described using the QTH LCU. Following irradiation the central thickness of each Rely-X Veneer resin-coated disc-shaped specimen was measured using the screw gauge micrometer and the Rely-X Veneer resin-coating thicknesses were determined. The 192 resin-coated disc-shaped specimens (Groups A-H) were stored in a desiccator for one week prior to testing and further analysis.

Biaxial Flexure Strength (BFS) Testing

A universal testing machine (Instron Model No. 5565, Instron Ltd., High Wycombe, UK) was used in conjunction with a ball-on-ring test configuration to determine the failure load (N) of the resin-coated specimens (Groups A-H). The resin-coated surface was placed centrally on a flat sheet of thin latex (0.2 mm thickness) which covered the 10 mm diameter knife-edge ring support to compensate for geometric variation in the specimens and accommodate lateral displacements to minimise shear due to friction [24]. The specimens were centrally loaded with a 4 mm diameter ball indenter operating at a crosshead speed of 0.01 (Groups A and E), 0.1 (Groups B and F), 1.0 (Groups C and G) and 10 mm/min (Groups D and H). The load data acquisition interval of the universal testing machine was set at 2 ms to capture the failure event at the higher crosshead speed (10 mm/min).

Using the bilayered analytical solution advocated by Hsueh et al. [25], the position of the neutral plane (t_n) was calculated from Equation 1 using the flexural moduli of the soda lime glass (E_1 - 40.0 (1.0) GPa [22]) and Rely-X Veneer luting cement (E_2 - 6.0 (0.2) GPa [26]) and the relative thicknesses of the soda lime glass and Rely-X Veneer resin-coating (t_1 and t_2 , respectively).

$$t_n = \frac{E_1^*(t_1)^2 - E_2^*(t_2)^2}{2(E_1^*t_1 + E_2^*t_2)}$$

Equation 1

where: $E_1^* = \frac{E_1}{1-\nu_1^2}$ and $E_2^* = \frac{E_2}{1-\nu_2^2}$

The Poisson's ratio (ν) of the bilayered resin-coated disc was determined from Equation 2.

$$\nu = \frac{(\nu_1 t_1 + \nu_2 t_2)}{t_1 + t_2} \quad \text{Equation 2}$$

where ν_1 (0.22 [27-28]) and ν_2 (0.27 [29]) were the Poisson's ratios of the soda lime glass and Rely-X Veneer luting cement, respectively. The biaxial flexure stress was calculated in the soda lime glass at the bonded interface ($z=0$) in the centre of the Rely-X Veneer resin-coated disc specimens ($r=0$).

$$\sigma = \frac{-3P(1+\nu)(z-t_n)}{2\pi(t_1+t_2)^3} \left[1 + 2\ln\left(\frac{a}{b}\right) + \frac{1-\nu}{1+\nu} \left(1 - \frac{b^2}{2a^2}\right) \frac{a^2}{R^2} \right] \left[\frac{E_1^*(E_1^*t_1 + E_2^*t_2)(t_1+t_2)^3}{(E_1^*t_1^2)^2 + (E_2^*t_2^2)^2 + 2E_1^*E_2^*t_1t_2(2t_1^2 + 2t_2^2 + 3t_1t_2)} \right] \quad \text{Equation 3}$$

($0 \leq z \leq t_1$) and

$$\sigma = \frac{-3P(1+\nu)(z-t_n)}{2\pi(t_1+t_2)^3} \left[1 + 2\ln\left(\frac{a}{b}\right) + \frac{1-\nu}{1+\nu} \left(1 - \frac{b^2}{2a^2}\right) \frac{a^2}{R^2} \right] \left[\frac{E_2^*(E_1^*t_1 + E_2^*t_2)(t_1+t_2)^3}{(E_1^*t_1^2)^2 + (E_2^*t_2^2)^2 + 2E_1^*E_2^*t_1t_2(2t_1^2 + 2t_2^2 + 3t_1t_2)} \right] \quad \text{Equation 4}$$

($-t_2 \leq z \leq 0$).

where P was the load at fracture, a and R were the radii of the supporting ring (5 mm) and specimen (5.7 mm), respectively. b was the radius of loading contact area determined using Equation 5.

$$b = \frac{t_1 + t_2}{3} \quad \text{Equation 5}$$

Statistical Analysis

A general linear model univariate analysis of the BFS group means was performed where the factors examined were resin-cementation seating load and crosshead speed of the applied load during BFS testing. Post-hoc all paired Tukey tests were also performed at $P < 0.05$. Two

separate one-way analysis of variances (ANOVAs) were used to compare the mean BFS data for the resin-coated discs with the dependent variables investigated, namely resin-cementation seating load (5 and 30 N) and crosshead speed of the applied load (0.01, 0.1, 1.0 and 10 mm/min). Additionally, the mean BFS and associated standard deviation data were plotted against the crosshead speed and linear logarithmic regression curves were fitted to the group mean values for the disc-shaped specimens resin-coated in a moisture-free environment.

Results

The general linear model univariate analysis identified the mean BFS was significantly influenced by the factors resin-cementation seating load ($P<0.001$) and crosshead speed of the applied load ($P<0.001$) with a significant interaction ($P=0.008$) between both factors. When the BFS data for the soda lime disc-shaped specimens resin-cemented with a 5 N seating load were examined for the factor crosshead speed of the applied load, the mean BFS and associated standard deviation when tested at 0.01, 0.1, 1.0 and 10 mm/min were 82.1 (11.7) MPa (Group A), 90.6 (10.3) MPa (Group B), 102.1 (14.2) MPa (Group C) and 115.6 (13.1) MPa (Group D), respectively (Table 1). The one-way ANOVA highlighted no significant difference in the mean BFS between Group A and B specimens tested at 0.01 and 0.1 mm/min ($P>0.101$) although a significant increase in mean BFS was identified for Group C ($P<0.010$) and Group D ($P<0.001$) specimens. A significant increase in mean BFS was identified for Group D specimens compared with Group C specimens ($P=0.002$) (Table 1). Increasing the cementation seating load to 30 N in a moisture-free environment prior to testing at crosshead speeds of 0.01, 0.1, 1.0 and 10 mm/min resulted in mean BFS values and associated standard deviations of 80.2 (13.3) MPa (Group E), 81.9 (13.8) MPa (Group F), 89.0 (14.5) MPa (Group G) and 95.8 (9.8) MPa (Group H). The one-way ANOVA identified no significant difference for Group E specimens when compared with Group F ($P=0.966$) and Group G ($P=0.092$) specimens, although a significant increase in mean BFS was observed for Group H specimens ($P<0.001$) tested at 10 mm/min. No significant difference in mean BFS was determined between Group G and H specimens ($P=0.271$) (Table 1).

When the four individual one-way ANOVAs which investigated the influence of resin-cementation seating load (5 or 30 N) were examined no significant differences in the mean

BFS was identified when the resin-coated specimens were tested at a 0.01 ($P=0.973$) or 0.1 mm/min ($P=0.174$). However, a significant increase in the mean BFS of the disc-shaped resin-coated at 5 N (compared with 30 N) was observed when tested at 1.0 ($P=0.016$) and 10 ($P < 0.001$) mm/min (Table 1).

The linear logarithmic regression curves fitted to the group mean BFS data plotted against the crosshead speed for the disc-shaped specimens resin-coated in a moisture-free environment highlighted significant differences between the pattern of resin-strengthening for the different cementation loads (Figure 1). At the lowest crosshead speed of 0.01 mm/min, convergence of the linear logarithmic regression curves fitted to the group mean BFS data was observed for the specimens resin-coated with a seating load of 5 N and a seating load of 30 N with no significant difference ($P=0.973$) identified between the group mean BFS values for Groups A and E. Similarly, at a crosshead speed of 0.1 mm/min the group mean BFS values for the specimens resin-coated with a seating load of 5 N (Group B) and 30 N (Group F) were not significantly different ($P=0.174$). However, the pattern of resin-strengthening was modified with increased test crosshead speed. For the specimens tested at crosshead speeds of 1 and 10 mm/min, the group mean BFS for the specimens resin-coated with a seating load of 5 N (Groups C and D, respectively) were significantly increased ($P=0.016$ and $P < 0.001$, respectively) when compared with the group mean BFS for the specimens resin-coated with a seating load of 30 N (Group G and H, respectively).

Discussion

All resin-coated disc-shaped specimens were significantly strengthened (at both the 5 N and 30 N seating loads,) when compared with the uncoated controls [21] at their respective crosshead rates confirming previous *in vitro* findings [4-8,20-23,26]. However, the pattern of resin-strengthening was modified depending on the resin-seating load and BFS test rate. The differences between the seating load data can be explained by considering the variable interactions of the resin-cement with the ceramic surface during application and removal of the seating load. Increased seating load during cementation may be assumed to increase the inter-penetration of the resin with the ceramic surface roughness (tortuosity). However, resistance to compressive deformation of the monomeric resin paste may result following the release of the applied seating loads (5 and 30 N, respectively). The load application process is likely to result in the prepared ceramic surface being placed in tension and the monomeric resin paste under compression. Following the release of the applied load, variability may be introduced as the elastic deformation of the resin-cement is reversed and the compression of the resin paste is removed. Such effects may lead to 'spring-back' which is related to the release of energy associated with the initial resistance to deformation on loading. Although these complex effects were not quantified they are expected to result in a modification of the degree of resin-cement penetration. It is suggested that the mechanical response of the increased penetrance of the resin especially when restricted to thin layers in cracks within the 'resin-ceramic hybrid layer' could be significantly altered. Therefore, for specimens resin-luted at a seating load of 5 N, the increased BFS data suggests a more intimate interpenetration of the resin within the cracks in the 'resin-ceramic hybrid layer'.

Earlier interpenetration, results in a restriction of the resin to thin films near the crack tip where Poissons constraint effects are proposed to be operative [30]. As a result, during

flexure, the interpenetrated resin extends perpendicular to the crack face producing a compensating Poisson contraction parallel to the crack surface [30] effectively increasing the stiffness of the resin such that the resin within the 'resin-ceramic hybrid layer' behaves more closely to the bulk ceramic [30-31]. This was observed in the almost linear increase in mean BFS observed for the soda lime specimens which were uncoated or coated at a load of 5N (Figure 1). The decrease in penetration expected from the resin in cracks within the 'resin-ceramic hybrid layer' following the removal of a load of 30 N was proposed as the associated modifying parameter for the resin-strengthening behaviour. Interestingly at the lower loading rates, differences between the group mean BFS data for the different seating loads were not significant and it is suggested that these observations are supported by the viscoelastic [32] and creep behaviour [33] of resins at slow testing rates which becomes the dominant or determining phenomenon.

The oral environment provides a constant source of water which challenges the performance of PLV and DBC restorations in service. Water is known to compromise the strength of silicate materials [34] and slow crack growth damage has been demonstrated with dental porcelain [35]. Ritter and Lin [36] reported that stress corrosion was inhibited by a protective resin-coating of a soda lime glass by reducing the available moisture at the crack tip. External water has been reported to be the primary determinant in the rate-dependent progression of cone cracks on the outer surface of monolithic ceramic restorations with a significant glassy phase [20]. Epoxy coatings have been demonstrated to protect the surface of soda lime glass from moisture-assisted degradation [37] and while the resin-cementation of the intaglio surface is considered initially protective against water ingress and subsequent degradation, Huang et al. [38] reported that water diffusion through the resin-cementation layer is probable. In addition, Addison et al. [39] demonstrated the significant degradation of resin-

strengthening following the immersion in water of resin-coated dentine porcelain disc-shaped specimens which could be attributable to either effects of moisture at the ceramic surface or effects of the mechanical responses of moisture at the resin itself through increased plastic deformation. However, the BFS data is consistent with the study by Roach and Cooper [40] which highlighted the dynamic susceptibility of soda lime glass to slow crack growth by demonstrating rate dependence when compared with plastic coated soda lime glass through a range of crosshead speeds from 0.5 to 500 mm/min.

Conclusions

The decrease in penetration expected from the resin in cracks within the 'resin-ceramic hybrid layer' following the removal of the higher seating load was proposed as the associated modifying parameter for the resin-strengthening behaviour. The response of the moisture, however, was difficult to interpret owing to the different behaviour of the 'resin-ceramic hybrid layer' with the two loading regimes examined.

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Crosshead Speed [mm/min]	Mean BFS when Resin-coated at 5 N [MPa]	Mean BFS when Resin-coated at 30 N [MPa]	One-way ANOVAs for resin-coating load (5 v 30 N)
0.01	Groups A 82.1 (11.7) ^a	Groups E 80.2 (13.3) ^d	Groups A and E P=0.973
0.1	Groups B 90.6 (10.3) ^a	Groups F 81.9 (13.8) ^d	Groups B and F P=0.174
1	Groups C 102.1 (14.2) ^b	Groups G 89.0 (14.5) ^{de}	Groups C and G P=0.016
10	Groups D 115.6 (13.1) ^c	Groups H 95.8 (9.8) ^e	Groups D and H P<0.001

Table 1: Mean biaxial flexure strength data for the soda lime glass disc-shaped specimens resin-coated at seating loads of 5 or 30 N in a moisture controlled environment and tested at differing crosshead speeds. One-way ANOVA results are highlighted with the superscript denominators denoting significant differences ($P<0.05$) within columns.

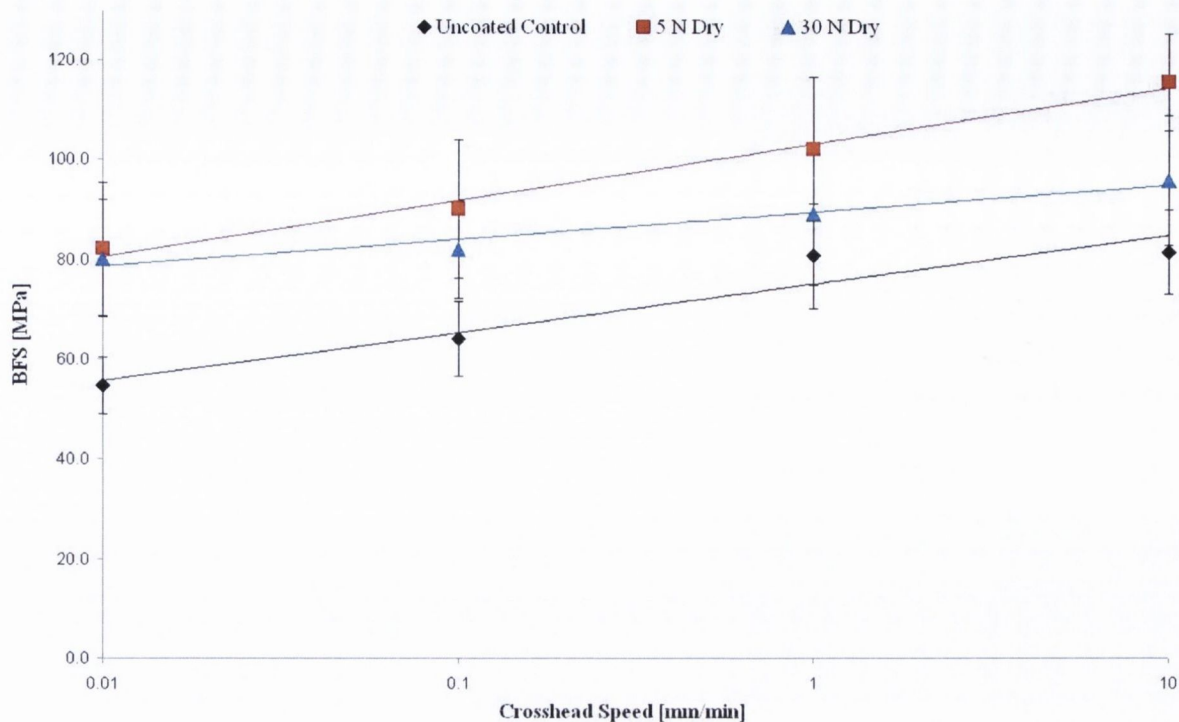


Figure 2: Linear logarithmic regression curves for the mean biaxial flexure strength data plotted against the test crosshead speed for the resin-coated soda lime disc-shaped specimens and the uncoated control groups [21] highlighting the differences in resin strengthening behaviour at the different resin-cementation seating loads.

