COMPARATIVE EVALUATION OF SHEAR BOND STRENGTH OF VENEERING PORCELAIN TO BASE METAL ALLOY AND ZIRCONIA SUBSTRUCTURES BEFORE AND AFTER AGING – AN IN VITRO STUDY

Dissertation Submitted to THE TAMILNADU DR. M.G.R. MEDICAL UNIVERSITY

In partial fulfillment for the Degree of

MASTER OF DENTAL SURGERY



BRANCH I PROSTHODONTICS AND CROWN & BRIDGE

APRIL 2011

CERTIFICATE

This is to certify that the dissertation titled "COMPARATIVE EVALUATION OF SHEAR BOND STRENGTH OF VENEERING PORCELAIN TO BASE METAL ALLOY AND ZIRCONIA SUBSTRUCTURES BEFORE AND AFTER AGING - AN IN VITRO STUDY" is a bonafide record work done by Dr. LAJU. S under our guidance and to our satisfaction during her post graduate study period between 2008 - 2011.

This Dissertation is submitted to THE TAMILNADU DR. M.G.R. MEDICAL UNIVERSITY, in partial fulfillment for the Degree of MASTER OF DENTAL SURGERY - PROSTHODONTICS AND CROWN & BRIDGE, BRANCH I. It has not been submitted (partial or full) for the award of any other degree or diploma.

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Introduction

Review of Literature

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INTRODUCTION

The porcelain-fused - to - metal systems have been extensively used in fixed partial dentures and still represents the gold standard. The advantage of the porcelain-fused-to-metal systems are to combine the fracture resistance of the metal substructure with the esthetic property of the porcelain⁷. However, metal ceramic restorations show the problem of metal discoloration at the margins, allergic reactions and sensitivity to various metals³. Hence, the increasing demand for the esthetic restoration as well as the questionable biocompatibility of some dental metal alloys has accelerated the development and improvement of metal free restorations^{1,7,55}.

The success of all-ceramic crowns and patient demand for metal-free, tooth-colored restorations has led to the development and introduction of restorative systems for all-ceramic fixed partial dentures. This restorative option demonstrated a high rate of failure at the connector sites. Since then, developments in dental ceramics have led to the introduction of new high-strength ceramic core materials for all-ceramic fixed partial dentures⁶.

The most recent core materials for all-ceramic fixed partial dentures are the yttrium tetragonal zirconia polycrystals (Y-TZP)-based materials. This material is currently being evaluated as an alternative core material for complete coverage restoration such as all-ceramic crowns and all-ceramic fixed partial dentures⁶.

In the early 1990s Yttrium Oxide Partially Stabilized Tetragonal Zirconia Polycrystal (Y-TZP) was introduced to the dentistry as a core material for all ceramic restoration and has been applied to clinical use through the CAD / CAM technique⁷. Due to the transformation toughening mechanism , Yttrium Oxide Partially Stabilized Tetragonal Zirconia Polycrystal has been shown to have superior mechanical properties compared to other all ceramic systems^{6,9,14,30}.

Zirconia is the only ceramic material which meets the flexural strength requirements for FPDs of 4 or more units as recommended by the International Organization for Standardisation (ISO, 1999)^{4,5,20,30}. Yet, while strong, due to limited translucency, zirconia has been veneered with esthetic porcelain to give clinical acceptance. Clinical studies of veneered zirconia restorations indicate that while the zirconia cores are very fracture resistant, however fracture / chipping of the porcelain veneer during mastication is a frequent problem^{3,35,37}.

The adhesion mechanism between metal and porcelain is believed to be due to combination of micromechanical bond, compatible coefficient of thermal expansion match, van der Waals force and mainly the suitable oxidation of metal and interdiffusion of ions between the metal and porcelain^{3,11,34,38,51,62}. According to the investigations on the wettability of the zirconia core with the veneering ceramic, micromechanical interactions were merely regarded. Many variables may affect the zirconia core – veneer bond

strength; such as surface finish of the core , which can affect the mechanical retention; residual stress generated by mismatch in coefficient of thermal expansion ; development of flaws and structure defects at core - veneer interface; and wetting properties and volumetric shrinkage of the veneer 3,4,7 .

The cause of fracture of veneering ceramic on zirconia all-ceramic cores was reported to be multifactorial in clinical application. Restoration geometry such as lack of proper veneering ceramic support, inadequate framework design and thickness of the ceramic layers seem to play a decisive role. Moreover direction, magnitude and frequency of the applied load as well as size and location of occlusal contact areas can contribute to failures of the veneering ceramic⁵⁰.

The success of the metal ceramic or ceramic veneered to zirconia core restorations depends primarily on strong bond between the veneering ceramic and the substructure. In the literature sufficient information are available regarding the bonding mechanism and the bond strength values of porcelain veneered to metal core where as there are less information available regarding the same with the porcelain veneered to zirconia core. There are several tests capable of evaluating the veneering ceramic – core bond strength such as flexural mode, twist, shear, tension or the combination of flexural and twist. Many authors in the literature suggested the use of shear bond strength test as one of the most reliable methods to evaluate the bond strength because it concentrates the applied tension on the interface between two materials.⁵⁴

The loss of strength for ceramic materials after aging in water is well documented.^{16,42} The bond strength of a ceramic coating to a metal substructure also decreases when the materials are exposed to water and / or physiologic media.^{16,42}The oral environment would appear to have all the factors necessary for the process of strength degradation of ceramics in aqueous environment.

In view of the above considerations, the aim of this study was to evaluate and compare the shear bond strength of veneering porcelain to base metal alloy and zirconia substructures before and after aging . Scanning electron microscopy (SEM) was used to determine the failure pattern of samples. Energy dispersive X-ray microanalysis (EDX) was used to evaluate the interface chemistry of the samples.

