EXTRUSION SHEAR BOND STRENGTH OF SELF-ADHESIVE RESIN CEMENT TO GLASS CERAMICS AND ZIRCONIUM OXIDE CERAMICS BEFORE AND AFTER AGING

Thesis

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INTRODUCTION

Dental restorations using all ceramic materials in association with adhesive cements have become the perfect choice in the last decade, primarily because of esthetic properties such as translucency, fluorescence and opalescence that better simulate the appearance of natural dentition. Other desirable characteristics include chemical stability, a coefficient of thermal expansion similar to dentin, biocompatibility and high compressive strength[1,2].

Over the past forty years, the technological evolution of ceramics for dental applications has been remarkable, as new materials and processing techniques are steadily being introduced. The improvement in both strength and toughness has made it possible to expand the range of indications to long-span fixed partial prostheses, implant abutments and implants[3]. Zirconia-based ceramics have a high strength and, therefore, restorations can be cemented with traditional water based cements or bonded with resin cements. If greater retention to tooth structure with minimal marginal leakage is required, bonded cementation with resin cements is recommended. Adhesion to tooth structure and to the ceramic restoration combines good marginal sealing and strengthening of the tooth-restoration complex to minimize marginal leakage and tooth fractures[4].

The cementation process is vital for the clinical success of any resin retained restoration. Being extensively technique sensitive the traditional multi-step resin cements have always been a challenge to manipulate and requires a skillful operator, a cooperative patient and perfect

conditions. The problem with the traditional resin cements is that they require complete isolation all through the procedure, careful time watched etching, thorough etchant washing, priming and uniform bonding.

That was the reason behind the evolvement of a single-step resin cement which is self-etching, self priming and self adhesive to overcome the mishaps that may result from the lengthy meticulous procedures with the conventional multi-step resin cements. This in turn reduces the risk of any faulty procedure which may result in bonding failure. The problem is that long-term evaluations of these materials are not yet available to implement their use instead of the conventional types of resin cements. The few available studies on resin bonding to zirconium-oxide ceramics suggest the use of resin cements that contain special adhesive monomers. Compared with silica-based ceramics, the number of in vitro studies on the resin bond to high-strength ceramics is small. The rapidly increasing popularity of all-ceramic systems requires further research before clinical recommendations can be given.

REVIEW OF LITERATURE

Over the past forty years, the technological evolution of ceramics for dental applications has been remarkable, as new materials and processing techniques are steadily being introduced. The improvement in both strength and toughness has made it possible to expand the range of indications to long-span fixed partial prostheses, implant abutments and implants[3].

Following the introduction of the first feldspathic porcelain by Land,[5] the interest and demand for non-metallic and biocompatible restorative materials increased for clinicians and patients. In 1965 McLean[6] pioneered the concept of adding Al_2O_3 to feldspathic porcelain to improve mechanical and physical properties. The clinical shortcomings of these materials, such as, brittleness, crack propagation, low tensile strength, low wear resistance and marginal inaccuracy, continued to limit their use[7].

1. Metal-Ceramic Restorations:

At the same time the combination of predictable strength and reasonable esthetics has continued to make traditional metal-ceramic restorations popular[8]. These types of restorations have been available since the 1960s and they have shown acceptable results and satisfactory outcome for both the patients and the clinicians. They rely on application and firing of a veneering ceramic onto a metal substructure to produce an esthetically acceptable restoration.

Veneering ceramics for metal-ceramics restorations – commonly named feldspathic porcelains- are usually leucite-based[9]. Feldspathic dental porcelains usually contain between 15 and 25 volume % leucite. This amount is adjusted so that the coefficient of thermal contraction of the porcelain is slightly lower than that of the metal, in order to place the ceramic under slight compression[10]. Numerous defects were reported by Mackert[10] in 1988 for these types of ceramics such as inherent pores, cracks and inclusions as well as the large difference in coefficient of thermal expansion between the leucite crystals and the surrounding glassy matrix, leading to the development of radial tensile stresses and tangential compressive stresses around the crystals upon cooling, Resulting in the formation of micro-cracks. Decoupling of the leucite particles from the matrix has been also reported by Mackert[11] later in 1994, potentially affecting the coefficient of thermal expansion of the ceramic.