3.5 STRENGTH DETERMINATION OF CURVED BRITTLE DENTAL MATERIALS

P Hooi, O Addison, GJP Fleming . Strength determination of curved brittle dental materials.
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Biaxial flexure strength testing has been employed as a clinically relevant testing modality for dental ceramics (Fleming et al., 2006) predominantly because the resulting failure mode of radial fracture is also observed in the clinical failure of all-ceramic restorations (Kelly, 1999). It has been proposed that the geometric features evident clinically in PLV and DBC restorations are not represented by flat disc-shaped specimens (Qasim et al., 2006; Rekow et al., 2009; Rafferty et al., 2010; Sornsuan et al., 2011). The load to failure testing of full contour all-ceramic crowns has been widely employed in an attempt to evaluate the influence of geometry on fracture but these ‘crunch-the-crown’ tests are not clinically relevant because they fail to replicate the fracture modes observed in the clinical setting (Kelly et al., 2012). The geometric simplicity of flat bilayered disc-shaped specimens has led to the development of closed-form analytical solutions to determine the BFS which is more useful than load-to-failure data that provides no information on the stress state within the specimen. The geometric limitations of flat disc-shaped specimens employed for BFS testing have been considered and the current study employed the previously validated FEA model (Manuscript 3.1) which was adapted to determine the stress state within the specimen, in the absence of an available analytical solution. The current study investigated the influence of non-planar geometries on the maximum tensile stress of curved (disc-shaped) specimens tested in biaxial flexure.

ABSTRACT

Objectives: To determine the influence of non-planar geometries on the maximum tensile stress of a soda lime glass analogue for dental porcelain by testing curved disc-shaped specimens in biaxial flexure.

Methods: Flat soda lime glass disc-shaped specimens were scribe-cut from the flat soda-lime glass stock and divided into five groups (n=20) with a flat control (Group A). Curvature was applied to the remaining scribe-cut flat discs by slump forming with radii of curvature of 40, 30, 20 and 10 mm and assigned to Groups B to E, respectively. The discs were alumina particle air abraded and hydrofluoric acid-etched before the surface texture and curvature was determined by profilometry. The discs were tested in biaxial flexure and the maximum tensile stress was determined with the load to failure data using a finite element analysis method. Analysis of group means was performed utilising a general linear model univariate analysis and post hoc all paired Tukey tests ($P < 0.05$). The mean maximum principal stresses and load to failure were plotted against radius of curvature.

Results: The radii of curvature of the disc-shaped specimens in Groups B-E differed significantly. The surface roughness of the discs with the minimum radius of curvature (Group E) was significantly reduced. Significant increases in the mean load to failure were observed as the radius of curvature was reduced (more severely curved). However, the maximum principal stress was not observed to demonstrate a similar sensitivity to radius of curvature for the curvatures investigated.

Conclusions: The findings highlighted the sensitivity of the load to failure data to specimen shape demonstrating that comparisons between load to failure data in isolation is problematic and highlights the potential complications associated with complex specimen geometry.

Introduction

The dental literature is replete with studies examining the fracture resistance of all-ceramic restorative materials with the ‘crunch-the-crown’ load to failure test being the most commonly used approach [1-8]. The clinical relevance of the ‘crunch-the-crown’ test has been challenged, most notably because the failure mode involves indenter contact surface damage which fails to replicate the clinical failure mode, namely sub-surface radial fracture [9]. Following ‘crunch-the-crown’ testing the only data available is the load to failure which provides no information on the stress patterns within the complex geometry of the specimen bulk. Comparisons between ‘crunch-the-crown’ testing studies are usually not possible [9] owing to inconsistencies between individual laboratory mechanical testing protocols [10,11]. In contrast, the testing of geometric planar specimens allows for the calculation of failure stresses using bending theory. By employing standardised geometries, sample preparation protocols and loading parameters, comparisons between materials and investigators are made possible. However, the data acquired from this approach alone has been proposed to provide a conservative measure of fracture behaviour [12] and fails to account for the influence of geometric factors which may modify the stressing patterns, or defect populations within a complex body such as a loaded restoration or tooth.

The finite element analysis (FEA) methodology has been widely used in the dental literature to simulate the stress distribution in all-ceramic crowns under an applied load. Examples include Cuddihy et al. [13] who simulated the influence of clinically representative root canal access cavity preparations through ceramic restorations with the FEA method to determine the stress patterns which resulted from the applied masticatory loads. Rekow et al. [14] used FEA to predict the impact of tooth preparation dimensions and stylised crown geometry on

stress distributions under a clinically relevant loading simulation while Dejak et al. [15] employed FEA to examine the influence of restoration flexural modulus and Poisson's ratio on the stress field generated in simulated crown models. The merits of such approaches implementing the FEA method to verify a novel technique for digitising all-ceramic restorations have also been demonstrated by Della Bona et al. [16]. The applications highlighted [13-16] were virtual case studies which employed the adaptive capabilities of the parametric simulation to assess the sensitivity of the model to discrete changes in the variables which define the model, including material properties (flexural modulus, Poisson's ratio), dimensional and/or geometric parameters. While these investigations are insightful, the usefulness of any FEA simulation is governed by the limitations of the specific features of the simulation model investigated. By implication, any features which have not been specified in the FEA model cannot manifest as determinants of the factors under investigation. Furthermore, the idealisations which are employed in the model may have a detrimental influence on the simulation by the failure to accurately represent the physical behaviour of the model itself.

For ceramic materials, the measured strength is understood to be dependent on the statistical distribution of defects of variable size, geometry and orientation within or at the surface of the body. Modelling the behaviour of such materials requires a probabilistic approach and whilst FEA methods can incorporate statistical elements, empirical measurements are essential to determine how geometric variability influences a statistical defect population in the fabricated complex shape.

The investigation of a curved specimen with a single radius of curvature has been proposed as a suitable model to examine the impact of geometric factors on radial fracture behaviour

[17]. However, the analytical solutions necessary to describe the stress distribution in the curved disc-shaped specimen tested in biaxial flexure are lacking in the scientific literature. Analytical solutions verified with an FEA simulation were proposed by Pecanac et al. [18] to determine the maximum tensile stress in curved bilayer disc specimens loaded in a ring-on-ring biaxial flexure strength testing configuration. The analytical solutions were limited, however, to radii of curvature of 170 mm which is not representative of the curvatures routinely encountered in partial or full contour dental restorations and therefore precluded their use in this study.

It is suggested that the influence of radii of curvature on the fracture behaviour can be assessed by mechanical loading of groups of specimens possessing simplified non-planar geometries (curved disc-shaped specimens) tested in biaxial flexure, in combination with individualised FEA models to subsequently determine the stress distribution. The aim of the study was to investigate the influence of non-planar geometries on the maximum tensile stress of curved (disc-shaped) specimens tested in biaxial flexure. Radii of curvature analogous to elements of complex dental geometries were of interest and the FEA method was integrated with the experimental testing as a surrogate solution to enable calculation of the maximum tensile stress generated at failure. The null hypothesis was that the maximum tensile stress is insensitive to radius of curvature was tested.

Materials and methods

Specimen preparation

Generic three-dimensional (3D) geometries of four curved disc-shaped specimens with different radii of curvature, were created with a parametric three-dimensional computer-aided design (CAD) system (Pro/Engineer, Wildfire 2.0, Parametric Technology Corporation, Needham, MA, USA). The 3D geometry was generated by sketching an axis of revolution and creating a parametric two-dimensional hemi-section of the curved disc-shaped specimen which was then revolved 360° about the axis of revolution to form a solid feature of the curved disc-shaped specimen. The nominal thickness and diameter specified of each curved disc-shaped specimen were at 1.0 and 12.0 mm, respectively with radii of curvature at 40, 30, 20 and 10 mm. Detailed drawings of the four curved disc-shaped specimen CAD geometries generated are shown in Figure 1 and were employed to design and manufacture the furnace mould tools used to fabricate the curved disc-shaped specimens (Advanced Glass Industries, Rochester, NY, USA) from flat soda lime glass stock (AGC Glass Europe, Brussels, Belgium). One hundred nominally identical flat soda-lime disc-shaped specimens (12.0 mm diameter, 1.0 mm thickness) were scribe-cut from the flat soda-lime glass stock, edged by chamfering and 20 discs were designated as the control (Group A) and remained flat (radius of curvature = ∞). Curvature was applied to the remaining scribe-cut flat discs by slump forming in a heated furnace mould to 575°C , rotated four times through 90° every 60 s to complete the slumping process. The curved discs were allowed to cool to ambient temperature at 1°C/s and disc-shaped specimens with radii of curvature of 40, 30, 20 and 10 mm were assigned to Groups B to E, respectively ($n=20$ for each group).

To provide a consistent and clinically relevant surface finish, one side of the flat discs (Group A) and the concave side of the curved discs (Groups B-E) were alumina particle air abraded with 50 μm alumina particles using a pressure of 4 bar, for 5 s (GOBI-2, Wassermann Dental-Maschinen GmbH, Hamburg, Germany). To minimise excessive localised abrasion, the nozzle of the alumina particle air abrader was maintained parallel to the central axes of the discs at 2 cm and traversed the surface in a smooth linear sweeping motion (Hooi et al., 2013). The disc-shaped specimens were acid-etched with 9.6% hydrofluoric (HF) acid (Porcelain Etch Gel, Pulpdent Corporation, Watertown, MA, USA) for 120 s, rinsed for 10 s with distilled water to remove the acid, prior to drying with oil free compressed air.

A random sample of three discs from each group were profilometrically evaluated on a stable bespoke multi-axis levelling device to determine the surface finish (Ra-value) and extract the radius of curvature. The radius of curvature was assessed diametrically in the x- and y-axes to calculate an average radius of curvature for comparison with the engineering design specification. Profilometric scanning was completed with an inductive gauge conisphere diamond stylus tip (2 μm radius) using a Talysurf CLI 2000 (Taylor Hobson Ltd., Leicester, UK). The displacement measurements were obtained across a rectangular area of 8 mm² (8 mm length and 1 mm width) coincident with the centre and extended radially to the periphery of the disc-shaped specimen. A total of 101 traces with a 10 μm step-size (y-direction) were performed at a stylus velocity of 1 mm/s and an applied load of 0.75 mN, recording datapoints every 10 μm (x-direction), with a 42 nm resolution (z-direction) from which the Ra-value and Wa-value were determined. The radii of curvature R were calculated in the x-direction and y-direction using an equation based on Heron's formula (Equation 1) using the distances between three points on the trace and an average radius of curvature determined.

$$R = \frac{abc}{4\sqrt{s(s-a)(s-b)(s-c)}} \quad \text{Equation 1}$$

where a , b and c are the distances between the three points, s is the semiperimeter defined by the three points and calculated using Equation 2.

$$s = \frac{a+b+c}{2} \quad \text{Equation 2.}$$

The central thickness of each flat and curved soda lime glass disc-shaped specimen (Groups A-E) was measured with a screw gauge micrometer fitted with a ball-nose adapter accurate to 1 μm (Mitutoyo Corporation, Tokyo, Japan) before being stored in a desiccator [19] prior to testing and analysis.

Biaxial flexure strength (BFS) testing

A ball-on-ring test configuration was used in the universal testing machine to determine the failure loads (N) of the flat (Group A) and curved soda lime glass disc-shaped specimens (Groups B-E). The disc-shaped specimens were tested with the roughened surface in tension. The discs were supported peripherally on a 10 mm diameter knife-edge ring support. A thin latex sheet (0.2 mm thickness) was also stretched over a 4 mm diameter ball indenter to diffuse focal contact stresses and the disc-shaped specimens were centrally loaded at a crosshead rate of 1 mm/min, where the load data acquisition was set at 2 ms intervals.

The maximum tensile stress at failure was calculated in the flat disc-shaped specimens using Shetty's [20] bending solution (Equation 3).

$$\sigma = \frac{-3P(1+\nu)}{4\pi} \left[1 + 2 \ln\left(\frac{a}{b}\right) + \frac{1-\nu}{1+\nu} \left(1 - \frac{b^2}{2a^2} \right) \frac{a^2}{R^2} \right] \quad \text{Equation 3}$$

where P was the measured load at failure (N), ν the Poisson's ratio (0.22) [21], t the specimen thickness, a , and R were the radii of the knife-edge support and the disc specimen and b was the radius of the contact zone which was the specimen thickness divided by 3 [22].

Without a closed-form analytical solution to calculate the biaxial flexure stress at failure in each of the curved soda lime glass disc-shaped specimens (Groups B-E), the maximum principal stress of each disc-shaped specimen at failure was determined by inputting failure loads into the FEA models individualized for each specimen ($n=80$). A two-dimensional axisymmetric FEA model for flat disc-shaped specimens previously validated for simulating the experimental ball-on-ring test [23] was adapted by the authors. The two-dimensional axisymmetric FEA model of the ball-on-ring test was generated using a linear P-code FEA software, Pro/MECHANICA (Wildfire 2.0, Parametric Technology Corporation, Needham, MA, USA). The three-dimensional CAD geometry of the four curved disc-shaped specimens specified with radii of curvature at 40, 30, 20 and 10 mm, were sectioned axially to create each respective two-dimensional axisymmetric finite element model. The boundary displacement constraints reflected the experimental test configuration whereby a frictionless ring support constraint was simulated by fixing the support nodes axially (vertical displacement) at the ring support radius while allowing free radial (horizontal) displacement.

The FEA was configured to use a maximum polynomial value of nine and convergence criteria of 3% for a specified measure parameter of maximum principal stress at an axial position ($r=0$) in the soda lime glass disc-shaped specimen on the surface in tension, in addition to the local strain energy. The measured central thickness, radius of curvature and failure load for each curved soda lime glass disc-shaped specimen tested were assigned to each model prior to running the analysis to determine the maximum principal stress at failure.

Eighty individual models were run to provide the maximum stress at the failure origin for each empirical measure.

Statistical Analysis

One-way ANOVAs ($P < 0.05$) were used to compare the mean of the measured radii of curvature and the mean surface roughness (Ra-value) of the alumina particle air abraded and HF acid-etched surfaces of each sample condition (Groups A-E) prior to strength determination. Post-hoc all paired Tukeys tests were also performed at $P < 0.05$ (SPSS for Windows 14, SPSS Inc, Chicago, USA). Additionally, the mean maximum principal stress and the mean load to failure with associated standard deviation data were plotted against the radius of curvature to examine the effect of geometry on the load to failure for the disc-shaped specimens (Figure 2). Further one-way ANOVA were performed at a 95% significance level to determine whether load to failure or strength data were significantly modified by the radius of curvature.