The objectives of the present study included the following:

- 1. To evaluate the shear bond strength of veneering porcelain to base metal alloy substructure before aging. (Group I)
- 2. To evaluate the shear bond strength of veneering porcelain to base metal alloy substructure after aging. (Group II)
- 3. To evaluate the shear bond strength of veneering porcelain to zirconia substructure before aging. (Group III)
- 4. To evaluate the shear bond strength of veneering porcelain to zirconia substructure after aging. (Group IV)

- 5. To compare the shear bond strength of veneering porcelain to base metal alloy substructure before and after aging. (Group I & Group II)
- To compare the shear bond strength of veneering porcelain to zirconia substructure before and after aging. (Group III & Group IV)
- To compare the shear bond strength of veneering porcelain to base metal alloy substructure and zirconia substructure before aging. (Group I & Group III)
- 8. To compare the shear bond strength of veneering porcelain to base metal alloy substructure and zirconia substructure after aging.
 (Group II & Group IV)
- 9. To compare the shear bond strength values obtained from the four groups (Group I, II, III &IV).
- To evaluate qualitatively the mode of failure of the samples by Scanning Electron Microscopy (SEM analysis) and Energy Dispersive X-ray microanalysis (EDX analysis)

REVIEW OF LITERATURE

George Straussberg et al (1966)²² proposed a design for porcelain fused - to - Gold restoration. He stated that the shape of the supporting gold must be such that the covering porcelain is not compelled to produce a sharp angle. Metal structures should be designed such a way to provide uniform thickness for the porcelain. The minimum thickness of gold should be 0.5mm. According to the author, the gold framework upon which the porcelain will be fused must be sufficiently rigid in all its parts to support the porcelain without excessive flexure. The framework must provide sufficient space for adequate thickness on the layers of opaque and translucent porcelain. The units of the fixed partial Denture must be securely and rigidly connected by sound, welldesigned solder joints.

Walter S. Warpeha et al (1976)⁵⁸ investigated the design and technique variables affecting fracture resistance of metal-ceramic restorations. Forty-four solid metal-ceramic crowns were fabricated and subjected to compressive load testing using universal testing machine. Study concluded that the design of the underlying metal structure had a significant relation to the ultimate fracture strength. A design with a definite acuteness of the underlying metal structure failed at significantly lower ultimate fracture strengths.

A metal conditioning agent did not decrease fracture resistance if applied properly. Fracture Strength was severely decreased when improper

thickness of the coating agent was used and porcelain was used to an unoxidized metal surface. Bond strength although contributing factor, may not be as important as metal design and proper manipulation of materials during fabrication of the restoration.

Thomas A. Wight et al (1977) ⁵⁶ determined four variables affecting the bond strength of porcelain to non-precious alloy. The variable were directional variations of metal preparation using the Paasche Air Eraser with aluminium oxide fast-cut abrasive, atmosphere variations in the furnace from low to high temperature limits of the degassing cycle, time variations at normal atmosphere of 1850°F and firing of the opaque layer of porcelain at different temperatures. Ticon alloy samples were fabricated and veneered with corresponding porcelain and for shear bond strength using Instron Universal testing machine.

It was determined that firing the opaque layer at 1840°F at a rate of 75°F per minute more than doubled the mean bond strength of all samples. The time at the upper limit of the degassing cycle also had a significant effect on the bond. As the time increased, the bond strength decreased. Complete bonds between porcelain and non-precious metals were demonstrated that the opaque firing and degassing be done in accordance with the foregoing findings.

P.H.DeHoff et al (1983)¹⁵ analyzed the stresses which develop during air cooling and shock testing of a simulated porcelain-metal crown. Strain

gauges were used to experimentally determine porcelain surface stresses during shock testing. The finite element method was used to calculate the stress patterns throughout the simulated crown. Based on the systems and methods employed in this study, transient stresses developed during normal air cooling were not high enough to cause porcelain crazing. High tensile stresses which developed in the thermal shock test during the cooling cycle at the porcelain surface were primarily caused by thermal gradients in the body porcelain.

J. L. Drummond et al (**1984**)¹⁶ determined the bonding strength of porcelain to a gold metal substrate and to a non-precious metal substrate after they had been aged in double–distilled water at 37°C for 4 and 12 months. A 7.4% and 18.1% decrease in the bond strength after 4 months and 12 months respectively, was observed for the porcelain gold system and a decrease of 21.2% and 21.4% after 4 and 12 months, respectively was observed for the non-precious-porcelain system.

R. Morena et al $(1986)^{42}$ investigated the fatigue of dental ceramics in a simulated Oral Environment. The dynamic fatigue method was used to obtain subcritical crack growth parameters for the three Dental Ceramics – a feldspathic porcelain, an aluminous porcelain and a fine grain-polycrystalline core material. The constant stressing rate experiments were carried out at 37°C for all three ceramics in distilled water and for the feldspathic porcelain, in artificial saliva as well. Feldspathic porcelain showed that fatigue failure within five years is a good possibility at stress levels which can be anticipated to occur in the oral environment. Little likelihood of failure was perceived for the fine-grain ceramic. The aluminous porcelain was intermediate between these two materials with respect to failure probability.

Warrren C. Wagner et al $(1993)^{60}$ investigated the effect of interfacial variables on metal porcelain bonding. Palladium alloy and corresponding feldspathic porcelain were used for this study. Variables tested were 1.Precoating the metal by sputtering various oxides before porcelaining, 2. Preoxidation of the metal base before porcelaining. 3. Porcelaining under reducing atmosphere roughened surfaces resulted in the highest bond strengths. A direct co-relation between roughness and bone strength was formed with greatest roughness leading to higher bond strength. Changing porcelain firing atmosphere strongly affected bond strength. Firing in a reducing atmosphere dramatically using reduced bond strength as compared to specimens fixed in a normal firing atmosphere. All sputtered oxide precoatings improved bond strengths over the control. Increasing the thickness of the Al₂O₃ precoatings improved the strength even further.

Terry R. Walton et al (2002)⁵⁵ reported the outcome of 515 metalceramic FPDs involving 1,209 abutments and 885 pontics placed by one operator in a specialist prosthodontic practice between January 1984 and December 1997 study revealed that cantilever and FPDs, non-vital abutments and anterior abutments had significantly greater failure rate. Tooth supported FPDs have an expected survival rate of 85% at 15 years when the described clinical and laboratory protocol was applied.

Ardlin Bl (2002)⁴ determined the chemical solubility and effect of aging in 4% acetic acid at 80°C for 168 hrs on flexural strength, surface and crystalline structures. The chemical solubility in 4% acetic acid was recorded by weight loss, and SEM was used to evaluate the surfaces of Y-TZP and dental feldspathic porcelain samples immersed in SnF. Study found that Y-TZP had high strength that were not affected by aging. However the crystal and surface structure were affected. Transformation from tetragonal to monoclinic structures occurred and small elevations on the ceramic surfaces were observed after aging.