Although Seghi et al[12] concluded In 1990 that the mechanical properties of feldspathic porcelains are the lowest of ceramic materials used in dentistry and dominated by the large amount of glassy phase, nevertheless it was estimated in 2005 that more than 50% of all dental restorations fabricated were metal-ceramics[13]. This is due to the fact that metal-ceramic restorations have been used in dentistry for more than four decades, their overall performance can be considered as quite successful. This is mainly due to sustained efforts by manufacturers to improve the quality of the materials offered, particularly in terms of crystal size and optical

properties, such as opalescence. Metal-ceramic technology is challenging, and optimal esthetics can only be achieved by skilled technicians.

2. All Ceramic Restorations:

Driven by a debatable need for metal-free restorations, the evolution of all ceramic systems for dental restorations has been remarkable in the last three decades. Processing techniques novel to dentistry have been developed, such as heat-pressing, slip-casting and Computer Aided Design – Computer Aided Machining (CAD-CAM). Concurrently, all ceramic materials have been developed to match dental requirements, offering increasingly greater performance from a mechanical standpoint. As opposed to metal-ceramics, all-ceramics contain a significantly greater amount of crystalline phase, from about 55 to about 99 volume %. This higher level of crystallinity is responsible for an improvement in mechanical properties through various mechanisms. Unfortunately, higher crystallinity is also associated with higher opacity, which is not always desirable for dental ceramics. However crystallinity is only one of many intrinsic factors contributing to materials performance. Other factors such as crystal size and geometry, modulus of elasticity, phase transformation and thermal expansion mismatch between crystal & glassy phase play a crucial role in determining the final mechanical response of the ceramic.

It should also be kept in mind that when it comes to all-ceramic systems, extrinsic factors such as working conditions play a major role in the long-term performance of the material. The oral environment

assembles a set of challenging working conditions that include humidity, acidic or basic pH, cyclic loading and peak loads that can reach extremely high levels when hard objects are accidentally encountered during mastication. A humid environment is susceptible to lead to stress corrosion and catastrophic failure in ceramic materials including a glassy phase [14]. The same is true for some highly crystalline materials such as 3Y-TZP, which has been shown to undergo micro-structural degradation in a humid environment at relatively low temperatures[15,16].

Table (1): Ceramic materials, systems and manufacturer-recommended clinical indications[17].

Core Material	Composition	Manufacturing Techniques	Clinical Indications
Glass ceramic			
Lithium-disilicate	(SiO_2-Li_2O)	Heat Pressed, Milled	Crowns, anterior FPDP, onlays
Leucite	$(SiO_2-Al_2O_3-K_2O)$	Heat Pressed	Onlays, crowns
Feldspathic	$(SiO_2-Al_2O_3-Na_2O-K_2O)$	Milled	Onlays, crowns, veneers
Alumina			
Aluminum-Oxide	(Al_2O_3)	Slip-cast, milled, densely sintered	Onlays, crowns, FPDP, veneers
Zirconia			
Yttrium tetragonal zirconia polycrystals	$(ZrO_2$ stabilized by $Y_2O_3)$	Milled	Onlays, crowns, FPDP, implant abutments

2.1 Glass Ceramics:

The popularity of heat-pressed ceramics relies on the ability to use the lost wax technique to produce dental ceramic restorations. Dental technicians are usually familiar with this technique, commonly used to cast dental alloys. In addition, the equipment needed to heat-press dental ceramics is relatively inexpensive. The first generation of heat-pressed dental ceramics contains leucite as reinforcing crystalline phase. The second generation is lithium disilicate based.

First generation leucite ceramics contain between 35 to 45 vol % leucite as crystalline phase[18]. Their flexural strength and fracture toughness values are about two times higher than those of feldspathic porcelain[19]. This increase in strength and toughness was explained by dispersion of fine leucite crystals from the heat-pressing process[20]. In addition, as pointed out earlier, tangential compressive stresses develop around the crystals upon cooling, due to the difference in thermal expansion coefficients between leucite crystals and glassy matrix. These stresses can contribute to crack deflection and improved mechanical performance[21].

Second generation heat-pressed ceramics contain about 65 vol% lithium disilicate as the main crystalline phase, with about 1 % porosity. Lithium disilicate has been extensively studied through the past decades since 1975 by Borom et al[22] and several researchers followed[23-27]. All studies seem to agree that the mechanisms leading to the crystallization of lithium disilicate in these systems are somewhat complex. The final microstructure consists of highly interlocked lithium disilicate crystals.

This interlocked microstructure and layered crystals along with the tangential compressive stresses around the crystals contribute to strengthening this type of glass ceramic. Overall, lithium disilicate glass ceramics have performed well as their strength is more than twice that of first generation leucite-reinforced all ceramic system and their good performance led to their expanded use to restorations produced by machining.