Results

Following alumina particle air abrasion and HF acid-etching the mean radii of curvature were 28.2 (1.6) mm, 19.86 (3.2) mm, 15.4 (0.4) mm and 8.25 (0.54) for the randomly selected disc-shaped specimens in Groups A to E, respectively and were determined using a one-way ANOVA to differ significantly between all groups (Table 1). The profilometric analysis of the disc-shaped specimens following surface modification identified mean Ra-values of 1.511 (0.053) μm , 1.383 (0.052) μm , 1.437 (0.129) μm , 1.449 (0.048) μm and 1.289 (0.041) μm for groups A-E respectively (Table 1). A one-way ANOVA and post-hoc Tukey tests demonstrated that discs with the minimum radius of curvature (Group E) had a significantly lower Ra-value (Table 1) than specimens from Groups A,C and D ($P < 0.008$). No other significant differences in the microscopic surface condition were detected. One-way ANOVAs further demonstrated that the magnitude of the load to failure was significantly modified by the radius of curvature of the disc-shaped specimens (Table 1, Figure 2). In contrast, no significant difference in the calculated maximum principal stress was detected when the radius of curvature was modified.

The actual radii of curvature determined from the profilometric analysis for the disc-shaped specimens in Groups A-E, respectively, were defined in the parametric 3D CAD geometry and the FEA model for each disc-shaped specimen tested. In addition, the FEA model included the measured central thickness and load at failure for each curved disc-shaped specimen investigated to determine the maximum principal stress resulting from the applied load at failure (Figure 3) and the group mean data compared (Table 1). The mean maximum principal stress and the mean load to failure with associated standard deviation data for each group were plotted against the radius of curvature to examine the influence of geometry on

the fracture resistance of the soda lime glass disc-shaped specimens (Figure 2). The plotted data demonstrated that as the radius of curvature was reduced, the load to failure was increased, in contrast to the pattern evident for the maximum principal stress which indicated the more curved geometry increased the load to failure without modifying the maximum tensile strength determined with the FEA simulation for the disc-shaped specimens.

Discussion

The testing of planar disc-shaped specimens in biaxial flexure has been widely employed to investigate the fracture behaviour of dental ceramic materials [24-26]. FEA simulations [27] and quantitative fractography [28,29] of failed all-ceramic restorations has identified that clinical failure often manifests by the tensile extension of flaws on the intaglio surface. The observed clinical failure mode of radial fracture failure is reproduced with the BFS test [30], however, it has been suggested that the fracture behaviour of a flat planar form specimen is not representative of the geometric complexities evident in full-contour restorations [14].

In the absence of an analytical solution for the curved disc-shaped specimen tested in biaxial flexure, the FEA method was implemented as a surrogate solution to determine the stress state in the individual nominally identical curved disc-shaped specimens with different radii of curvature. The approach although laborious accounted for both the geometric influence in stressing pattern and the unique defect distributions in each of the tested specimens.

A parametric two-dimensional axisymmetric FEA model of the ball-on-ring BFS test for flat planar disc-shaped specimens was established and demonstrated to minimally diverge (3.3%) from Shettys bending solutions for equivalent geometry [23]. The parametric FEA model was conducive to shape modification by varying the radius of curvature and layer thickness of the individual disc-shaped specimen tested in biaxial flexure which regenerated seamlessly within the integrated FEA software. The stress distribution was examined for the disc-shaped specimens with different radii of curvature. The experimentally determined load to failure data avoided the necessity to incorporate an arbitrary failure criterion in the linear FEA simulation. The accuracy of the FEA simulation was demonstrated by the convergence of the

solution to within 3% for a maximum polynomial order of six in identifying the maximum tensile stress. The reduction in the radius of curvature (effectively more severe curvature) was observed to impact on the stress distribution in the curved disc-shaped specimens tested in biaxial flexure. Significantly increased loads to failure were observed in BFS testing for equivalent disc thicknesses when compared with flatter specimens. However, the reduction in the radius of curvature was not observed to significantly impact the maximum tensile stress calculated at failure.

The pronounced sensitivity of the load to failure to changes in the curvature of the specimen is definitive evidence of the problems associated with the comparison of the widely reported load to failure data [1-8]. Whilst expert opinion has frequently been provided challenging the use of such techniques, ‘crunch-the-crown’ data is consistently published within the dental literature [1-8]. Specimens employed in ‘crunch-the-crown’ testing contain multiple geometric features to represent the full-contour restoration under investigation. It is reasonable to expect that the opportunity for deviations between specimens is increased with added geometric complexity in the absence of stringent quality controls between specimens, batches and ultimately between studies for comparison. The current investigation highlights that load to failure testing can be performed when complimented by numerical simulations that are specimen specific and where specimen failure is determined to be representative of the clinically operative failure mechanism. However, even for a simplistic model the sensitivities to shape variation between individual specimens from an ‘identical’ series is high. The fabrication of the nominally identical disc-shaped specimens with a single radius of curvature with a standardised protocol was monitored for consistency in the current study by profilometric inspection to verify the specified dimensional and geometric parameters were

controlled and any identified deviations from the specifications were factored into the simulation models.

The findings demonstrated that the adapted flat planar solution adequately described the failure stress of all curved structures in the radii examined. The surface state responsible for the specimen at failure was assumed to be equivalent for all specimen groups given that a common surface treatment was undertaken. However, for specimens with the smallest radius of curvature, significant differences in the microscopic surface topography were observed and were assumed to result from the complexities of an abrasive particle interactions with the curved surface. Although this raises a cautionary note about the accuracy of the measured values, it highlights further the need for modelling more complex geometries where identical and clinically relevant experimental treatments may yield different results.

Conclusion

The current study effectively demonstrated the complications associated with interpreting load to failure data and provides support for the relevance of solutions derived from bending theory. In addition, the data supports the synergistic use of bespoke computational analysis with empirical data collection.

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Group	Mean Radius of Curvature [mm]	Mean Thickness [mm]	Mean Load to Failure [N]	Mean Maximum Principal Stress [MPa]	Mean Ra-value [μm]
A	Flat	0.93 (0.01) ^e	40.6 (3.5) ^g	97.7 (8.0) ^k	1.511 (0.053) ^m
B	28.21 (1.62) ^a	0.92 (0.02) ^e	46.0 (6.0) ^{gh}	98.7 (11.1) ^k	1.383 (0.052) ^{no}
C	19.86 (3.20) ^b	0.93 (0.02) ^e	50.8 (5.9) ^h	98.9 (9.8) ^k	1.437 (0.048) ^{mn}
D	15.43 (0.44) ^c	0.93 (0.02) ^e	64.1 (5.4) ⁱ	112.8 (9.2) ^l	1.449 (0.048) ^{mn}
E	8.25 (0.54) ^d	1.05 (0.08) ^f	93.7 (15.5) ^j	105.0 (15.4) ^{kl}	1.289 (0.041) ^o

Table 1: The mean radius of curvature, mean sample thickness, mean load to failure in BFS testing, mean maximum principal stress calculated using the FEA method and mean Ra-values for the flat (Group A) and curved (Groups B-E) soda lime glass disc-shaped specimens examined. One-way ANOVAs and post hoc Tukey tests were employed and different superscript denominators within columns denotes significant differences (P<0.05).

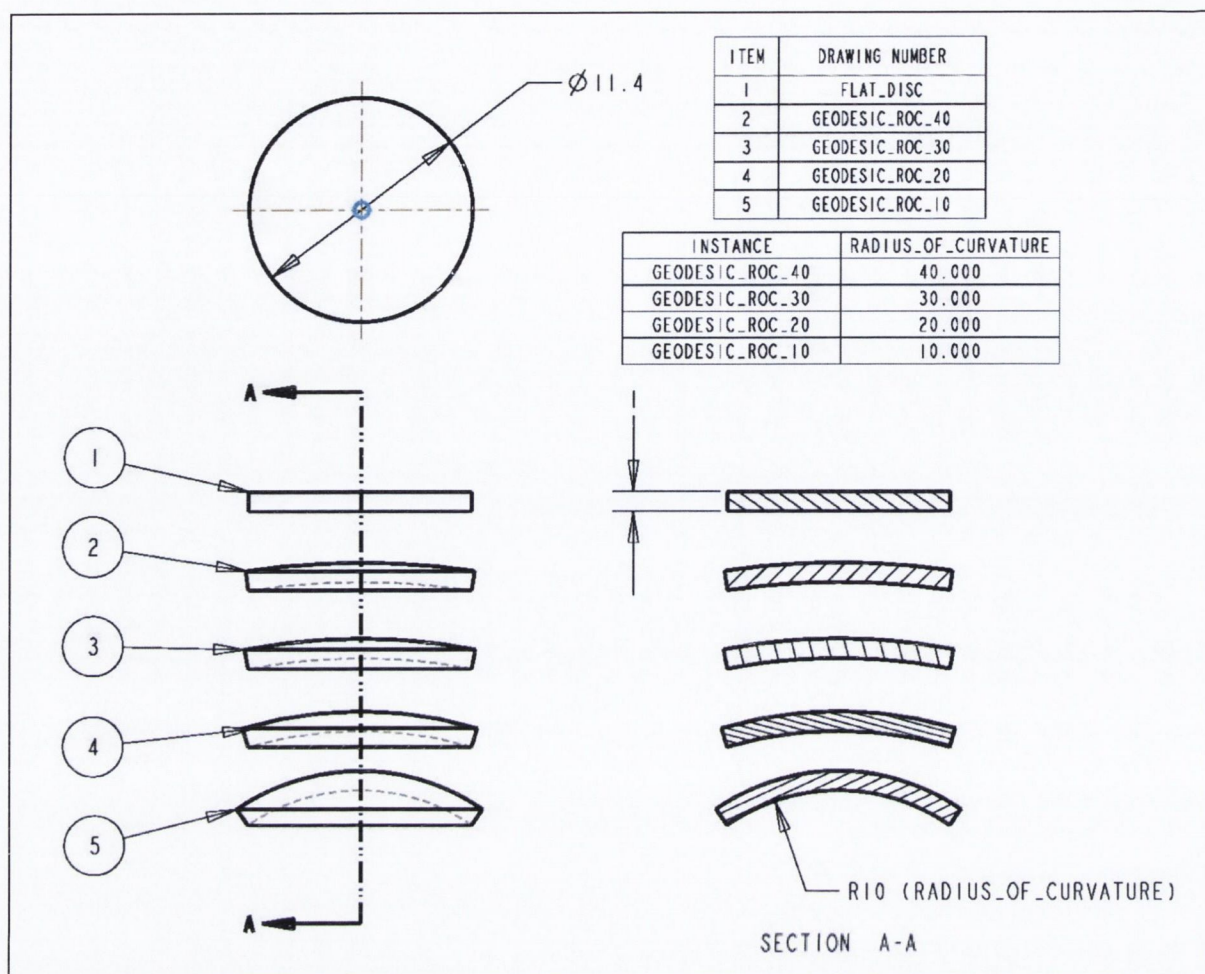


Figure 1: Two-dimensional drawing of the flat and curved disc-shaped specimens. The disc-shaped specimens are shown from top (Item 1) to bottom (Item 5) with decreasing radius of curvature (flat, 40, 30, 20 and 10 mm for items 1 to 5, respectively). The section view taken through the axes of the disc-shaped specimens in the front view shows the curvature through the centre of each specimen.

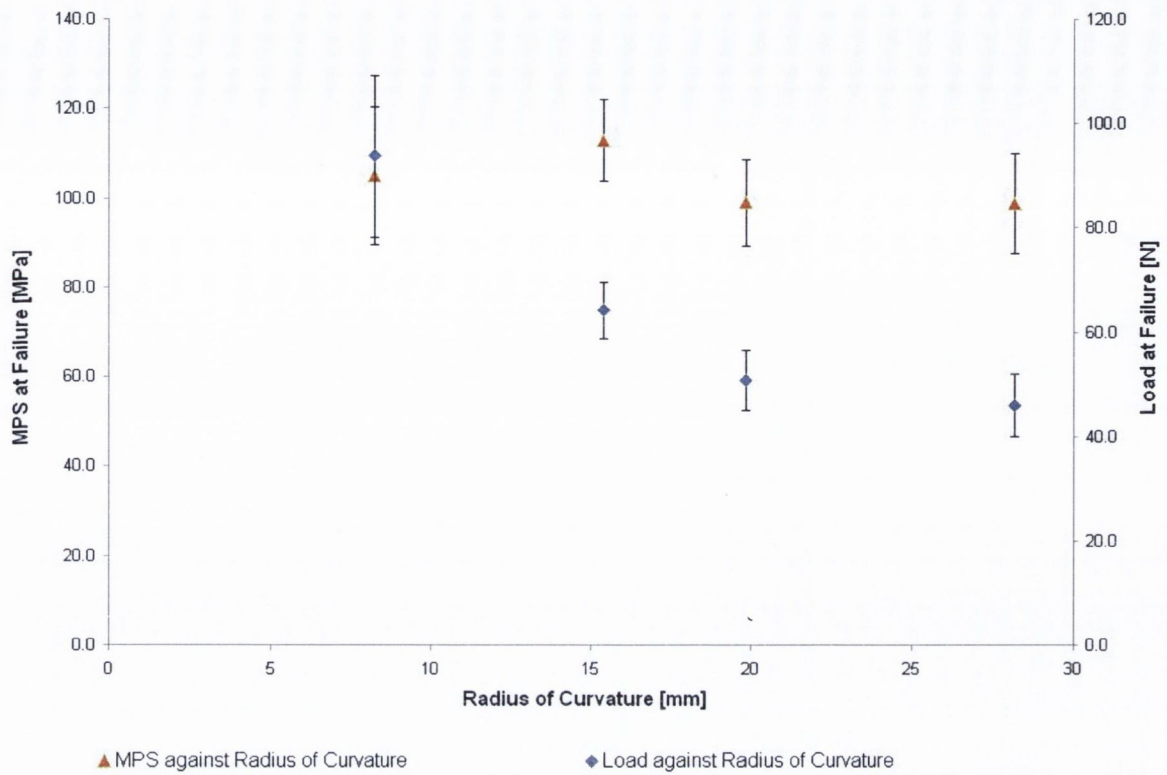


Figure 2: The plot of mean maximum principal stresses and the mean loads to failure data against the radius of curvature and respective standard deviations highlighted the increased load at failure for reduced radius of curvature. The maximum principal stress was not sensitive to the different radii of curvature.