Giuseppe Isgro et al (2003)²⁴ determined the effect of different surface treatments on the strength of a heat pressed ceramic core material and veneering porcelain as well as the influence of veneering porcelain on the strength of a 2 layer ceramic structure The study concluded that the heat pressed ceramic core were stronger than the veneering porcelain for the airborne-particle abrasion, as fired, and ground surface treatments. For overglazed treatment, there was not a significant difference between the core and the veneer material. The ground 1-layer core was significantly stronger than the 2-layer with core tested in tension. There was no significant difference between 1-layer and 2-layer veneer overglazed disc when tested with veneer in tension.

J.B. Quinn et al (2003)⁵² had done a research to measure the fracture toughness for several groups of dental ceramics and determined how this property is affected by chemistry and microstructure. The first group consisting of micaceous glass ceramics and second group consisted of feldspathic porcelain, varied significantly in microstructure and in chemistry. Upper toughness limits for the micaceous glass ceramics and feldspathic porcelain were significantly raised compared to the base glasses. The highest toughnesses were associated with high percent crystallinity, large grains and high aspect ratios. Very large increase in fracture toughness were unlikely to be attained by change in microstructure alone. A functional relationship determined for micaceous glass-ceramics enabled quantitative predictions of fracture toughness based on the microstructure.

Massimiliano Guazzato et al (2004)⁴⁰ compared biaxial strength, reliability and the mode of fracture of bilayered disks made of two core materials (In-ceram Alumina and In-ceram Zirconia), both veneered with conventional feldspathic porcelain (Vita alpha). Finite element analysis was used to estimate the maximum tensile stress at fracture and showed all specimens with the core material on the bottom surface were statistically significantly stronger and more reliable than those with the porcelain on the bottom surface. Among them, In-ceram Zirconia was stronger than In-ceram Alumina. Massimiliano Guazzato et al (2004)⁴¹ investigated strength, fracture toughness and microstructure of Nine all-ceramic material such as DC Zirkon, an experimental Yttria partially stabilized Zirconia, In-ceram Zirconia slip and In-ceram Zirconia dry pressed were compared. Study revealed that the Zirconia based dental ceramics are stronger and tougher material than the conventional glass ceramic.

Narong Potiket et al (2004)⁴⁷ evaluated and compared fracture resistance of crowns made of 3 different types of all ceramic crown systems – 0.4mm and 0.6mm aluminium oxide coping crowns and zirconia ceramic coping crowns and metal ceramic crowns. Within the limitations of the study design there was no significant difference in fracture strength of teeth prepared for all-ceramic crowns with 0.4mm and 0.6mm aluminium oxide copings or 0.6mm zirconia ceramic copings and teeth prepared for metal-ceramic crowns. Fracture after loading occurred through the teeth, not through the restorations.

Hana M. Al-Dohan et al $(2004)^{26}$ in this study determined the shear strength of core-veneer interface in bi-layered ceramics. Within the limitation of this study that Eris veneering porcelain applied to IPS – Empress II core showed the highest shear strength values and were not significantly different from the metal ceramic control surface. The bond strength of veneering porcelain to zirconia core was not significantly different from IPS Empress II/Eris or the metal ceramic control. All ceram applied to the procera alumina core showed a significantly weaker bond compared to the other systems. Remnants of the opaque on the core were observed microscopically after failure surface analysis of failure modes demonstrated that the bond between the core and the veneer was cohesive in the veneer and adhesive at the interface for most systems tested.

Ariel J. Raigrodski (2004)⁶ stated that new high strength core/framework materials have been developed for all ceramic FPDs. However, most of these systems are limited with respect to replacement of anterior and premolar teeth, require large connector dimensions and may require the use of more technique sensitive chemical procedures such as adhesive cementation. The most contemporary systems use Y-TZP as the core material and may be an alternative treatment modality for replacing a missing tooth both in the anterior and posterior segments. In addition such systems prove to be simple to handle and less technique sensitive from a clinical stand point, while providing patients with esthetics and functional restorations. Cercon and Lava systems use partially sintered zirconia block. Whereas DC Zirconia uses fully sintered zirconia block.

Heinz Luthy et al (2005)²⁸ in this study, load bearing capacity of four unit-posterior frameworks made of glass ceramic with lithium disilicate crystals, of zirconia-reinforced glass infiltrated alumina and of zirconia stabilized with 3 mol% Yttria were compared. Yttria stabilized zirconia showed the best mechanical properties as demonstrated by the high values of average load bearing capacity, reliability and characteristic load bearing capacity with respect to the other ceramics studied. However, for four-unit posterior Yttria stabilized zirconia framework, the connector size 7.5mm is insufficient to withstand occlusal forces reported in the literature.

Daniel M. Schweitzer et al $(2005)^{12}$ conducted a study to compare the bond strength of a pressed ceramic fused to metal versus feldspathic porcelain fused to metal. Under the conditions of this study the debonding / creak initiation strength of a low fusing pressable received – based glass ceramic fused to metal was equivalent to that of a feldspathic porcelain fused to metal.

Janet B. Quinn et al (2005)³¹ analyzed the clinical failure of three ceramic whole–crown restoration ceramic material systems used included appropriate veneers applied to core materials of cold isostatically pressed alumina, injection – magnesia spinal, and hot pressed lithium disilicate. The surface topography was examined for classical fractographic features, utilizing both optical and scanning electron microscopes independently as well as group examination by four fractographers. In all three cases, fracture originated at or within the core material, where the core was thinnest and stresses were present.

Giuseppe Isgro et al (2005)²³ conducted study to evaluate the thermal compatibility between a ceramic core and veneering porcelain by measuring the bending of layered and to compare the result with the thermal mismatch value. The defection of the layered disc was strongly influenced by the degree

of thermal mismatch during fabrication procedures. The greatest deflection occurred during the 2^{nd} dentin firing and with the largest mismatch.

Renata Marques de Melo et al $(2005)^{53}$ compared the shear bond strength between a porcelain system and four alternative alloys. Alloys used were two Ni-Cr alloys – 4 ALL and Wiron 99, and two Co-Cr alloys – IPS d. SIGN20 and Argeloy NP IPS d.sign porcelain system was used for this study. Specimens were subjected to a shear load on a universal testing machine using a 0.5mm/m cross head speed.