2.2 Alumina based Ceramics:

Slip-cast ceramics for dental restorations were introduced in the 1990s. A porous infrastructure is produced by slip-casting, sintered, and later infiltrated with a lanthanum-based glass, producing two interpenetrating continuous networks, one composed of the glassy phase and the other being the crystalline infrastructure. Three crystalline phases are available, namely alumina (Al_2O_3), spinel ($MgAl_2O_4$) and zirconia-alumina (12 Ce-TZP-Al_2O_3).

Alumina based slip cast ceramics contain 68 vol % alumina, 27 vol % glass and 5 vol % porosity[20]. The microstructure consists of blocky alumina grains of various sizes and shapes. Evidence of grain pull-out, bridging and crack deflection was reported with this type of ceramic[20], indicative of efficient crystalline reinforcement, and accounting for high mechanical properties. It has also been suggested that the coefficient of thermal expansion mismatch between the alumina crystals and the infiltration glass could contribute to strengthening due to thermal residual stresses. The presence of large alumina crystals with a high refractive index, and a non-negligible amount of porosity, account for

some degree of opacity in this all-ceramic system which is overcome in the spinel system but on the expense of the mechanical properties[28,29].

Densely sintered alumina-based ceramics produced by dry pressing, followed by sintering have been available since the early 1990s and are still currently used. The technique involves computer-aided production of an enlarged die in order to compensate for sintering shrinkage (12-20%). Dry pressing and sintering of high purity alumina-based core ceramic is then performed at high temperature (1550°C). This leads to a highly crystalline ceramic with a measured flexural strength of 601 ± 73 MPa[30-32]. The high strength core is then veneered with translucent porcelain to achieve adequate esthetics. Clinical results have demonstrated an excellent in vivo performance at 15 years[33].

2.3 Zirconia based Ceramics:

Zirconia is the oxide of zirconium (Zr) metal found in nature combined with silicate oxide with the mineral name Zircon (ZrO_2 SiO_2) or Baddeleyite. Although it has been given the nickname "ceramic steel", the correct terminology is zirconia dioxide. It is technically incorrect to refer to the material as zirconia oxide, but the dental vernacular refers to it as zirconia, just as it uses alumina for the term alumina oxide and magnesia for magnesium oxide. Zirconia as a pure oxide does not occur in nature. The interest in using zirconia as a biomaterial is based on its mechanical strength, as well as its chemical and dimensional stability and elastic modulus similar to stainless steel[34]. Zirconia has a normal density of 6.0 g/cm^2, where the theoretical density (i.e., 100% dense) of zirconium oxide is 6.51 g/cm^2. The closer these two density values are,

the less space between the particles, resulting in greater strength and a smoother surface[35].

Depending on the temperature, zirconia can exist in three forms; cubic phase, tetragonal phase and monoclinic phase. Pure zirconia has a cubic structure at temperatures greater than 2370° C. The cubic phase has a cubic form with square sides and moderate mechanical properties with a density of 6.27 g/cm^2. The tetragonal phase exists at temperatures ranging from 1170° C to 2370° C. The tetragonal structure has a straight prism with rectangular sides and the most satisfactory mechanical properties with a density of 6.1 g/cm^2. The monoclinic phase occurs at temperatures below 1170°C[36] and has a deformed parallelepipedonal (i.e., a prism with six faces) shape, as well as the weakest mechanical properties with a density of 5.6 g/cm^2. In terms of strength, it is essential to limit the amount of the monoclinic phase due to its lower density. To stabilize zirconia at room temperature and control phase transformations, metal oxides, such as yittria (Y_2O_3) or ceria (CeO_2), are added to the crystal structure[37]. The addition of "stabilizing oxides" yields multiphase materials called partially stabilized zirconia[38]. Technically, if yittria is added for stabilization, then it is referred to as Yittria-stabilized Tetragonal Polycrystals (Y-TZP). An ensuing crack generates tensile stresses that induce a change from a tetragonal configuration to a monoclinic configuration and a localized volume increase of 3% to 5%. This volume increase results in a change of tensile stresses to compressive stresses generated around the tip of the crack. The compressive forces counter the external tensile forces and stop the further advancement of the crack [39,40]. This characteristic accounts for

the material's low susceptibility to stress fatigue and high flexural strength of 900 MPa to 1200 MPa[41,42].