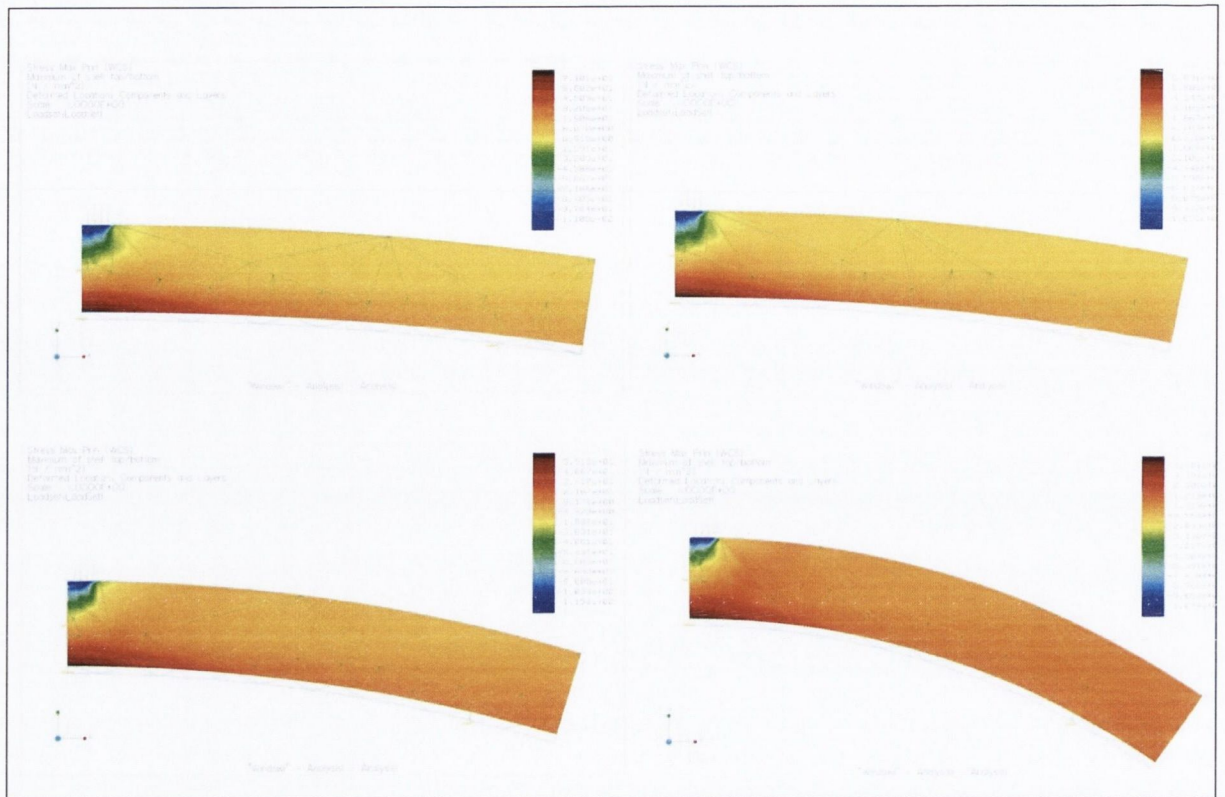


Figure 3: The two-dimensional axisymmetric finite element analysis result showing the contour plot of the maximum principal stress in the soda lime glass curved disc-shaped specimen for each radii of curvature, starting with the least curved disc-shaped specimen shown in the top left (radius of curvature = 28.21 (1.62) mm) to the most curved disc-shaped specimen shown in the bottom right (radius of curvature = 8.25 (0.54)). The maximum principal stress was identified on the bottom surface of soda lime glass at the centre of the specimen ($r=0$).

4. GENERAL DISCUSSION

4.1 THE CLINICAL PROBLEM

The aesthetic quality of PLV and DBC feldspathic dental porcelain restorations remains unrivalled in meeting the patient demands for cosmetic restorative dentistry yet the incidence of premature restoration failure continues to inconvenience both the patient and clinician in terms of time and the associated cost. Fracture of PLV and DBC restorations can result in the partial or total loss of the restoration which would be immediately apparent to the patient and requires prompt intervention. However, less obvious fracture patterns or the development of fracture lines may be more problematic as the bulk restoration may remain intact and subsequent microleakage could lead to dental caries (Lutz et al., 1991). The presence of dental caries (Lutz et al., 1991; Uno and Asmussen, 1991) and the associated biological complications secondary to microleakage (Eick and Welch, 1986; Uno and Asmussen, 1991) could delay and therefore compromise the efficacy of treatment. In describing the clinical failure of PLV and DBC restorations it is therefore important to define and standardise the operative/technical variability; to identify the failure mode and the contributing aetiological factors (Anusavice, 2012). Following the clinical assessment of a failed all-ceramic restoration the absolute reason for the failure may not be immediately apparent to the practitioner. However, if no attempt is made to understand the cause and origin of the failure, then the clinical prognosis following re-intervention may be further compromised by repeat failures and therefore the opportunity to optimise the clinical outcome missed. The cause and origin of the failure may be patient, operator or material dependent. Patient-specific (trauma, poor oral hygiene), iatrogenic-specific (poor retention and resistance form of the tooth preparation, impression technique) or material-specific (internal porosity within the

restoration, poor choice of luting agent) factors could act in isolation or in combination resulting in a multi-factorial aetiology.

The continued loading of PLV and DBC restorations could potentiate the tensile extension of pre-existing flaws on the intaglio surface of the restoration and typically results in fracture of the restoration into two fragments (Kelly, 1999). The resultant tensile stresses may be a consequence of normal functional occlusal forces or parafunctional loading, both of which need to be accounted for when designing the occlusal schema of the restoration. The form of the prepared tooth may therefore impact on the resultant clinical performance of the restoration with the presence of sharp line angles and poor margin design potentiating unfavourable stress concentrations (Magne and Douglas, 1999; Castelnovo et al., 2000; Bernal et al., 2003; Qasim et al., 2006). The extent of tooth reduction will determine the full contour dimensions of the restoration and there exists an optimum thickness which minimises the stress distribution within the restoration under load (Wakabayashi and Anusavice, 2000; Thompson, 2000; Fleming et al., 2005; Sornsuan and Swain, 2012). Alternatively, defects would be expected in ceramic restorations following manufacture by the dental technician which could potentiate fracture. It is however only following visual inspection of the fracture fragments that obvious bulk defects such as porosities or surface flaws which are known to cause stress concentrations in all-ceramic restorations can be identified.

The development of high strength core ceramic materials which are subsequently veneered with an aesthetic porcelain has been an approach undertaken by dental material manufacturers in an attempt to improve the clinical performance of all-ceramic restorations. However, the increased thickness of the full contoured bilayered restoration comes at the expense of the sacrifice of sound tooth structure. With the aggressive reduction of tooth

structure there is diminished bulk core support for the restoration and an increased risk of pulpal irritation. Aggressive tooth preparation also involves utilising instrumentation in close proximity to the pulp where the wider dentinal tubules are more susceptible to physiological damage (Pashley, 1990) and ultimately loss of vitality results in up to 13% of prepared teeth (Abou-Rass, 1982; Bergenholtz and Nyman, 1984; Felton et al., 1989; Reichen-Graden and Lang, 1989; Gonzalez et al., 1991; Saunders and Saunders, 1994). Furthermore, the veneered high strength ceramic core crown structure is susceptible to porcelain veneer chipping fracture (Cehreli et al., 2009; Ortorp et al., 2009; Al-Amleh et al., 2010; Groten and Huttig, 2010; Anusavice, 2012). Techniques to repair chipped porcelain remain unsatisfactory (Kimmich and Stappert, 2013) and the alternative is to remove and replace the restoration but the increased toughness of the high strength ceramic core (Rizkalla and Jones, 2004) is problematic when sectioning the restoration with conventional dental instrumentation (Park et al., 2006; Choi et al., 2010).

Monolithic PLV and DBC restorations fabricated with aesthetic feldspathic dental porcelains or aesthetic glass ceramics are biologically more favourable to tooth structure due to the minimal tooth reduction involved in the conservative tooth preparation. The clinician should endeavour to deliver the maximum reinforcement for the all-ceramic restoration by employing the optimum pre-cementation conditioning regimen and resin-cementation technique when adhering the restoration. With the appropriate pre-cementation surface conditioning and resin-cementation technique, the maximum reinforcement can be achieved (Addison et al., 2007a-d, 2008) with the potential to optimise the clinical performance (Malament and Socransky, 1999ab, 2001). It should be noted that the intaglio surface is most vulnerable to tensile stress induced as a result of external occlusal load (Kelly, 1999). The tensile stress field imposed across the intaglio surface interacts with the microscopic flaw

population which exists as a result of the manufacturing process and cracks may propagate which could result in failure (Griffith, 1921). Therefore, it is hardly surprising that modification of surface topography by pre-cementation operative techniques (alumina particle air abrasion and HF acid-etching) can impact upon the strength of dental ceramic restorative materials (De Jager et al., 2000; Addison et al., 2007a-c).

The routine clinical approach employed is to roughen the intaglio surface of the ceramic restoration to improve adhesion to the cement and optimise the synergism (McLean, 1988) between the restoration and the supporting tooth structure mediated by the cement (Fleming et al., 2006). Resin-based luting materials are commonly employed to optimise the retention of the restoration (Lacy et al., 1988; Burke et al., 1998; Ozcan and Vallitu, 2003) and have been associated with a strengthening effect (Rosenstiel et al., 1993; Pagniano et al., 2005; Addison et al., 2008) and enhanced clinical performance (Malament and Socransky et al., 1999a,b; 2001; 2010). Although the strengthening mechanism is not fully understood, evidence exists to suggest that a resin-ceramic hybrid layer is operative in mediating the observed resin-reinforcement (Addison et al., 2008). Furthermore, static fatigue effects due to water exposure (Kingery, 1976a) have been demonstrated to evolve from external cone cracks in monolithic ceramic dental crowns with a significant glass phase (Lee et al., 2002). However, the impact of atmospheric moisture exposure to the intaglio surface on the pattern of resin-strengthening has not been significantly assessed in the dental literature. Additionally, attempts to investigate the influence of geometry on the performance of all-ceramic restorations have challenged the relevance of employing flat planar geometric specimens. The current investigation aimed to examine the impact of pre-cementation surface treatment and resin-cementation operative variables on the strength of dentine porcelain disc-shaped specimens. An analogue material for dentine porcelain was identified and validated

for the investigation of testing rate effects and the influence of geometric curvature on the strength was further determined.

4.2 PRE-CEMENTATION OPERATIVE VARIABLES

The magnitude of resin-strengthening has been demonstrated to be dependent on the interpenetration of the resin with the ceramic surface defect population to form what has been termed as the resin-ceramic hybrid layer (Addison et al., 2007d). The magnitude of reinforcement achievable was therefore a function of the ceramic surface texture (Bhamra et al., 2002; Fleming et al., 2006; Addison et al., 2007d; Addison et al., 2008), but also dependent on the flexural modulus of the RBC material (Fleming et al., 2006; Addison et al., 2007d) and the resin-coating thickness (Addison et al., 2008). The manufacture of PLV and DBC restorations has been demonstrated to introduce defects of varying size and distribution (Fleming et al., 2000). These defects can be introduced during specimen fabrication (Gill, 1932; Fleming et al., 2000) and influenced by the technical skill of the operator (Piddock et al., 1984; Fleming et al., 2000) through specimen sintering or heat-pressing techniques (Isgro et al., 2010) or even CAD-CAM routes (Addison et al., 2012). While optimal condensation (Gill, 1932; Fleming et al., 2000) and sintering techniques have been identified for various materials that minimise defect formation (Fleming et al., 2000), pre-cementation conditioning of the intaglio surface of PLV and DBC restorations is routinely performed in the clinical setting (Calamia, 1985; McLean, 1988) and can dramatically modify the surface defect population.

The clinical techniques routinely employed to condition the intaglio surface of the all-ceramic restorations include alumina particle air abrasion and hydrofluoric (HF) acid-etching. The

efficacy of both techniques to favourably modify the intaglio surface has been demonstrated (Ozcan and Vallitti, 2003) and the techniques are routinely utilised in combination. The alumina particle air abrasion process modifies the surface characteristics by the volumetric removal at the surface with the introduction of a new defect population (Addison et al., 2007a; Isgro et al., 2010). The HF acid-etching process results in surface dissolution, dependent on the composition and fraction of crystalline and glassy phases, however, in minimally crystalline materials etching can result in the extension of pre-existing surface defects (Yen et al., 1993; Addison et al., 2007b). It is important to note that whilst there have been extensive efforts to maximise adhesion between resin-cement and ceramic surfaces, the impact of such modification on strength is rarely considered.

The alumina particle air abrasion or HF acid-etching pre-cementation conditioning regimes were employed in isolation or combination throughout the current study (Manuscripts 3.1 to 3.5). Standardisation of the surface conditioning protocols was considered important for the comparison of data within the current study and between previous studies in the dental literature. Alumina particle air abrasion parameters include the different grades of alumina particles, air stream delivery pressure and angle of incidence of particle delivery (Addison et al., 2007a) while the HF acid concentration and etching time have been demonstrated to variously influence the resulting surface condition (Addison et al., 2007b).

4.2.1 Alumina particle air abrasion

Alumina particle air abrasion and HF acid-etching are laboratory/chair-side techniques routinely used to condition the intaglio surface of all-ceramic restorations prior to the cementation. The toxicity of HF acid is well documented in the medical literature (Caravati, 1988; Bertolini 1992; Kirkpatrick et al., 1995) although the incidence of adverse events is

rarely documented in the dental literature (Kimmich and Stappert, 2013). Particular concern regarding the safety profile of HF acid arises when it is employed for the intraoral repair of fractured porcelain, where adjacent soft tissue and the close proximity of enamel and dentine risk unintentional exposure with the acid during the clinical procedure. Given the safety concerns associated with HF when employed in dentistry (Lacy, 1989), alumina particle air abrasion is considered to be less hazardous and more preferable from a health and safety perspective.

The alumina particle air abrasion technique involves the removal of the surface layer of the ceramic and the introduction of a new surface defect population, such that micromechanical retention is enhanced. The alumina particle air abrasion of dental porcelain has been demonstrated to increase the surface energy which improves the ability of a luting material to wet the surface and thereby achieve increased interpenetration with the asperities over the surface area (Oh et al., 2002). This is reported to result in an improved adhesive bond between the alumina particle air abraded dental porcelain surface and luting materials (Wolf et al., 1993; Rungruanganunt and Kelly, 2012). Furthermore, while there is established evidence to demonstrate the primary efficacy of alumina particle air abrasion to enhance bonding, a secondary impact of surface modification on the fracture strength of dental ceramics has been reported (Addison et al., 2007a). A comprehensive investigation of the impact of the alumina particle air abrasion parameters, namely different grades of alumina particles, air stream delivery pressure and angle of incidence of particle delivery, on the BFS of dentine porcelain may lead to the identification of optimum protocols for each material under investigation (Addison et al., 2007a). For the purposes of this enquiry, the optimum alumina particle air abrasion protocol was determined by examining the surface roughness (Ra-value) to establish if the macroscopic defect population was consistent, in addition to

assessing the reliability of the BFS data (Addison et al., 2007a) giving insight into the consistency of the critical defect population.

In the current study, the optimum alumina particle air abrasion protocol reported by Addison et al. (2007a) was employed to introduce a new and homogenous surface defect population on the dentine porcelain disc-shaped specimens examined in Manuscript 3.1. While the surface roughness (Ra-value) was not reported in Manuscript 3.1, the Ra-value was recorded (2.749 (0.316) μm) and was similar to the Ra-values reported by Addison et al. (2007a) for the same material following an identical alumina particle air abrasion protocol of 2.770 (0.378) μm . Furthermore, the alumina particle air abraded dentine porcelain disc-shaped specimens were determined to have a mean BFS and associated standard deviation of 58.0 (4.0) MPa (n=25) in Manuscript 3.1. This is consistent with the previously reported BFS data in the literature for the same material under the same alumina particle air abrasion regimen, namely 58.9 (4.2) MPa (n=30) (Addison et al., 2007a), 58.0 (4.7) MPa (n=60) (Addison et al., 2007d) and 57.0 (4.0) MPa (n=30) (Addison et al., 2008). The same dentine porcelain powder and modelling liquid were employed (although from different batches), to condense the dentine porcelain disc-shaped specimens (at a standardised powder to liquid mixing ratio) and the manufacturers recommended firing schedule was maintained. The mean BFS values should not be significantly different between studies as has been demonstrated. It is suggested that the minimal differences in the BFS data reported are most likely the result of the potential for operator induced variability in the manual delivery of the alumina particle air abrasion to modify the macroscopic surface features of geometric form and waviness. In an effort to negate this impact, all disc-shaped specimens were alumina particle air abraded where the abrasion technique was performed in a smooth sweeping motion to prevent excessive localised abrasion for the prescribed time period. The destructive capability of

alumina particle air abrasion has been reported whereby volumetric removal, subsurface damage and surface chipping are possible (Darvell, 2002). However, the selected alumina particle air abrasion protocol balanced the increased surface area available to enhance the adhesive bond (Oh et al., 2002) with the reported adverse reduction in the BFS associated with a rougher surface (Griffith, 1921; Addison et al., 2007c).