Study concluded that shear bond strength evaluation of the interface formed by base metal alloys (Co-Cr, Ni-Cr) with a dental porcelain product revealed no statistically significant differences in bond strength for the 4 alloys and single ceramic tested.

Ariel J. Raigrodski et al (2006)⁵ assessed the efficacy of zirconiabased posterior 3 unit FPDs and came to the conclusion that zirconia-oxidebased posterior 3 unit FPDs demonstrated good performance in terms of clinical fracture resistance, marginal integrity, marginal discoloration, and secondary decay, after short term (18 to 36 months) service minor chipping of veneering porcelain, which did not require the replacement of the restoration, was detected primarily at the second molar retainers. Neither delamination of the veneering porcelain nor fracture of the framework was detected at the connector or the retainers. **Irena Sailer et al** (2007)²⁹ in this prospective clinical cohort study reported the success rate of 3 to 5 unit zirconia frameworks for posterior fixed partial denture after 5 years of clinical observation. The success rate of the zirconia framework was 97.8% however the survival rate was 73.9% due to other complications. Secondary caries was found in 21.7% of the FPDs, and chipping of the veneering ceramic in 15.2%. There were no significant differences between the periodontal parameters of the test and control teeth.

Paolo Francesco Manicone et al (2007)⁴⁹ overviewed the basic properties and clinical applications of zirconia ceramics. Zirconia is a ceramic material with adequate mechanical properties for manufacturing of medical devices. Zirconia cores for fixed partial dentures on anterior and posterior teeth and on implants are now available. Zirconia opacity is very useful in adverse clinical situations, for example, for masking of dischromic abutment teeth. Radiopacity can aid evaluation during radiographic controls. Zirconia frameworks are realized by using computer aided design and manufacturing (CAD/CAM) technology. Cementation of Zr-ceramic restorations can be performed with adhesive luting. Mechanical properties of Zirconia oxide FPDs have proved superior to those of other metal – free restorations. Zirconia implant abutments can also be used to improve the esthetic outcome of implant – supported rehabilitations. Orthopaedic research led to this material being proposed for the manufacture of hip head prosthesis. **Petra C. Guess et al** (2008)⁵⁰ had done a study to evaluate the shear bond strength between different zirconia cores and veneering ceramics and their susceptibility to thermocycling. Three zirconia core ceramics cercon base, Vita In – Ceram YZ cubes, De- zirkon and their manufacturer recommended veneering ceramics – cercon cerams, vita VM9, IPS – e. max ceram were used for this study. A metal ceramic system (Degudent U94, vita VM13) was used as a control group for the three all ceramic test groups half of each group was thermo cycled and tested in universal testing machine for shear bond strength. The results indicated that the SBS between zirconia core and veneering ceramics was not affected by thermocycling. None of the zirconia core and veneering ceramics could attain the high bond strength values of the metal ceramic combination. The all – ceramic groups showed combined failure modes as cohesive in the veneering ceramic and adhesive at the inter face, where as the metal ceramic showed predominately cohesive fractures.

Moustafa N. Aboshelib et al (2008)⁴⁵ evaluated the effect of combining both press- on and layering veneering ceramics in one restoration on bond strength with zirconia frame works. The double veneer technique combines the high bond strength and superior interfere quality achieved using press –on ceramics with the superior esthetics and individual characterization obtained using layering ceramics. The technique promises superior function and performance of the double veneered restoration.
In this study, microtensile bond strength of zirconia veneer was measured in a universal testing machine. Zirconia bars were veneered with 3 mm thick press-on veneer ceramic or veneered with 1 mm thick press-on veneer and an additional 2 mm thick of layering veneer ceramic. Test revealed that microtensile bond strength of zirconia and press-on ceramic was not affected by the addition of a second layer of either veneer ceramic.

Moustafa N. Aboushelib et al (2008)⁴⁴ studied the bond strength between different veneer ceramics and zirconia framework. This bond was proven to be sensitive to the surface finish of the frame work material and to the type of the veneer ceramic and its method of application. The type of zirconia frame work had a significant effect on the core–veneer bond strength which was material decadent. The bond strength to colored zirconia was significantly weaker compared to white zirconia framework. Different surface treatments had different effect on the core-veneer bond. Strength according to the zirconia material used. Although no marked chemical differences between the examined zirconia material could be found, there were structural differences, especially between white and colored zirconia and for different zirconia frame works of different manufacturers, which significantly affected core-veneer bond strength values.

Marit Oilo et al (2008)³⁹ experimented whether the firing procedures affect the mechanical properties of a zirconia ceramic. Industrially sintered yttria- stabilized zirconia were divided into three groups. One set of specimen

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remained untreated. Another set of specimens were heat treated once to simulate 1st step of veneering process and third set of specimens were heat treated five times to mimic the full veneering process. Flexural strength, microhardness, dimension, and surface roughness were measured. The untreated specimens showed a statistically significant higher flexural strength and microhardness than both of the test groups. No significant differences were found for fracture patterns, dimensions or surface roughnesss.

J.W. Kim et al (2008)³⁷ in the study hypothesized that veneer chipping/ delamination is a result of the propagation of near-contact-induced partial cone cracks on the occlusal surface under mastication. To test this hypothesis, flat porcelain-veneered zirconia plates cemented on to dental composites and clinically loaded them at an inclination angle as a simplified model of zirconia-based restorations under occlusion. Post mortem damage evaluation of porcelain/zirconia/composite trilayers by a sectioning technique revealed that deep-penetrating occlusal surface partial cone fracture in the predominant fracture mode of porcelain veneers.

Jens Fisher et al (2008)³³ assessed the effect of different surface treatments on the bond strength of veneering ceramics to zirconia. In a shear test, the influences of polishing, sand blasting, and silica-coating of the zirconia surface on bonding were assessed with five different veneering ceramics, failure in every case occurred in the veneering ceramic adjacent to the interface with a thin layer of ceramic remaining on the zirconia surface,

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indicating the bond strength was higher than the cohesive strength of the veneering ceramic. Findings of this study revealed that bonding between veneering ceramics and zirconia might be based on chemical bonds. On this note, sandblasting was not a necessary surface pretreatment to enhance bond strength and that regeneration firing was not recommended.