Zirconia is provided to dental laboratories in various sized blocks and can be milled into single-unit copings or multiple-unit fixed bridge frameworks. The zirconia blocks are fabricated in two basic ways:

A. *UNIAXIAL DRY PRESSING*

This method involves applying pressure in a uniaxial direction to the ceramic powder confined in a mold[43]. The irregular-shaped ceramic particles are pressed to a high "green" strength from interlocking and plastic deformation of the particles. This method's major disadvantage is that the zirconia block will have varying degrees of density due to the particle/particle and mold wall/particle frictional effect. For example, depending on from which part of the block a three-unit fixed bridge framework is milled, the integrity of the framework could be compromised due to the different zones in the zirconia's density. Usually these uniaxial dry pressed blocks will be square or rectangular in shape.

B. *COLD ISOSTATIC PRESSING*

In this method, the zirconia powders are placed in a deformable mold that is subject to isostatic (i.e., uniform in all directions) external pressure. This process results in a green-stage, chalk-like zirconia block with uniform density. Usually these blocks are cylindrical in shape. Regardless of the fabrication method, at this stage, the zirconia blocks are referred to as "green" blocks. These blocks can be further stabilized and densified (ie, 95% of theoretical density)[39] through sintering in a

special furnace without pressure. At this stage, the blank is referred to as a pre-sintered block. Further compression and heat can be applied to the blank, resulting in improved strength with complete densification. This process is referred to as Hot Isostatic Post-compaction (HIP) and removes any residual porosity[44].

The "green" zirconia blocks are milled at a larger dimension to compensate for 20% to 25% shrinkage during the sintering stage[40]. Having the highest amount of porosity, these blocks are the fastest to mill and produce the least amount of wear on the milling machinery. In contrast, the HIP zirconia blocks (i.e., "white" blocks) are milled at a 1:1 ratio since they are completely densified. Due to their increased hardness[39], these blocks are the slowest to mill and produce the most wear on the milling machinery.

Various examples of green-stage block systems (Cercon, Degudent, Frankfurt, Germany; Lava, 3M ESPE, St. Paul,MN; Zirkon; Zahn® USA, Atlanta, GA; and Hint-Els Zirkon TPZ-G, DigiDent, Girrbach, Pforzheim, Germany) exist. There are also pre-sintered stage block systems (VITA In-Ceram® YZ Cubes, CEREC InLab, Charlotte, NC; KaVo EVEREST ZS-Blanks, KaVo Dental Corp., Lake Zurich, IL; Hint-Els Zirkon TZP-W, DigiDent, Girrbach, Pforzheim, Germany; and DC-Shrink, POPP DCS, LLC, Greendale, WI) and HIP or completely sintered stage block systems (Denzir® Premium HIP Zirconia, etkon USA, Arlington, TX; Hint-Els Zirkon TZP-HIP, DigiDent, Girrbach, Pforzheim, Germany; ZirKon™ Pro50™, Cynovad, Saint-Laurent (Quebec), Canada; and KaVo EVEREST ZH-Blanks, KaVo Dental Corp, Lake Zurich, IL).

There are three basic types of zirconia single-coping or fixed partial denture framework fabrication methods available today:

I. MANUALLY CONTROLLED SYSTEM OR MANUAL-AIDED DESIGN/MANUAL-AIDED MANUFACTURING (MAD/MAM) METHOD (also referred to as copy milling)

This method is based on the pantographic principle that was employed hundreds of years ago to copy or enlarge paintings, then later for engraving. The same principle is utilized at the hardware store when making duplicate keys. Using exact mechanical-tactile model surveying and analogous milling[45], it is considered to be highly precise in transfer accuracy. First, a coping or framework is manually fabricated in wax or composite, and then the pattern is placed into the pantographic machine. The copying arm of the machine traces the wax pattern while the cutting arm, which has a carbide cutter, mills a selected "green" or pre-sintered zirconia block. The final shape is 20% to 25% larger in order to account for shrinkage during the sintering step. The zirconia block has a density barcode label, so the copy mill machine can be adjusted properly to allow for shrinkage during the sintering phase.

Besides the lower cost factor for these types of milling machines (Zirkon and Ceramill, AmannGirrback Gmbh, Koblach, Austria; TiZan™ Mill, Schutz Dental Group, Shelton, CT), this method of milling allows the dental technician to correct any discrepancies found in the tooth preparation by compensating during the waxing of the pattern.

II. MANUFACTURER-SPECIFIC CLOSED SYSTEM

The second method of fabrication used by dental laboratories is scanning the tooth-prepared models, designing the single coping or