To avoid the sample preparation problems associated with inconsistent dentine porcelain disc-shaped specimens, where a variability in the measured strength can be introduced during specimen fabrication (Gill, 1932; Fleming et al., 2000), specimen sintering or heat-pressing (Isgro et al, 2010), CAD-CAM routes (Addison et al., 2012) and the technical skill of the operator (Fleming et al., 2000; Piddock et al., 1984), an analogue material was chosen as an experimental model. Soda lime glass was selected as the analogue material for dentine porcelain enabling reliable, nominally identical samples to be produced (Manuscript 3.2). However, since the clinically relevant failure mode of PLV and DBC restorations is dominated by the tensile extension of pre-existing surface flaws which results in radial fracture, the influence of surface condition must be analogous to the previously reported data for dentine porcelain. The assessment of the 'as-cut' surfaces of the soda lime glass disc-shaped specimens reported in Manuscript 3.2 revealed geometrically flat surfaces which were highly polished (Ra-value of 0.68 (0.29) μm) and optically transparent with surface defects of minimal crack length with associated mean BFS (118.7 (13.8) MPa). The alumina particle air abrasion protocol was employed for the soda lime glass disc-shaped specimens to introduce a new and homogenous defect population which provided a more clinically relevant and controlled surface texture than the 'as-cut' surface. The surface roughness (Ra-value) and surface waviness (Wa-value) determined for the alumina particle air abraded soda lime glass disc-shaped specimens were reported to be 3.26 (0.166) μm and 5.726 (1.3) μm , respectively.

The Ra-value was increased when compared with the Ra-value for the dentine porcelain (2.749 (0.316) μm) but the surface waviness (Wa-value) could not be compared because the parameter was not reported for the dentine porcelain. It was expected that differences in surface texture would manifest from the compositional variation between the soda lime glass and the dentine porcelain when employing an identical alumina particle air abrasion protocol for the soda lime glass disc-shaped specimens. The soda lime glass contained no crystalline phase while the dentine porcelain contained up to 10% alumina. In addition, the flexural modulus, toughness and the hardness have been reported to impact on the surface texture following alumina particle air abrasion (Darvell, 2002). The flexural modulus of the soda lime glass (40 GPa) determined in Manuscript 3.2 was considerably reduced when compared with the flexural modulus of the dentine porcelain (64 GPa) reported by Zeng et al., (1998). Similarly, a reduced Vickers hardness for the soda lime glass (5.78 ± 0.13 GPa) has been reported (Bousbaa et al., 2003) when compared with the dentine porcelain (7.14 ± 0.41 GPa) (Cesar et al., 2007). Following alumina particle air abrasion the group mean BFS and associated standard deviation of the soda lime disc-shaped specimens was determined to be 44.4 (4.8) MPa and the low standard deviation was indicative of a consistent surface defect population (Manuscript 3.2).

4.2.2 Hydrofluoric acid-etching

The surface conditioning of PLV and DBC restorations with HF acid is another technique employed to roughen the surface and enhance the adhesive bond with the luting material prior to cementation. Although alumina particle air abrasion or HF acid-etching can be utilised in isolation, both methods are routinely combined sequentially by the operator in an attempt to optimise the surface texture (Ozcan and Vallittu, 2003). While the safety profile of HF acid is an important concern for both the patient and the operator, the lack of evidence to contra-

indicate its use in dentistry, together with the established clinical efficacy in pre-cementation surface conditioning ensure its continued use clinically and especially pre-operatively in laboratory techniques (Özcan et al., 2012). Dental ceramic materials which contain a significant glass phase have been shown to be susceptible to dissolution by HF acid. When the ceramic microstructure contains both glassy and crystalline phases, HF acid results in a surface more conducive to wetting and adhesive bonding with luting materials (Horn, 1983). Therefore, as the crystalline component is more resistant to degradation by HF acid (Della Bona and Anusavice, 2002) the potential dissolution of the glassy phase leads to a significant increase in surface roughness. The influence of etching a feldspathic porcelain or glassy dental ceramic specimen with HF acid is more variable resulting in generalised surface dissolution but also enhanced corrosion at pre-existing defects (Yen et al., 1993). The overall surface texture and the observed strength which results from HF acid-etching of commercially available silicate glass is characterised by the modification of pre-existing surface defects, crack tip blunting and even complete removal of the crack resulting in a strengthening effect (Kobayashi, 1962; Pavelchek and Doremus, 1974; Kolli et al., 2009). This finding is not completely compatible with the increased strengthening of dental porcelains where HF acid-etching has been demonstrated to both strengthen when the surface contains large defects and weaken when the initial defect population contains only small flaws.

While it is evident that material composition influences the interaction between the substrate and the HF acid, the application of HF acid by the operator is routinely indiscriminate and non-specific in terms of the parameters which define the procedure. The pre-existing surface texture, the HF acid-etch concentration and HF acid-etching time have been demonstrated to variously influence not only the resulting surface condition, but also the BFS and reliability

of the BFS data (Addison et al., 2007b) when a dentine porcelain material was employed. The relationship between the HF acid-etch parameters and the interaction with the surface defect population was reported to be complex and Addison et al. (2007b) demonstrated that pre-existing defects were variously modified by the HF acid, in contrast to alumina particle air abrasion where a new defect population was introduced. The alumina particle air abrasion procedure provided the desired surface roughness conducive to enhanced bonding (Ra value of 2.770 (0.378) μm) with associated BFS data (58.0 (4.0) MPa) for the dentine porcelain investigated in Manuscript 3.1 and therefore HF acid-etching was not employed.

The alumina particle air abrasion protocol employed for the soda lime glass disc-shaped specimens (Manuscript 3.2) was followed by a HF acid-etch protocol identified by Addison et al. (2007b). Following HF acid-etching no significant difference was observed in the surface roughness (Ra-value of 3.21 (0.14) μm) when compared with alumina particle air abrasion (Ra-value of 3.26 (0.166) μm). However, a significant increase in the surface waviness (Wa-value of 8.15 (2.93) μm) was identified compared with the alumina particle air abraded soda lime glass disc-shaped specimens (Wa-value of 5.726 (1.3) μm). The amplitude of the larger surface features imposed by alumina particle air abrasion were therefore preferentially increased and superimposed over longer wavelengths. This finding is consistent with the investigation of Addison et al. (2007b) where a favourable HF acid-etching regimen (concentration and etching time) was identified to enhance the mechanical performance of a dentine porcelain PLV material.

The alumina particle air abrasion procedure followed by the HF acid-etch step achieved a significant increase in mean BFS (52.6 (5.5) MPa) compared with the alumina particle air abrasion in isolation (44.4 (4.8) MPa). This finding confirms what was already known,

namely that the strengthening of soda lime glass occurs following HF acid-etching (Dabbs and Lawn, 1982; Saha and Cooper, 1984; Sglavo et al., 1993). This was important in establishing the control data to validate the assumption that soda lime glass was an analogue material for dentine porcelain since the pattern of strengthening observed in Manuscript 3.2 was consistent with the previously reported data for the dentine porcelain (Manuscript 3.1). Therefore we suggest that to evaluate the mechanism of resin-strengthening, the employment of soda lime glass disc-shaped specimens could be used (Manuscripts 3.3 to 3.4) to simulate the behaviour routinely observed clinically in PLV or DBC restorations.

4.2.3 Silane coupling agent

Silane coupling agents have been available for over 50 years and are frequently used in dentistry to prime the intaglio surface of all-ceramic restorations prior to resin-cementation. In addition, silane coupling agents have been reported to enhance the bond between the RBC materials and ceramic materials containing a significant glass phase (Lung and Matinlinna, 2012). The bi-functional silane molecule mediates and promotes adhesion between the organic RBC material and the inorganic silicate glass phase of the restoration. However, the clinical efficacy of the resin-ceramic bond was identified to be inadequate when dental ceramics which did not contain a significant silicate glass phase were employed (Matinlinna, 2004). It has previously been reported, for the dentine porcelain used in Manuscript 3.1, that the application of a silane coupling agent, prior to adhesive bonding with an unfilled resin, was associated with no significant increase in mean BFS (Addison et al., 2007c). However, the application of a silane coupling agent prior to cementation using a filled resin resulted in a 20% increase in the mean BFS value reported (n=30) for the dentine porcelain investigated (Addison et al., 2008b). As a result, the application of a silane coupling agent to the dentine porcelain surface prior to resin-cementation was undertaken in the current study (Manuscript

3.1). Silane coupling agents have been widely demonstrated in industrial applications to enhance adhesive bonding to soda lime glass (Ritter 1974; Blum et al., 1990; Hand et al., 1998a,b; Ritter et al., 2001). Therefore, when soda lime glass disc-shaped specimens were used in the current study (Manuscripts 3.2 to 3.4) the treated surfaces (alumina particle air abraded and HF acid-etched) were also treated with the silane primer to optimise resin-strengthening by enhancing the chemical adhesion.

4.3 RESIN-STRENGTHENING REVIEW

In 1999, the 14 year findings of a comprehensive prospective clinical study into the variables determining the survival of 1444 restorations fabricated from Dicor - a fluoride containing tetra silicic glass ceramic substrate were reported by Malament and Socransky (1999a,b). The authors demonstrated the glass ceramic restorations were significantly more resistant to fracture when adhesively bonded to the prepared tooth structure using resin-based materials compared with when luted with acid-base cements (Malament and Socransky, 1999b). On further examination of the data reported, the 14 year survival rate of the glass ceramic restorations which were acid-etched prior to bonding to the tooth structure was 74% compared with 55% for non-acid-etched glass ceramic restorations. Additional information gleaned from the Malament and Socransky studies (Malament and Socransky, 1999a,b, 2001, 2010) was that the relative risk of failure of Dicor glass ceramic restorations luted with zinc phosphate cements was 2.1 times that for restorations adhesively bonded using the resin-based materials available in the early 1980s. Further evidence for the use of adhesive technologies to support dental ceramics was evident in the results of a 6-year follow-up of 115 fired ceramic inlays reported by van Dijken et al. (1998). In the study, 3 of the of the 58 inlays luted with a resin-based material suffered partial or total fracture compared with 16 of

the 57 inlays luted with a glass-ionomer cement providing further clinical evidence for the use of resin-based adhesive technologies to support dental ceramics.

The clinical outcomes reported by the Malament and Socransky (1999a,b) and van Dijken et al. (1998) data supported the proposals made in 1992 by Marquis and in 1993 by Nathanson (1993) that adhesive cementation strengthens feldspathic porcelain and dental glass ceramic restorations (Marquis, 1992; Nathanson, 1993). Although the two independent investigators provided different hypotheses for the exact resin-strengthening mechanism, both authors agreed that the interaction of the resin-based material with the critical flaws on the ceramic surface conferred the observed strengthening (Marquis, 1992; Nathanson, 1993). Over the last decade there has been a significant increase in the use of PLV and DBC restorations (Burke et al., 1998) which combine conservative tooth preparation ideals with adhesive bonding strategies to aesthetic but relatively weak feldspathic dental porcelains (McLean, 1988). The clinical success of these PLV and DBC restorations fabricated from feldspathic porcelains or glass ceramics is remarkable given that failure rates in the anterior dentition do not differ dramatically from restorations constructed from ceramic substrates with up to ten-fold increased flexural strengths (Burke et al., 2002).

Despite the survival rates of PLV and DBC restorations clinically, the universal adoption of adhesive cementation technologies to support all-ceramic restorations by practitioners has been slow and is contrary to the evidence based-literature. Different luting materials have been indicated as luting agents for fixed prosthodontics including zinc phosphate, zinc polycarboxylate, glass-ionomer, resin-modified glass-ionomer cements, and resin-based luting materials (Diaz-Arnold et al., 1999). The problem with the acid-base cements (Wilson and Nicholson, 1993) was the strength of dental ceramic materials is susceptible to an acidic

environment. Smith and Ruse (1986) highlighted for a zinc phosphate cement that during the initial stages of setting the environment at the crack tip may spend up to 15 minutes at a pH level of lower than 4.0. Unfortunately, the manufacturers recommended zinc phosphate powder to liquid mixing ratio routinely mixed by dental surgery assistants in the clinical setting is achieved only 30% of the time with the remaining 70% of luting materials having substantially less powder contents (Fleming et al., 1999). As a result, zinc phosphate cements mixed in a clinical setting will therefore spend an increased time period at pH levels less than 4.0 such that the performance of cemented all-ceramic restorations is influenced by the cement manipulation regime (Fleming et al., 1999). Billington et al. (1991) identified that the powder to liquid mixing ratio routinely used in dental clinics for glass-ionomer cements is similarly influenced by operator induced variability. However, the acidic component polymers or copolymers of acrylic or maleic acid in the glass-ionomer cement is not as strong as phosphoric acid in a zinc phosphate dental cement. Additionally, the acrylic acid backbone is bulky suggesting mobility is difficult but nonetheless, the performance of glass-ionomer and zinc polycarboxylate cemented all-ceramic restorations has been reported to be similarly influenced by the cement manipulation regime (Fleming and Narayan, 2003). Resin-modified glass-ionomer cements are susceptible to water sorption as a result of the amount of hydroxyl-ethyl methacrylate (HEMA) present. In the 1990s, Advance (Caulk/Dentsply Inc., USA) was used to cement all-ceramic restorations but over a time period of less than six months the resin-modified glass-ionomer cement absorbed moisture transported through the dentinal tubules which resulted in the complete fracture of all the all-ceramic restorations. Therefore, acid-base and resin-modified glass-ionomer cements cannot be advocated for luting PLV and DBC restorations. Using the biaxial flexure strength testing methodology, strengthening of PLV and DBC ceramic materials with resin-cement has been shown suggesting resin-based luting materials are the only feasible option.