Isabelle Denry et al (2008)³⁰ reviewed the specific types of zirconia available in dentistry, together with their properties. The two main processing technique, soft and hard machining, were assessed in the light of their possible clinical implications and consequences on the long-term performance of zirconia.

J. Robert Kelly et al (2008)⁵³ reviewed the concepts and background from the ceramics engineering literature regarding metastable Zirconia ceramics to establish a context for understanding current and emerging Zirconia-based dental ceramics.

Burak Taskonak et al (2008)⁸ suggested that testing environment has an effect on flexural strength and critical flaw sizes of the dental ceramic.Critical flaw sizes of the core and veneer specimens will be controlled by the presence of the water and changing stressing rate in the testing environment. Flexural strength of the ceramic bars will decrease with slower stressing rate in a water testing environment however, their fracture toughness will remain the same. Hang Wang et al (2008)²⁷ investigated the effect of different surface treatment methods and in particular the effect of the CAD/CAM milling procedures on the flexural string 15 of zirconia frame work. In this study the author stated that the surface damage produced by the CAD/CAM milling procedures significantly reduced the strength of zirconia which could be further weakened by different surface treatment methods resulting much lower than the ideal strength of the material.

Hamid M. Ashkanani et al $(2008)^{25}$ conducted a study to evaluate the flexural and shear strength of ZrO_2 and a high noble alloy with corresponding porcelains the result showed that there were no significant difference among the groups in flexure, except between thermal cycled metal ceramic and ZrO_2 groups. There was a significant difference between the metal ceramic and ZrO_2 groups in shear. Thermal cycling did not have a clear effect among different groups in both tests.

J. Fischer et al (2008)²⁰ compared the flexural strength of veneering ceramic for zirconia. 10 different veneering ceramics for zirconia and three different veneering ceramics for the metal ceramic technique were tested. Three– point flexural strength, biaxial flexural strength and four point flexural strength of these groups were measured.

Study concluded that strength values of veneering ceramics for zirconia are similar to those of veneering ceramics for the metal ceramic technique. Four-point flexural strength values of all material tested were

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significantly lows than those obtained with the three-point flexure test the biaxial flexural strength in general ranged between the four-point flexural strength and the three point flexural strength.

M. Erhan Comlekoglu (2008)¹⁹ evaluated the bond strength of four different margin ceramics based on fluroapatite and feldspath to a zirconia ceramic. Zirconia (zirconzahn) were fabricated according to the manufacturers' instructions (4mm diameter, 2mm thickness) and ultrasonically cleaned. Four different margin ceramic (thickness 5 mm) (Cerabein Zr, Ceramco PFZ, e.max and Triceram) were condensed and fired on to the zirconia core. After ultrasonic cleaning, the specimens were embedded in PMMA. The specimens were stored in distilled water at 37°C for 1 week and shear bond strength test were performed in a universal testing machine (cross head speed 0.5mm/min) the result showed that the shear bond strength values of ceramic margin. Ceramic to zirconia was significantly lower than those of cerabein, e.max and Triceram margin ceramic system.

Eleftheria Tsalouchou et al (2008)¹⁸ conducted a study to test the fatigue and fracture properties of the zirconia core material after being veneered with a sintered (IPS e. max Ceram) or a heat pressed veneer material (IPS e.max ZirPress) according to this study both veneering materials did not significantly affect the fatigue and fracture behaviour of the Y-TZP core.

Eleana Kontonasaki et al (2008)¹⁷ investigated the microstructural changes in three dental ceramic after their sintering according to manufacturer's instruction and evaluated their physical mechanical and biological properties.

Low fusing feldspathic ceramic, low fusing glass-ceramic and high fusing leucite based ceramics were used for this study. The low fusing glass ceramic and the high fusing leucite based cereamic presented significantly higher fracture toughness and microhardness and lower modulus of elasticity compared to the low fusing feldspathic ceramic. The biological behaviour of these ceramics were almost equivalent.

Burak Taskonak et al (2008)⁹ analyzed zirconia – base fixed partial denture using fractographic technique optical and scanning electron microscopy and found out that primary fractures initiated from the gingival surfaces of the connectors at veneer surfaces in four out of the five samples. Delamination between the glass veneer and zirconia core were observed in Y-TZP based FPDs and a secondary fractures initiated from the zirconia core. Secondary fracture controlled the ultimate failure.

Interfacial delamination in glass veneer/zirconia core dental ceramic structure controlled the fracture initiation sites and failure stresses of zirconia core. The design and dimension of the connectors as well as span size of the FPD can be the key factors in causing fractures at relatively low ocelusal loads but high fractures stresses. Anders Sundh et al (2008)² conducted a study to evaluate the bending resistance of implant – supported CAD/CAM processed restorations made out of zirconia or manually shape made out of reinforced alumina abutments. Units of titanium abutment attached to a titanium implant fixtures were used as references. Author reported that the all ceramic abutments exhibited values that were equal or superior to that of the control and exceeded the reported value, upto 300N for the maximum incisal bite forces.

Bu–Kyung Choi et al $(2009)^7$ calculated the shear bond strength of veneering porcelain to zirconia and metal core. Zirconia – based restorations have the common technical complications of delamination or porcelain chipping from the zirconia core. Thus the shear bond strength between zirconia core and veneering porcelain requires investigations in order to facilitate the materials clinical use. The study showed that there was a significant difference between the metal ceramic groups and zirconia groups in the shear bond strength. According to the author many variables may affect the zirconia core – veneer bond strength such as surface finish of the core, residual stress generated by mismatch in coefficient of thermal expansion, development of flaws and structure defects at core – veneer interface and wetting properties and volumetric shrinkage of the veneering porcelain.

Moustafa N. Aboushelib et al $(2009)^{43}$ determined the micro-tensile bond strength and impact energy of fracture of CAD – veneered zirconia restorations. A new CAD/CAM system was used to fabricate a resin replica of

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the esthetic ceramic required to veneer a framework. The replica was seated on the zirconia framework and further processed using a press-on technology. The bond strength between zirconia and the CAD veneer was evaluated using microtensile bond strength test manually layered zirconia specimens served as a control result showed that there was no significant difference in the microtensile bond either of the used veneers. Even though the impact energy of fracture of the CAD-veneered and manually layered specimens was almost identical, the former demonstrated a cohesive fracture of the veneer while the latter failed by delamination of the veneer ceramic.