Investigators have demonstrated the magnitude of resin-strengthening reinforcement achieved for PLV and DBC materials was significantly modified by the selection of resin-based material (Fleming et al., 2006; Addison et al., 2007d, 2008). Resin-based materials have evolved considerably since Bowens' formulation (Bowen, 1963) and in recent years non-methacrylate monomeric resins (Guggenberger and Weinmann, 2000) and filler technologies encompassing nanotechnology (Mitra et al., 2003) have been developed. Manufacturers have increased the filler volume fraction, thereby increasing the mechanical properties (flexural strength and flexural modulus) (Musanje et al., 2001; Masouras et al., 2008) since in terms of mechanical properties the filler is dominant over the resin (Amirouche-Korichi et al., 2009). Today, while the promotion of superior mechanical properties appears to be the goal in RBC development for restorative applications, the improvement of resin-based luting materials appears to be governed by optimisation of the resin viscosity and resin film thickness in efforts to simplify restoration seating and improve marginal adaptation. As a result, the resin-based materials routinely employed for luting feldspathic porcelain and dental glass ceramic PLV and DBC restorations are routinely limited to flowable and luting resin formulations. These formulations have significantly reduced filler loading, lower flexural moduli (Addison et al., 2007d) and increased viscoelasticity when compared with commercial RBC restoratives (Musanje et al., 2001; Masouras et al., 2008). Using the biaxial flexure strength testing methodology, strengthening of PLV and DBC ceramic materials with resin-cement has been shown to be insensitive to defect size (Fleming et al., 2006) but sensitive to macroscopic surface roughness (Addison et al., 2007c) and resin-cement elasticity (Addison et al., 2008). Therefore, the traditionally proposed theories of crack healing (Fabes and Uhlmann, 1990; Marquis et al., 1992) or the crack closure stresses secondary to polymerisation shrinkage (Roach et al., 1988; Nathanson, 1993) have been demonstrated to

incompletely explain the magnitude of observed strengthening (Fleming et al., 2006; Addison et al., 2007d, 2008).

4.3.1 Crack-closure and crack healing theories

The presence of critical surface defects is widely accepted as the predominant determinant of the fracture behaviour of brittle materials under tensile loading. Several different theories have been proposed to explain the observed strength increase following the application of a thin compliant surface coating. The infiltration of individual surface defects by a resin-coating was suggested to result in localised compressive crack closure stresses which resists crack propagation under tensile loading (Roach et al., 1988). However, the origin of the closure stresses have not been qualified although thermal expansion mismatch of the resin and ceramic was proposed (Roach et al., 1998). Feilzer et al. (1990) observed the flexure of a flat glass microscope slide covered with a thin layer of resin following light irradiation. The authors used a simplistic profilometer deflection test to visualise the stress induced deformation of the RBC material and suggested the residual compressive stresses in the resin were imparted globally across the flat glass microscope slide (Feilzer et al., 1990). Nathanson (1993) proposed the resin-strengthening of dental porcelain was induced by the polymerisation shrinkage of resin-cements which stresses the molecules together. An improved version of the simplistic profilometer deflection test used by Feilzer et al. (1990) was employed by Isgro et al. (2011) to demonstrate the deflection of a dental feldspathic porcelain following resin-cementation. The magnitude of the compressive stress induced by the resin-polymerisation shrinkage was manifest as increased deflection of the dental feldspathic porcelain specimen disc.

Fabes and Uhlmann (1990) and Fabes and Berry (1990) applied strengthening mechanism based on crack 'healing' to sol-gel coatings on glass by suggesting the infiltration of surface cracks by a surface coating partially 'healed' the crack, reduced the associated crack length and by inference the crack stress intensity. This theory was first muted in the dental literature by Marquis (1992) who suggested a crack shortening mechanism whereby the resin-coating partially or totally penetrated the dental ceramic defects. In essence the result was proposed as crack healing where resin-penetrance was complete failure was suggested as not resulting from the original defect. Where resin-penetrance is incomplete the resultant embedded flaw possessed a different geometry and by inference, a changed stress intensity was created which was suggestive of the resin-strengthening process.

4.3.2 Resin-ceramic hybrid layer

Fleming et al. (2006) highlighted the capacity of the resin-cements to strengthen both polished and indented surfaces with no statistical differences between strength increases which suggested the resin-strengthening mechanism was not dependent on the nature of the surface flaw population. These findings were not consistent with the crack healing proposal put forward by Marquis (1992) or the crack closure theory based on the polymerisation shrinkage of resin luting materials championed by Nathanson (1993). If crack healing was operative then the apparent resistance to fracture of the resin-coated indented discs would have been markedly increased compared with the resin-coated polished discs examined in the Fleming et al. (2006) study. Similarly, if the crack closure theory where polymerisation shrinkage of resin luting materials can increase the apparent resistance of cemented discs by stressing the molecules together rather than away from each other was correct again the apparent resistance to fracture of the resin-coated indented discs would have been markedly increased compared with the resin-coated polished discs. Therefore resin-strength

enhancement of the resin-layer was independent of individual defect severity and the crack-closure and crack-healing theories were incorrect. One of the problems associated with the study by Fleming et al. (2006) was that the resin-coating thicknesses were not controlled and the polished surface of the dentine porcelain specimens was unrepresentative of the pre-cementation surface encountered in the clinical situation.

Addison et al. (2008) tested the hypotheses that resin-strengthening of a dentine porcelain was dependent on the ceramic surface texture (polished, polished indented and alumina particle air abraded) and resin cement thickness ($60 \pm 20 \mu\text{m}$, $120 \pm 20 \mu\text{m}$, $180 \pm 20 \mu\text{m}$ and $250 \pm 20 \mu\text{m}$). For the first time in the dental literature, regression analysis was employed which allowed an estimation of the scale of the 'actual' strengthening for a theoretical 'zero' thickness of resin cement on each surface texture. Interestingly, resin-strengthening of the polished and polished indented specimens was similar despite the surfaces containing defects of different geometries which confirmed the findings of Fleming et al. (2006). In addition, the previously suggested resin-strengthening mechanisms of crack healing (Marquis, 1992) and the creation of crack closure forces (Nathanson, 1993) were further nullified as both theories specifically implied a sensitivity to individual defect size and geometry. However, Addison et al. (2008) did identify the magnitude of resin-strengthening observed following resin-coating of the clinically relevant alumina abraded surface was increased when compared to the polished and indented specimens confirming that resin-strengthening was dependent on the ceramic surface texture (macroscopic surface roughness). In an earlier study, Addison et al. (2007) examined the impact of RBC materials with markedly different elastic behaviour on the resin-strengthening mechanisms operative in a simulated PLV and DBC restorations. The authors hypothesised that ceramic strength enhancement is dependent upon the elastic modulus of the RBC material chosen. The authors employed the mathematical methods

described by Hsueh et al. (2005) which enabled bi-axial flexure stress calculations at axial positions throughout the ceramic/resin bilayer during failure. Addison et al. (2007) observed the maximum flexural stresses generated in each RBC material coating were considerably below the ultimate flexural strength and elastic limits of each respective RBC coating. Therefore, it was suggested that resin-strengthening was dependent on the behaviour of the portion of the resin-coating which was inter-penetrating the ceramic surface texture. Additionally, a linear relationship was observed between the mean BFS at the ceramic/resin interface and the elastic modulus of the individual resin-based materials which highlighted a dependence of the strengthening processes on the elastic properties of the RBC. These observations were again consistent with the findings of Fleming et al. (2006) where the strengthening of ceramics following resin-coating was independent of defect population.

A further advantage of the study by Addison et al. (2007) was that the sample number for each group was $n=60$ in an effort to enhance the Weibull analysis (McCabe and Carrick, 1986) which had been severely lacking in studies in the dental literature. Interestingly, the Weibull distributions for the feldspathic porcelain discs resin-coated with the luting and hybrid RBCs were distinguishable as two separate distributions related to failure at low and high stress levels and implied that although strengthening had occurred the pattern of failure was consistent with the original defect population. In contrast when the flowable RBC was used as the resin cement, a uniform Weibull distribution at all stress levels was evident suggesting a reduced impact of the original surface defect population. The modification of the Weibull distributions was explained by the increased penetrance of smaller flaws compared with heavily filled resins (Fabes and Uhlmann, 1990). In line with the suggestions of Wang et al. (1995), it was suggested that when resins were restricted to thin layers in cracks then their mechanical response was significantly altered. This was manifest under bi-axial flexure

where the resin within a crack extended perpendicular to the crack face thereby producing a compensating Poisson contraction parallel to the crack surface (Wang et al., 1995). Addison et al. (2007) proposed this contraction was restricted by the resin thickness compared with the stiffer bulk ceramic which also had a lower Poisson's ratio which effectively increased the stiffness of the resin such that the resin in the crack behaved more closely to the bulk ceramic (Wang et al., 1995). The alumina particle abraded surface used in the current study consisted of a multiplicity of defect sizes and resin-interpenetration was considered to introduce what was termed for the first time as the 'resin-ceramic hybrid layer'.

4.3.3 Reinforcement associated with the resin-ceramic hybrid layer

Resin-strength enhancement of dental porcelain was identified as being independent of defect severity (Fleming et al., 2006) but dependent on the elastic modulus of the RBC system (Addison et al., 2007) and the RBC layer thickness (Addison et al., 2008). The current study examined the combined influence of the elastic modulus of the RBC system and the RBC layer thickness on the reinforcement of resin-coated dentine porcelain disc-shaped specimens tested in biaxial flexure (Manuscript 3.1). The choice of RBC materials used was based on their elastic moduli and it is generally accepted, that increasing the filler volume fraction of RBC materials results in an increased modulus (Li et al., 1985; Musanje et al., 2001; Masouras et al., 2008; Amirouche-Korichi et al., 2009; Ilie and Hickel, 2009), assuming the integrity is maintained between the resin matrix and filler particles mediated through the silane coupling agent (Bowen, 1963; Braem et al., 1989). As a result, the RBC materials were selected with markedly different filler contents in an effort to obtain different flexural moduli (Manuscript 3.1). The BFS data reported (Manuscript 3.1) was analysed with regression techniques to determine the patterns of resin-strengthening and identify if the reinforcement was sensitive to RBC flexural modulus and layer thickness. The extrapolation of the linear

regression curves fitted to the mean BFS data for the three different RBC materials estimated the theoretical resin-strengthening in the absence of resin-bulk. The increasing biaxial flexure strengths determined at a theoretical 'zero' resin-coating thickness with increasing resin flexural modulus suggested that the magnitude of resin-strengthening was dependent on the flexural modulus of the RBC while also demonstrating further evidence to indicate the presence of the 'resin-ceramic hybrid layer'. While the pattern of resin-strengthening observed in Manuscript 3.1 was consistent with previously reported data (Addison et al., 2008), the magnitudes of the BFS data were reduced. It should be noted that the bulk ceramic thickness was 1.9 mm in the current study (Manuscript 3.1) compared with 0.9 mm reported by Addison et al. (2008) which accounts for the reported differences in increased strength.

The soda lime glass material was evaluated (Manuscript 3.2) to determine if a controlled surface texture could be introduced which closely matched that observed on PLV and DBC restorative materials. The soda lime glass was chosen to investigate the resin-strengthening mechanism as a considerably more consistent specimen fabrication process could be assured when compared with dental porcelain. The soda lime glass analogue would be required to demonstrate a similar pattern of strength dependence of pre-cementation operative parameters and resin-cementation variables previously characterised with the dentine porcelain. The surface texture of the soda lime glass disc-shaped specimens following a clinically relevant pre-cementation surface conditioning protocol was comparable with the reported surface texture data for dentine porcelain. When linear regression techniques were employed to analyse the BFS data and quantify comparable magnitudes of strengthening at a theoretical 'zero' thickness of resin, the observed pattern of reinforcement was also consistent with the dentine porcelain BFS data. The consistency in the observed data validated the soda lime glass as an analogue material for dentine porcelain to examine the interaction between

surface condition and the RBC materials as determinants in characterising the resin-ceramic hybrid layer.

4.4 RESIN-CEMENTATION OPERATIVE VARIABLES

The seating load applied during resin-cementation has been observed to variously modify the interaction between a ceramic surface and RBC luting materials (Addison et al., 2010). In the clinical setting, the ceramic surface possesses a texture which is established by the pre-cementation operative variables to condition it prior to cementation. The subsequent resin-cementation parameters include seating load and ambient environmental condition which govern the dynamic physical and chemical interaction between the RBC material and the ceramic surface during the cementation process. The previously validated soda lime glass analogue material for dentine porcelain (Manuscript 3.2) was employed to study the influence of seating load and moisture availability during resin-cementation on the strengthening observed (Manuscript 3.3 and 3.4). Control of the loading rate during strength determination allowed for the investigation of stress-rate phenomena on the fracture behaviour of the resin-ceramic hybrid layer.

4.4.1 Resin seating load

The pattern of resin-strengthening was modified depending on the resin-seating load and can be explained by considering the variable interactions of the resin-cement with the ceramic surface on application of the seating load and following its removal. Increased seating load during cementation may be assumed to increase the inter-penetration of the resin with the ceramic surface roughness. However, resistance to compressive deformation of the monomeric resin paste may result following the release of the applied seating loads (5 and 30

N, respectively). The load application process is likely to result in the prepared ceramic surface being placed in tension and the monomeric resin paste under compression. Following the release of the applied load variability may be introduced as the elastic deformation of the resin-cement is reversed and the compression of the resin paste is removed. Such effects may lead to 'spring-back' which is related to the release of energy associated with the initial resistance to deformation on loading. Although these complex effects were not quantified they are expected to result in a modification of the degree of resin-cement penetration. It is suggested that the mechanical response of the increased penetrance of the resin especially when restricted to thin layers in cracks within the 'resin-ceramic hybrid layer' could be significantly altered. Therefore, for specimens resin-luted at a seating load of 5 N, the increased BFS data suggests a more intimate interpenetration of the resin within the cracks in the 'resin-ceramic hybrid layer'.

Earlier interpenetration, results in a restriction of the resin to thin films near the crack tip where Poissons constraint effects are proposed to be operative (Wang et al., 1995). As a result, during flexure, the interpenetrated resin extends perpendicular to the crack face producing a compensating Poisson contraction parallel to the crack surface (Wang et al., 1995) effectively increasing the stiffness of the resin such that the resin within the 'resin-ceramic hybrid layer' behaves more closely to the bulk ceramic (Wang et al., 1995; Addison et al., 2007d). This was observed in the almost linear increase in mean BFS observed for the soda lime specimens which were uncoated or coated at a load of 5N. The decrease in penetration expected from the resin in cracks within the 'resin-ceramic hybrid layer' following the removal of a load of 30 N was proposed as the associated modifying parameter for the resin-strengthening behaviour. Interestingly at the lower loading rates, differences between the BFS data for the different seating loads were not significant and it is suggested

that these observations are supported by the viscoelastic (Musanje and Darvell, 2004) and creep behaviour (Baroudi et al., 2007) of resins at slow testing rates which becomes the dominant or determining phenomenon.

4.4.2 Ambient moisture effects

The oral environment provides a constant source of water which challenges the performance of PLV and DBC restorations in service. Water is known to compromise the strength of silicate materials (Kingery, 1976a) and slow crack growth damage has been demonstrated with dental porcelain (Baker and Preston, 1946). Ritter and Lin (1991) reported that stress corrosion was inhibited by a protective resin-coating of a soda lime glass reducing moisture at the crack tip. External water has been reported to be the primary determinant in the rate-dependent progression of cone cracks on the outer surface of monolithic ceramic restorations with a significant glassy phase (Lee et al., 2002). Epoxy coatings have been demonstrated to protect the surface of soda lime glass from moisture-assisted degradation (Hand et al., 2003) and while the resin-cementation of the intaglio surface is considered initially protective against water ingress and subsequent degradation, Huang et al. (2008) reported that water diffusion through the resin-cementation layer is probable. In addition, Addison et al. (2008) demonstrated the significant degradation of resin-strengthening following the immersion in water of resin-coated dentine porcelain disc-shaped specimens which could be attributed to either the effect of moisture at the ceramic surface or the mechanical response of moisture in the resin itself, namely increased plastic deformation.

Where the determinants on BFS to be examined are associated with surface dominated rate dependent factors, the susceptibility of soda lime glass to slow crack growth in the presence of moisture make it eminently suitable in assessing this primary contribution to rate-

dependent fracture (Lee et al., 2002). It was proposed that moisture availability during resin-cementation may have impacted on the observed pattern of resin-strengthening (Manuscript 3.3). When resin-cementation was performed in ambient air where environmental moisture was not controlled, the rate dependent pattern of reinforcement demonstrated that moisture-assisted corrosion factors were operative (Manuscript 3.3). In contrast, a modified pattern of reinforcement was observed when moisture was removed during the resin-cementation process (Manuscript 3.4).

4.5 DETERMINATION OF BIAXIAL FLEXURE STRESS

In an attempt to provide clinical guidance and maximise the clinical performance of resin-luted PLV or DBC restorations, axisymmetric finite-element analysis of molar crowns (Anusavice and Hojjatie, 1992) and quantitative fractography of failed restorations (Kelly et al., 1989; Kelly, 1999; Kelly et al., 1996; Quinn et al., 2005) identified the failure mode to be tensile in nature and the fracture origin to be controlled by a critical flaw on the inner surface of the crown (Kelly et al., 1989; Anusavice and Hojjatie, 1992; Kelly et al., 1996; Kelly, 1999; Quinn et al., 2005). Therefore the suitability of an in vitro research methodology to investigate the underlying resin-strengthening mechanisms of PLV or DBC restorations depends on the ability to replicate the clinically observed failure mode and the fracture origin (Fleming et al., 2006; Addison et al., 2007; Addison et al., 2008). One test considered was the ‘crunch-the-crown’ testing methodology, however, failure has been shown to be initiated at the loading contact zone on the external surface of the crown (Harvey and Kelly, 1996; Kelly, 1999) and therefore the clinically observed failure mode or fracture origin are not replicated in the testing protocol. In addition, the ‘crunch-the-crown’ test returns only the load at which failure is deemed to have occurred which is a scalar quantity that cannot be compared and

does not inform the investigator about the resultant stress state, within the complex geometry of the specimen, which causes fracture. As a result of these inadequacies the ‘crunch-the-crown’ test has essentially been made redundant from the materials science testing armamentarium (Isgro et al., 2011). Kelly et al. (2012) went further recently when after criticising the appropriateness of ‘crunch-the-crown’ protocols the authors stated they planned to engage editors to take an ‘ethical stand against the publication of studies using “crunch-the-crown” protocols in particular’. Testing in biaxial flexure replicates the clinically observed failure mode and fracture origin of PLV or DBC restorations and the simplified disc-shaped specimen has a surface area to volume ratio approximating such restorations. Using the BFS testing methodology, strengthening of PLV and DBC ceramic materials with resin-cement has been shown to be insensitive to defect size (Fleming et al., 2006) but sensitive to macroscopic surface roughness (Addison et al., 2008) and resin-cement elasticity (Addison et al., 2007d). Therefore, the traditionally proposed theories of crack healing (Marquis, 1992) or the crack closure stresses secondary to polymerisation shrinkage (Nathanson, 1993) have been demonstrated to incompletely explain the observed strengthening (Fleming et al., 2006; Addison et al., 2007d; Addison et al., 2008).

4.5.1 Analytical solution

Investigations involving the ‘crunch-the-crown’ load-to-failure testing protocol are widely reported in the dental literature (Burke and Watts, 1994; Ferro et al., 1994; Pallis et al., 1994; Yoshinari et al., 1994; Evans and O’Brien, 1999; Burke, 1999; Behr et al., 2003; Bindl et al., 2006; Bindl et al., 2007; Aboushelib et al., 2007; Blatz et al., 2008; Borges et al., 2009), however, comparisons between studies have limited scientific appeal (Kelly et al., 2012). The inconsistencies between laboratory mechanical testing protocols used to simulate the in vivo failure mechanism encountered by luted all-ceramic restorations which involves loading the

crown through a ball or bar arrangement at unrealistic clinical loading rates has compromised the usefulness of the data in predicting clinical performance (Ban and Anusavice, 1990). Load-to-failure testing of full-contour all-ceramic restorations has demonstrated that the unrealistic contact loading of complex occlusal surface geometries impact upon the observed load-to-failure data (Lawn, 1993; Kikuchi et al., 1997). As a result, the utilisation of a standardised mechanical testing regimen was considered important for the current study. The experimental approach undertaken involved testing disc-shaped specimens in biaxial flexure as the failure mechanism and fracture origin is consistent with the observed clinical failure mode for PLV and DBC all-ceramic restorations (Kelly, 1999; Fleming et al., 2000).

The analytical approach was employed to determine the maximum tensile stress generated in a disc-shaped specimen under flexure (Manuscripts 3.1 to 3.4) which provided an informed understanding of the failure event by calculating the biaxial flexure stresses with closed-form solutions (Hsueh et al., 2005). Different analytical solutions have been utilised in the dental literature to calculate the maximum tensile stress for monolayered disc-shaped specimens tested in biaxial flexure (Timoshenko and Woinowsky-Krieger, 1959; Shetty et al., 1980; Young, 1989). The maximum tensile stress generated in a homogenous disc-shaped specimen was dependent on the measured load to fracture, Poisson's ratio of the ceramic substrate, testing configuration and specimen dimensions when assessed using the different analytical solutions (Timoshenko and Woinowsky-Krieger, 1959; Shetty et al., 1980; Young, 1989). The flexural modulus of the substrate was considered to be a factor in the monolayer solutions which assumed negligible deflection and was consistent with the observed brittle fracture behaviour of a ceramic. A comparative evaluation of the analytical solutions routinely employed to calculate the Weibull characteristic strength was reported by Addison and Fleming (2008). The Timoshenko and Woinowsky-Krieger (1959) equation resulted in a

6% increase in the characteristic stress when compared with the solutions by Rosenstiel et al. (1993) and Hsueh et al. (2005) which were essentially the monolayer equation detailed by Shetty et al. (1980). The discrepancies in the predicted characteristic stresses between the equations were evident from the variables employed in the analytical solutions. The Timoshenko and Woinowsky-Krieger (1959) equation was an approximation calculated as a function of the load at failure, ceramic layer thickness, radius of the ring support and the Poisson's ratio of the ceramic. However, the radius of the loaded area and the radius of the disc-shaped specimen were not taken into account. The advanced solution championed by Shetty et al. (1980) included a calculation for the radius of the loaded area and the radius of the disc-shaped specimen, in addition to the dependent variables accounted for in the equation by Timoshenko and Woinowsky-Krieger (1959). As a result, the characteristic stress data determined using the advanced Shetty et al. (1980) equation was considered to be more correct. This highlighted the care that should be taken when calculating the biaxial flexure stress generated in homogenous ceramic discs when determining absolute differences in the characteristic stress and BFS data for different ceramic materials.

The analytical solutions which are used to describe the stress distribution in a bilayered disc-shaped specimen under flexure are more complex than the equations employed for the monolayered specimens. The closed-form solution by Rosenstiel et al. (1993) for the maximum tensile stress in the bilayer was dependent on the load at failure, component layer thicknesses, radii of the ring support, loaded area and disc-shaped specimen in addition to the flexural moduli of the layer components. However, the Rosenstiel et al. (1993) solution failed to account for the different Poisson's ratios of the component materials in the bilayer (Rosenstiel et al., 1993). The more comprehensive equations described by Hsueh et al. (2005) can be employed to identify the stress at any axial position in the bilayer by calculating a

modified neutral plane as a function of the same dependent variables in the equation by Rosenstiel et al. (1993), in addition to the Poisson's ratios of the bilayer components. Addison and Fleming (2008) demonstrated that there was good agreement between the characteristic stress data predicted with the bilayer equations described by both Rosenstiel et al. (1993) and Hsueh et al. (2005). However, the application of the monolayer equation by Timoshenko and Woinowsky-Krieger (1959) to the bilayer disc-shaped specimens identified a significant deviation in characteristic stress (12% and 11%, respectively) when compared with the stress data calculated using the bilayer equations of Rosenstiel et al. (1993) and Hsueh et al. (2005). The findings demonstrated that the monolayer equations did not accurately represent the stress distribution in a bilayer and that comparison between studies is problematic if different analytical solutions have been employed, despite adopting consistent testing protocols.

The analytical solution by Hsueh et al. (2005) has been advocated as highly versatile in calculating the maximum tensile stress in the bilayer disc-shaped specimen (Addison et al., 2008). The boundary conditions were utilised to calculate the modified neutral plane required to determine the maximum tensile stress and reflected the test parameters, namely the flexural moduli, the Poisson's ratios and the relative thicknesses of the component parts, namely the ceramic and RBC materials. The assessment of the analytical solution previously established that increasing the RBC flexural modulus (Fleming et al., 2006; Addison et al., 2007d) and resin-layer thickness (Addison et al., 2008) within the range of flexural moduli routinely observed by RBC materials used to lute all-ceramic restorations and for the range of luting thicknesses commonly observed clinically independently reduced the maximum tensile stress in the ceramic. Further interrogation of the analytical solution demonstrated that the impact of the RBC layer thickness was more pronounced when the RBC flexural modulus was

increased (Figure 2, Manuscript 3.1). This pattern was consistent with the observed experimental data for the dentine porcelain (Manuscript 3.1) and soda lime glass (Manuscript 3.2) by the increased slopes of the linear regression curves for the RBC materials with increased flexural modulus. Furthermore, the solution predicted that as the resin-coating thickness approaches a theoretical 'zero' thickness, the stress calculated at the resin–ceramic interface ($z=0$) for a fixed load is identical irrespective of resin-coating flexural modulus, however, the analytical solution could not account for the functional interaction with the resin-hybrid layer observed experimentally.

4.5.2 Finite element analysis

The design of industrial components with complex geometries almost universally involves FEA as a tool to simulate the stress distribution under an applied load to predict the mechanical performance since no analytical solution generally exists. The FEA methodology has been widely used in the dental literature to virtually simulate the stress distribution in all-ceramic crowns under an applied load. Examples include Cuddihy et al. (2013) who simulated the influence of clinically representative root canal access cavity preparations with the FEA method to determine the stress patterns which resulted from the applied masticatory loads. Rekow et al. (2006) used FEA to predict the impact of tooth preparation dimensions and stylised crown geometry on the stress distribution under a clinically relevant loading simulation while Dejak et al. (2012) employed FEA to examine the influence of restoration flexural modulus and Poisson's ratio on the stress field generated in simulated crown models. Furthermore, the merits of implementing the FEA method to verify a novel technique for digitising all-ceramic restorations have been demonstrated by Della Bona et al. (2013). The applications highlighted (Rekow et al., 2006; Dejak et al., 2012; Della Bona et al., 2013; Cuddihy et al., 2013) were virtual case studies which employed the adaptive capabilities of

the parametric simulation to assess the sensitivity of the model to discrete changes in the variables which define the model including material properties (flexural modulus, Poisson's ratio), dimensional and/or geometric parameters. While these investigations are insightful, the usefulness of any FEA simulation is governed by the limitations of the specific features of the simulation model investigated. By implication, any features which have not been specified in the FEA model cannot manifest as determinants of the factors under investigation. Furthermore, the idealisations which are employed in the model may have a detrimental influence on the simulation by the failure to accurately represent the physical behaviour of the model. In the context of the investigations on resin-strengthening, identification of the existence and impact of the resin-ceramic hybrid layer would not have been possible with an FEA model in isolation, where an idealised bond has been assumed between the resin and ceramic layer, in the absence of integrated experimental testing.

The FEA method was implemented differently in the current investigation to take advantage of the adaptive capabilities of the parametric model in determining the stress distribution in individual disc-shaped specimens tested in biaxial flexure. This was possible by integrating the simulation model with the BFS experimental testing methodology. Therefore, the novel approach employed was analogous to the systematic use of the analytical solution for individual disc-shaped specimens tested in biaxial flexure. However, in the absence of an analytical solution, the FEA model provided a surrogate solution. This enabled the determination of the maximum tensile stress in the disc-shaped specimen where the model was adapted to the specific load to failure, geometry and dimensions of each individual specimen tested in biaxial flexure. The FEA verification model of the disc-shaped specimen loaded to failure in biaxial flexure was an essential step in establishing confidence in the analytical solution and the FEA model (Manuscript 3.1). In Manuscript 3.1, it was considered

important to compare the accuracy of the analytical solution utilised to determine the maximum tensile stress generated for a disc-shaped specimen tested in biaxial flexure with a finite element analysis (FEA) method. The experimental ball-on-ring test was simulated with the FEA model to verify the analytical prediction for biaxial flexure stress, thereby validating the experimental result. The two-dimensional axisymmetric finite element model generated for the resin-coated bilayer disc tested in a ball-on-ring test configuration had a specific applied load (200 N) which was consistent with the load used in the analytical solution, to determine the maximum tensile stress in the centre of the ceramic ($r=0$) at the ceramic–resin interface ($z=0$). The boundary displacement constraints reflected the ball-on-ring test configuration. Simulation of the frictionless ring support constraint was attained by fixing the nodal axial (vertical) displacement at the ring support radius while allowing free transverse (horizontal) displacement. The load was applied over a contact area equivalent to that determined by the Hsueh et al. (2005) equation and an idealised intimate bond was assumed (Manuscript 3.1). The biaxial flexure stress determined from the experimental ball-on-ring test configuration was quantified as a measure of the maximum principal stress in the FEA model predicted at the axial centre ($r=0$) of the ceramic at the resin-ceramic interface ($z=0$) (Guazzatto et al., 2004). For the prescribed boundary conditions specified, the predicted maximum tensile stresses calculated using the analytical solution were confirmed to be within 3.3% by the two-dimensional axisymmetric FEA model. The maximum tensile stress determined with the analytical solution (Hsueh et al., 2005) was therefore verified with the FEA model employed in Manuscript 3.1 and was also consistent with the simulation model previously reported and verified by Hsueh et al. (2006). The consistency between the FEA model and the analytical solution demonstrated the accuracy of the FEA model in predicting the maximum tensile stress (Manuscript 3.1).

The clinical relevance of employing flat disc-shaped specimens to investigate the fracture behaviour has been challenged (Qasim et al., 2005; Rekow et al., 2006; Rafferty et al., 2010) and studies involving the ‘crunch-the-crown’ methodology were presumably performed in an attempt to account for geometric factors, but the information provided by the reported failure loads is of limited value. The geometric complexity of full-contour restorations is challenging to examine in a load to failure configuration without closed-form analytical solutions to determine the resultant stress patterns. Furthermore, the investigation of highly complex geometry is not necessary from an analytical perspective to understand the influence of geometric factors on the stress distribution and fracture behaviour. A simplified specimen design which employs non-planar geometry defined by a single radius of curvature (a curved disc-shaped specimen) has been proposed as a suitable model to examine the impact of geometric factors on radial fracture behaviour (Qasim et al., 2006). However, the analytical solutions necessary to describe the stress distribution in the curved disc-shaped specimen are lacking in the scientific literature. Analytical solutions verified with an FEA simulation were proposed by Pecanac et al. (2011) to determine the maximum tensile stress in curved bilayer disc specimens loaded in a ring-on-ring BFS testing configuration. However, the analytical solutions were limited to radii of curvature of 170 mm which were not representative of the curvatures routinely encountered in all-ceramic PLV and DBC restorations and therefore precluded their use in this study.