Akihiko Shirakura et al $(2009)^1$ investigated the influence of veneering porcelain thickness for all ceramic and metal ceramic crowns on failure resistance after cyclic loading (1000 cycles at 5° and 55° C for 5 sec dwell time) incisal thickness used were 2 mm and 4 mm. All ceramic crowns consisted of alumina (Procera All Ceram) frame work and veneering porcelain (Cerabein) metal ceramic crowns consisted of high noble metal (leo) frame work and veneering porcelain (IPS classic).

The result showed that all ceramic crowns had significantly higher success and survival rates after cyclic loading, but lower failure loads than metal ceramic crowns. The thickness of the veneering porcelain affected the failure load of the metal ceramic crowns, but not that of the all – ceramic crowns. Metal ceramic crowns with 2 mm veneering porcelain demonstrated higher failure loads than crowns with 4 mm porcelain.

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Jens Fischer et al $(2010)^{32}$ evaluated the shear bond strength of different veneering ceramics to CE-TZP. The effect of different surface treatments (polished with 3 mm diamond paste or air-borne particle abraded) was evaluated with veneering ceramic (Cerabien ZR). Shear bond strength of 5 additional veneering ceramics (IPS e.max, Initial ZR,TriCeram, Vintage ZR or VITA VM9) to polished CE – TZP was measured. Polished Y-TZP veneered with two ceramics served as a control. Mean Shear bond strength values were calculated. Study concluded that airborne particle abrasion was not required to increase the shear bond strength of veneering ceramics to CE – TZP. The application of a liner resulted in a small but significant decrease in the shear bond strength to CE – TZP to veneering ceramics was slightly but significantly different compared to that of Y-TZP.

MATERIALS AND METHOD

The present in vitro study was conducted to evaluate the shear bond strength of veneering porcelain to base metal alloy and zirconia substructure before and after aging. Twenty base metal alloy rectangular blocks of 9mm length x 4mm height x 4mm width (Fig. 1) were prepared and veneered with corresponding veneering porcelain (3mm length x 4mm height x 4mm width) to obtain twenty samples (Fig. 23a & 23b). Samples were divided into two groups (Group I and Group II). Each group contained ten samples. Group I samples were categorized as porcelain veneered base metal alloy samples before aging. Group II samples were immersed in distilled water at 37° C (Fig.25a) for one month to simulate oral environment (aging) and Group II samples were categorized as porcelain veneered base metal alloy samples after aging.

Twenty ziroconia rectangular blocks of 9mm length x 4mm height x 4mm width (Fig.1) were prepared and veneered with corresponding veneering porcelain (3mm length x 4mm height x 4mm width) to obtain twenty samples (Fig.24a & 24b). Samples were divided into two groups (Group III and Group IV). Each group contained ten samples. Group III samples were categorized as porcelain veneered zirconia samples before aging. Group IV samples were immersed in distilled water at 37°C (Fig.25b) for one month to simulate oral environment (aging). Group IV samples were categorized as porcelain veneered zirconia samples after aging.

A total of forty test samples (both before aging groups and after aging groups) were tested for shear bond strength in Universal testing machine.

The following materials were used for the preparation of the base metal alloy core – porcelain veneer samples:

- 1. Inlay wax (GC Corporation, Tokyo, Japan) (Fig. 3a)
- 2. Sprue wax (Bego,Germany)(Fig. 3d))
- Silicon casting ring & crucible former. (Delta, Delta labs, Arumbakkam, Chennai)(fig. 3e)
- 4. Surfactant spray (Aurofilm, Bego, Germany) (Fig.3f)
- 5. Phosphate-bonded investment material (Bellasum, Bego, Germany) (Fig.3g)
- 6. Investment Liquid (Begosol, Bego, Germany) (Fig. 3h)
- 7. Base metal alloy pellets (Bellabond Plus, Bego, Germany)

(Ni-65.2%,Cr-22.5%,Mo9.5%) (Fig. 3i)

- 8. Aluminium oxide powder for air abrasion 50μ (Delta , India) (Fig.6)
- 9. Self cure clear acrylic (DPI RR polymer and monomer) (Fig.27)
- 10. Distilled water (Metro Labs, Pondichery , INDIA)

- 11. Feldspathic porcelain (Ivoclar IPS Classic Ivoclar Vivadent, Liechtenstein),C4 Shade. (Fig.10a)
- 12. Opaque porcelain, (Ivoclar- IPS Classic Ivoclar Vivadent, Liechtenstein) (Fig.10b)
- 13. Universal buildup liquid (Delta Lab, Chennai) (Fig.10c)
- 14. Ceramic Slab (Vita, Bad Sackingen, Germany) (Fig.11a)
- 15. Ceramic Holder (Ivoclar Vivadent , Liechtenstein) (Fig.11b)
- 16. Ceramic Honeycomb tray (Vita, Bad Sackingen, Germany) (Fig.11c)
- 17. Ceramic Brushes (Ivoclar Vivadent , Liechtenstein) (Fig.11d)
- 18. Tissues (Premier Aryco, India) (Fig.11e)
- 19. Glaze (Ivoclar IPS classic Ivoclar Vivadent , Liechtenstein)

The following materials were used for the preparation of zirconia core – porcelain veneer samples:

1. Will – Ceram^R ZTM Zirconia K block. (Degudent,Germany)

$$(Zr O_2 (Hf O_2) - >94 wt \%, Y_2 O_3 - 5.15 wt \% \pm 0.20,$$

Hf O₂ - <3.0 wt%, Al₂ O₃ - 0.25 wt % ± 0.10) (Fig.13)

2. CAD/CAM Wax (Al dente / dental producte dipping wax for CAD/ CAM systems, Al dente / dental producte GmbH , Germany) (Fig.14)

- 3. Cercon scan powder (Degudent, Germany) (Fig.15)
- 4. Cercon wax sticks / wax sprue (Degudent , Germany) (Fig.16)
- 5. Cercon Ceram Kiss Liner (Degudent ,Germany) (Selenium& Feldspathic porcelain) (Fig.20)

6. Cercon ceram Kiss veneering ceramics (Degudent, Germany) (Feldspathic Veneering porcelain - Si $O_2 - 60 - 70$ vol %, $Al_2O_3 - 7.5 - 12.5$ vol %, $K_2O - 7.5 - 12.5$ vol %, $Na_2O - 7.5 - 12.5$ vol %) (Fig.21)