Therefore, in the absence of an analytical solution for the curved disc-shaped specimen tested in biaxial flexure, the FEA method was implemented as a tool to determine the stress state in the individual nominally identical curved disc-shaped specimens of varying radii of curvature. The influence of the radius of curvature on the stress distribution in the curved soda lime glass disc-shaped specimens was examined using a ball-on-ring testing assembly

(Manuscript 3.5). The previously validated parametric flat two-dimensional axisymmetric FEA model of the ball-on-ring test was adapted and employed as a surrogate analytical solution to determine the maximum tensile stress in the curved disc-shaped specimens. The three-dimensional CAD geometry of the four curved disc-shaped specimens which were specified with radii of curvature (40, 30, 20 and 10 mm) were sectioned axially to create each respective two-dimensional axisymmetric finite element model. The boundary displacement constraints were chosen to reflect the experimental test configuration whereby a frictionless ring support constraint was simulated by fixing the support nodes axially (vertical displacement) at the ring support radius while allowing free radial (horizontal) displacement. The load applied to the outer surface of the soda lime glass disc-shaped specimen was equivalent to that determined by the Hsueh et al. (2005) equation for estimating the load contact area over a central area. The FEA simulation was configured to use a maximum polynomial value of nine and convergence criteria of 3% for a specified measure parameter of maximum principal stress at an axial position ($r=0$) in the soda lime glass disc-shaped specimen on the surface in tension, in addition to the local strain energy. The measured central thickness, radius of curvature and load to failure for each radius of curvature soda lime glass disc-shaped specimen tested was assigned to each FEA model prior to the analysis to determine the maximum principal stress at failure. The parametric model was eminently conducive to shape modification by varying the radius of curvature and layer thickness of the individual disc-shaped specimen tested in biaxial flexure which regenerated seamlessly within the integrated FEA software. The stress distribution was examined for the disc-shaped specimens with different radii of curvature. The experimentally determined failure load conveniently avoided the necessity to incorporate a failure criterion in the linear FEA simulation which was appropriate for the observed brittle failure mode. The accuracy of the FEA simulation was demonstrated by the convergence of the solution to within 3% for a

maximum polynomial order of six in identifying the maximum tensile stress for the curved disc-shaped specimens from the load and boundary conditions employed in the BFS test.

4.5.3 The influence of geometric curvature

It has been suggested that the load to failure testing of flat disc-shaped specimens fails to account for the influence of geometric factors which are likely to influence the fracture behaviour of full-contour restorations with complex geometric form (Rekow et al., 2006). In an attempt to identify the influence of geometric factors associated with full-contour restorations on the failure mode, the ‘crunch-the-crown’ test has been widely employed. The load to failure or fracture resistance reported from the ‘crunch-the-crown’ test has been considerably less useful than the information provided by an understanding of the stress distribution at failure. Additionally, reporting the failure load data in isolation may unintentionally introduce the risk of obscuring the actual factors under scrutiny. There are limited but valuable insights on the influence of geometric factors on the fracture resistance and contact failure of bulk supported simply curved structures which have demonstrated that geometric curvature impacts on the fracture behaviour (Qasim et al., 2005; Rudas et al., 2005; Rekow et al., 2009). However, the influence of curvature on the fracture behaviour of monolayer disc-shaped specimens tested in biaxial flexure in the absence of bulk support has not been examined.

As a result, curved disc-shaped specimens with different radii of curvature were tested in biaxial flexure to determine the impact of radius of curvature on the maximum tensile stress of the curved disc-shaped specimen. The verification FEA model for the flat disc-shaped specimens tested in biaxial flexure which was employed (Manuscript 3.1) was a prerequisite for establishing a reliable model for the investigation of the curved disc-shaped specimens.

From an analytical perspective it was considered more informative to identify the important factors to be examined and design a simplified model which replicated the clinical failure mode so that the influence of geometry on the maximum tensile stress could be assessed. The reduction in the radius of curvature was observed to redistribute the stress in the curved disc-shaped specimens when tested in biaxial flexure with an associated increase in load to failure when compared with the flat disc-shaped specimens of infinite radius of curvature. However, the reduction in the radius of curvature was not observed to significantly impact the maximum tensile stress of the curved disc-shaped specimens at failure. The results demonstrated that while the load to failure was sensitive to radius of curvature, no significant difference in strength was observed. Furthermore, the pronounced sensitivity of the load to failure to the radius of curvature of the simplified curved disc-shaped specimen tested in biaxial flexure highlighted the complications associated with the reported load to failure, or fracture resistance, without identifying the stress distribution in more geometrically complex specimens. Maintaining nominally identical specimens for testing with the 'crunch-the-crown' method is problematic as a consequence of the additional geometric and dimension complexity of full-contour restorations.

The consistent fabrication of nominally identical disc-shaped specimens with a single radius of curvature was monitored by profilometric inspection to ensure the specified dimensional and geometric parameters were controlled. The surface state responsible for the specimen at failure was assumed to be equivalent for all specimen groups given that a common surface treatment was undertaken. However, for specimens with the smallest radius of curvature, significant differences in the microscopic surface topography were observed and were assumed to result from the complexities of abrasive particle interactions with the curved surface. Although this raises a cautionary note about the accuracy of the measured values, it

highlights further the need for modelling more complex geometries where identical and clinically relevant experimental treatments may yield different results. The current study effectively demonstrated the complications associated with interpreting load to failure data and provides support for the relevance of solutions derived from bending theory. In addition, the data supports the synergistic use of bespoke computational analysis with empirical data collection.

5. CONCLUSIONS

The remarkable clinical success of resin-luted PLVs and DBCs has been attributed to a synergism between the tooth preparation and the all-ceramic restoration which is mediated by the resin-based luting material. Laboratory investigations have demonstrated the operative techniques employed during the resin-cementation process and the RBC material itself influence the strength of the all-ceramic material. Furthermore, the testing methodology employed impacts on the determination of the strength of all-ceramic restorative materials. In the current investigation:

- The flexural modulus and thickness of the RBC material used to cement disc-shaped specimens of similar thickness and material to that employed by PLV or DBC restorations had a significant impact on the magnitude of resin-strengthening. The observed variable influence of the resin-coating thickness on the mean biaxial flexure strength was of interest. Therefore, the magnitude of resin-strengthening observed is likely to impact on the clinical performance of PLV and DBC restorations.
- Analysis of the pattern of resin-strengthening demonstrated further evidence to support the existence of the resin-ceramic hybrid layer in mediating the observed resin-strengthening reinforcement.
- The suitability of soda lime glass as an analogue material for porcelain disc-shaped specimens was demonstrated when tested in biaxial flexure. The soda lime glass demonstrated the same patterns of strength dependence influenced by pre-cementation parameters and resin-cementation variables characterised previously with feldspathic

and glass-ceramic materials. Consistent soda lime disc-shaped specimens were prepared rapidly resulting in discs with uniform surface condition, geometry and size.

- The clinical failure of all-ceramic restorations is frequently reported to have occurred while biting something 'soft'. Crosshead test rate and resin-cementation load have been shown to significantly modify resin-strengthening mediated by the resin-ceramic hybrid layer. The rate-dependent pattern of resin-strengthening highlighted that slow crack growth phenomena are evident under the varying occlusal load of the masticatory cycle.
- The resin-cementation seating load was observed to significantly impact on the pattern of resin-strengthening. The decrease in penetration expected from the resin in cracks within the 'resin-ceramic hybrid layer' following the removal of the higher seating load was proposed as the associated modifying parameter for the resin-strengthening behaviour.
- The influence of resin-cementation in the absence of moisture on the resin-reinforcement observed was difficult to interpret owing to the different behaviour of the 'resin-ceramic hybrid layer' with the two loading regimes examined.
- The flat disc-shaped specimen is not representative of the geometric complexity of full contour crowns when investigated with BFS testing. The usefulness of the load to failure testing of specimens with complex geometry is complicated by the lack of analytical solutions to determine the stress distribution at failure. The finite element method was implemented as a surrogate solution to determine the stress state in the

individual nominally identical curved disc-shaped specimens with different radii of curvature. The synergistic use of bespoke finite element analysis with empirical data collection was demonstrated for the analysis of load to failure data in the absence of an analytical solution.

- The biaxial flexure testing of curved soda lime glass discs with different radii of curvature demonstrated the impact of geometric curvature on the load to failure data. Significantly increased loads to failure were observed in BFS testing for curved discs when compared with flatter specimens.. However, the reduction in the radius of curvature was not observed to significantly impact upon the maximum tensile stress calculated at failure.
- The pronounced sensitivity of the load to failure to changes in the curvature of the specimen is definitive evidence of the problems associated with the comparison of the widely reported load to failure data. Even for a simplistic model the sensitivities to shape variation between individual specimens from an ‘identical’ series is high. Comparisons between the load to failure data in isolation are problematic and the study highlighted the potential complications associated with complex specimen geometry including full-contour crown specimens. The ‘crunch-the-crown’ test methodology is widely employed, however, expert opinion has challenged the merits of employing the technique. The findings of the current study demonstrated evidence to highlight the problems associated with the ‘crunch-the-crown’ test which reports only the load at failure.

6. RECOMMENDATIONS FOR FURTHER WORK

The current investigation has demonstrated that the BFS of resin-coated dentine porcelain disc-shaped specimens was sensitive to the flexural modulus and the thickness of the RBC luting material in isolation and in combination (Manuscript 3.1). A soda lime analogue substrate was identified (Manuscript 3.1) which was susceptible to a similar pattern of resin-strengthening when tested in biaxial flexure (Manuscript 3.3) and testing rate effects have been demonstrated (Manuscript 3.4). The magnitude of the load to failure data determined from the biaxial flexure strength testing was sensitive to the curvature of the disc-shaped specimen suggesting the importance of controlling specimen geometry in further resin-strengthening investigations (Manuscript 3.5). The recommendations for further work were inspired by the findings of the current investigation to provide the insight to improve operative techniques and optimise clinical performance.

- Soda lime glass is eminently suitable for testing rate-dependent fracture behaviour and the current investigation has demonstrated a rate-dependency in the BFS testing of the flat soda lime glass disc-shaped specimens (Manuscript 3.2). It has been reported that curvature is a determinant of crack evolution in bulk supported curved glass specimens under an applied load (Rudas et al., 2005). However, the influence of testing rate on the fracture behaviour of curved discs tested in biaxial flexure where geometric curvature is a factor, has not yet been examined. It is postulated that the rate-dependent fracture behaviour will be sensitive to the curvatures which are representative of the geometries encountered in all-ceramic restorations, when examined in biaxial flexure at clinically relevant testing rates. The fracture stability may be evaluated as a function of curvature

by assessing the statistical pattern of failure resulting from the rate-dependent BFS testing.

- Previous investigations have identified the importance of surface texture on the strength of ceramics (Fleming et al., 2006; Addison et al., 2007c). The load to failure has been demonstrated to be sensitive to curvature (Manuscript 3.5). However, the impact of the surface defect distribution on the fracture behaviour of curved discs loaded in biaxial flexure has not been investigated. Furthermore, disc curvature has been reported to significantly influence fracture propagation, but not fracture initiation (Rudas et al., 2005). The curvature influenced the stress distribution in the disc when tested in biaxial flexure. Variation in the homogeneity of surface modifications may influence the nature and distribution of defects in the stress concentrated zone. It is postulated that the influence of surface texture on the fracture behaviour is dependent on the modified distribution of critical stress intensities associated with the different specimen curvatures. An investigation to characterise the impact of surface texture on the fracture behaviour of curved discs tested in biaxial flexure will offer insights which may result in improved pre-cementation operative techniques to enhance clinical performance.
- Resin-reinforcement has been observed for the dentine porcelain (Manuscript 3.1) and the soda lime analogue for dentine porcelain (Manuscript 3.2). Additionally, the impact of radius of curvature on the fracture behaviour of curved disc-shaped specimens was demonstrated (Manuscript 3.5). However, the impact of resin-coating on the fracture behaviour of curved disc-shaped specimens has not yet been investigated. Previous studies on curved specimens has examined contact damage of brittle substrates with compliant bulk support (Qasim et al., 2005; Rudas et al., 2005; Rekow et al., 2009) but

these studies have not determined the impact of resin-cementation on the fracture behaviour of the curved discs tested in biaxial flexure. Future investigations should examine the influence of resin flexural modulus and resin thickness on the fracture behaviour of curved discs to identify if resin-strengthening is evident and provide clinical guidance to optimise the reinforcement achievable.

- The radii of curvature of the disc-shaped specimen was determined to influence the load to failure data but not the maximum tensile stress when tested in biaxial flexure. The rigidity of a body is a function of the flexural modulus and the second moment of area but the influence of a range of clinically relevant layer thickness ratios on the fracture behaviour of bilayer resin-coated curved discs remains to be examined with biaxial flexure testing. An enhanced understanding of the impact of clinically relevant dimensional (thickness) and geometric (curvature) factors on the fracture behaviour of all-ceramic restorations will contribute to improving the operative guidelines for tooth preparation design and restoration configuration.
- The specimens in the current investigation were formed by slump forming to ensure that consistent geometric form and surface texture was achieved. An alternative manufacturing route is the machining of specimens using computer-aided design/computer-aided manufacture (CAD/CAM) technology. Surface defects and subsurface damage can be introduced to the ceramic restoration by the machining process. However, identifying the impact of the machining process on the fracture behaviour of the ceramic has been complicated by the load to failure testing of full-contour restorations (Zhang et al., 1994; Chen et al., 1999; Bindle et al., 2006; Attia et al., 2006; Zahran et al., 2008). The findings of the current investigation (Manuscript 3.5)

demonstrated the problems associated with reporting load to failure data in isolation. In contrast to the previous studies, Addison et al., (2012) investigated the impact of machining induced variability on biaxial flexure strength of flat disc-shaped specimens. Utilising the CAD/CAM machining process would enable the consistent fabrication of nominally identical non-planar specimens with increased geometric complexity. The interaction of multiple geometric features may be evaluated to assess the impact of machining induced variability on the pattern of failure and resin-reinforcement. Analysis of the load to failure data for the curved discs tested in biaxial flexure could be analysed with the integrated FEA and experimental approach employed in Manuscript 3.5 to determine the stress distribution in each specimen. The impact of machining induced variability on the fracture behaviour of the ceramics specimens fabricated with clinically relevant curvatures would be useful in providing guidance for the machining process to optimise the clinical performance of the restorations.

- The influence of sintering on the deformation of ceramic discs has been demonstrated as a result of induced residual thermal stresses. Additionally, the deformation of ceramic disc-shaped specimens has been observed as a result of the RBC polymerisation shrinkage stress induced in the ceramic following resin-cementation. The influence of pre-existing curvature on the deformation which results in the curved discs from the thermally induced residual stresses as a result of sintering has not yet been assessed with BFS testing. Furthermore, the influence of the RBC polymerisation shrinkage stress which is imposed on discs with different radii of curvature following resin-cementation has not yet been examined. The deformation induced in curved discs with different radii of curvature by the RBC polymerisation shrinkage stress may impact on the stress distribution when tested in biaxial flexure. It would be useful to identify if severely curved geometries

demonstrate increased magnitudes of resin-strengthening following the residual stress-induced deformation when compared with the previously reported strength data for flat discs (Isgro et al., 2010; Isgro et al., 2011).

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