The following lab equipments were used for the study:

- 1. Vacuum power mixer (The continental , Whip Mix, Kentucky, USA)
- 2. Burnout Furnace. (SUNBIM, INDIA) (Fig.4)
- 3. Induction Casting Machine. (Fornax GEU, Bego, Germany) (Fig.5)
- 4. Sand Blaster (Ideal Blaster, Delta labs, Delta, Chennai) (Fig.7)
- 5. Alloy Grinder (Whipmix, , USA) (Fig.8)
- 6. Steam Cleaner (OMEC, MUGGIO MILANO, Italy) (Fig.9)
- Dental Porcelain furnace (Vita Vacumat 100, Vita, Zahnfabric H, Bad Sackingen) (Fig.12)
- CAD / CAM Milling Machine (Cercon brain , Degudent, Germany) (Fig.18)

- 9. CAD/CAM Sintering Machine (Cercon heat, Degudent, Germany) (Fig.19)
- 10. Dental Ceramic furnace (Programat P 500, Ivoclar , Vivadent.) (Fig.22)
- 11. Incubator (Fig.26)
- 12. Universal Testing Machine (Model LR 100 K, Lloyd instruments, Farnham, UK) (Fig.30)
- 13. Scanning Electron Microscope (Jeol, JSM-6390LA) (Fig.33)

Description of the Universal testing machine :

The universal testing machine (Model LR 100 K, Lloyd instruments, Farnham, UK)(Fig.30) was used to test for shear bond strength of the samples used in this study. This machine rests on a table top. It consists of a lower chamber, upper chamber, a display board to display the amount of force needed to fracture the veneering porcelain from substructure, and a computer. The upper member is attached to the lower with the help of two horizontal bars, which also enclose the hydraulic pressure machine attached to upper member. The lower portion has a bench vice test specimen fixture to hold the test specimen. The upper portion has a levis grip on which a monobeveled chisel blade can be attached. The whole unit is attached to the computer for recording and converting data as required.

Description of the Scanning Electron Microscope

Scanning electron microscope (Jeol, JSM-6390LA)(Fig33) use a beam of highly energetic electrons (1 KeV-1MeV) to examine objects on a very fine scale (0.2nm onwards). They can reveal the fine structure of variety of materials. As the name suggests, SEM uses a scanned beam rather than a fixed beam. It is used primarily for the examination of thick (i.e. electron opaque) samples. The specimens to be magnified may have some conductivity and may get charged up. Hence they are coated with a platinum layer to prevent the charging up and in order to increase the secondary emissions. Sometimes the specimens may be coated with tungsten when higher magnification is essential. The incident electron probe scans the sample surface and the signals produced are used to modulate the intensity of a synchronously scanned beam on a CRT screen. The electrons which are back scattered from the specimen are collected to provide (i) topographical information if low energy secondary electrons are collected (ii) atomic number and reorientation information if the higher energy, back scattered electrons are used, or if the leakage current to the earth is used. The magnification is given immediately by the ratio of the CRT scan size to the specimen scan size.

Methodology :

I . Preparation of the base metal alloy core – porcelain veneer samples(Fig. 23a):

A. Preparation of base metal alloy substructure

B. Veneering of base metal alloy substructure with porcelain

A. Preparation of base metal alloy substructure :

- 1. Wax pattern fabrication for base metal alloy substructure.
- 2. Investment procedure of wax pattern
- 3. Burnout of wax pattern and casting for base metal alloy substructure.
- 4. Finishing of base metal alloy substructure.

B. Veneering of base metal alloy substructure with porcelain :

- 1. Preparation of base metal alloy substructure prior to ceramic application.
- 2. Opaque layer application.
- 3. Application of body ceramic.
- 4. Glazing of samples.

A. Preparation of base metal alloy substructure :

1. Wax pattern fabrication for base metal alloy substructure :

Twenty blocks of size 9mm length x 4mm height x 4mm width were fabricated using Inlay wax(GC Corporation, Japan)(Fig.3a). Each wax pattern dimensions were checked for accuracy using metal scale and caliper. Sprues (Bego,Germany)(Fig.3d) of 2.5mm diameter and 13mm length were attached to the patterns. The other ends of the sprues were attached to the crucible former. The wax patterns were sprayed with wax surfactant spray (Aurofilm, Bego,Germany)(Fig.3f) to improve wettability of wax patterns.

2. Investment procedure of wax patterns :

Suitable size of the silicon casting ring (Delta, Chennai)(Fig.3e) was selected and positioned on the crucible former around the prepared wax pattern. The phosphate-bonded investment material (Bellasum, Bego, Germany)(Fig.3g) was mixed with investment liquid (Begosol , Bego, Germany)(Fig.3h) in a vacuum power mixer machine(The Continental, WhipMix ,USA) and the prepared wax patterns were invested. Since the ring less casting procedure was adopted in this study, the silicon casting ring was removed after the investment material had set.

3. Burnout of wax pattern and casting for base metal alloy substructure :

The investment mold was placed in the burnout furnace set (Sunbim,India)(Fig.4) at room temperature. Investment mold was allowed to heat continuously till 950°C at the rate of 8°C / min and held for 30 min at 950°C. Casting procedure was performed quickly to prevent heat loss from the mold. After burnout, investment mold was taken out of the furnace and were placed in the casting machine. Casting was done in induction casting machine (Fornax GEU, Bego, Germany)(Fig.5). The Nickel - Chromium alloy (Bellabond plus, Bego, Germany)(Fig.3i) was heated sufficiently (melting range $1325^{\circ}C - 1370^{\circ}C$ Casting temp1450°C) till the alloy ingot turned in to molten state and the crucible was released and centrifugal force ensured completion of casting procedure. Investment with cast was allowed to cool down to room temperature . Divestment was done and casting was retrieved . Sprues were cut with carborundum disk (LM Pianotti S.r.l, ITALY) The same procedure was carried out for all samples . A total of twenty samples were obtained.

4. Finishing of base metal alloy substructure :

Heatless stone (Mizzy, USA) was used to reduce the sprue attached area of the base metal alloy substructure . Finishing of base metal alloy substructure was done with a clean ceramic – bound abrasive. B. Veneering of base metal alloy substructure with porcelain :

1. Preparation of base metal alloy substructure prior to ceramic application :

The surface of the rectangular base metal alloy block (4mm x 4mm area) which had to be veneered with porcelain was sand blasted with 50 μ Al₃O₂ particles (Delta, India)(Fig.6) and steam cleaned prior to addition of feldspathic porcelain (Ivoclar- IPS classic , Ivoclar Vivadent , Liechtenstein)(Fig.10a,b,c).

2.Opaque layer application

Two layers of opaque porcelain were applied to the base metal alloy surface and fired.C4 shade was used to veneer the base metal alloy substructure. The porcelain firing procedure was done in a dental porcelain furnace (Vita Vacumat100, Vita ZahnfabricH, BadSackingen)(Fig.12) following the manufacturer's recommendations as mentioned below:

Firing Schedule for Feldspathic Porcelain

Procedure	T Max (°C)	Pre heat (Mins)	Heat Up Rate(Min)	PeakTemp (mins)	Vacuum time (mins)
I Opaque	980	4	6	1	6
II Opaque	970	4	6	1	6
I/II Body	920/910	4	8/9	1	8/9
Glaze	870	4	8	1	8

3.Application of body porcelain:

Dentin porcelain of same shade was applied over the same area and fired. The excess porcelain was removed by using a sintered diamond bur with a low speed handpiece. So that the final dimension of the veneering ceramic was 3mm length x 4mm height x 4mm width.

4.Glazing of samples :

The samples were finished and glazed.

In this manner, twenty porcelain veneered base metal alloy samples (Fig.23b) were prepared and divided into two groups (Group I and Group II). Each group contained 10 samples . The group I test samples were used to determine the shear bond strength before aging. The group II test samples were used to determine the shear bond strength after aging.

Aging of the test samples :

Group II samples were immersed in distilled water in stainless steel tray with lid (Fig.25a) and kept in an incubator at temperature of 37°C for one month to simulate oral environment (aging) prior to testing .

II. Preparation of the zirconia core - porcelain veneer samples (Fig.24a):

- A. Preparation of zirconia substructure (core)
- **B.** Veneering of zirconia substructure
 - A. Preparation of zirconia substructure (core):
 - 1. Preparation of wax pattern for zirconia substructure
 - 2. Copy milling of zirconia substructure
 - 3. Sintering of zirconia substructure
 - B. Veneering of zirconia substructure

A. Preparation of zirconia substructure (core):

1. Preparation of wax pattern for zirconia substructure :

The required dimension for the zirconia substructure in the present study was 9mm length x 4mm height x 4mm width. CAM system was used for this study. CAD/CAM wax (dental producte GmbH,Germany) (Fig.14) was used to make a rectangular block having dimension of 9mm x 4mm x 4mm. Prior to mounting the prepared wax block in the milling machine, Ag scan powder (cercon scan powder, Degudent, Germany) (Fig.15) was applied over the wax block for scanning. This wax block was sprued with wax stick (cercon wax sticks)(Fig.16) and mounted on the rectangular scanning frame (Fig.17), which was in turn attached to the milling machine (Fig.18).

2. Copy milling of zirconia substructure :

In the present study, zirconia substructure was obtained by copy milling of zirconia block ("green" state). Will – Ceram^R Z TM Zirconia K block (Fig.13) was used obtain the zirconia substructure . CAD/CAM Brain (Milling Machine, Degudent, Germany)(Fig.18) was used to mill the zirconia block in desired dimension. Milling of the block was done with an enlargement factor of approximately 1. 26 (or 26 %) relative to the final desired dimension. This compensated for the shrinkage that occured during full sintering.

3. Sintering of the zirconia substructure :

The zirconia block ("green" state) was sintered in a sintering furnace (Cercon heat, Degudent, Germany)(Fig.19). The sintering temperature for the rectangular zirconia block was 1500°C. The sintering cycle as suggested by the manufacturer was followed :

Sintering Cycle

Ramp I	2°C / min to 500°C
Hold 1	500°C * 1hr
Ramp 2	5 – 10°C / min to 1500°C
Hold 2	1500°C * 2 hrs
Cooling	Natural cool
Opening	Wait until below 400°C

In this manner, twenty zirconia cores with required dimensions of 9mm length x 4mm width x 4mm height were obtained.

B.Veneering of zirconia substructure with porcelain :

The surface of the rectangular zirconia block (4mm x 4mm area) which had to be veneered with corresponding veneering porcelain was sand blasted with $50 \ \mu \ Al_3O_2$ particles at a pressure of 4 psi for 30 sec according to manufacturer's recommendation . After sand blasting, steam cleaning was done for 15 sec and air dried. Liner (Cercon ceram kiss liner , Degudent, Germany)(Fig.20) was applied and fired . Veneering was done using the layering technique as recommended by the manufacturer as given in the firing table below. The veneering ceramic (C4 shade) (Cercon ceram kiss , Degudent , Germany) (Fig.21) was built up to the final dimension and fired according to the firing program of the manufacture . Due to the shrinkage of the porcelain three separate firings were required to the establish the correct dimension of veneering ceramic as 3mm length, 4mm height and 4mm width.

Firing	Preheating °C	Drying time min	Heating rate °C/min	Final tem °C	Hold time	Vaccum min hpa
Powder liner1	450	6	55	970	1:00	50
Powder liner 2	450	6	55	960	1:00	50
Paste liner 1	575	8	55	970	1:00	50
Paste Liner 2	575	8	55	960	1:00	50
Shoulder 1+2	450	6	55	850	1:30	50
Dentin 1	450	5	55	830	1:30	50
Dentin 2	450	5	55	820	1:30	50
Glaze	450	3	55	800	1:00	-
Correction (final)	450	5	55	680	1:00	50
Final Shoulder(FSM)	450	5	55	680	1:00	50

General Recommendations for firing – Cercon Ceram kiss

In this manner, twenty porcelain veneered zirconia samples (Fig. 24b) were prepared and divided into two groups (Group III and Group IV). Each group contained ten samples. The Group III test samples were used to determine the shear bond strength before aging. The Group IV test samples were used to determine the shear bond strength after aging.

Aging of the test samples :

Group IV samples were immersed in distilled water in a stainless steel tray with lid (Fig.25b) and kept in an incubator at 37°C for one month to simulate the oral environment (aging) prior to testing.

Mounting of samples for shear bond strength test :

Each test sample was embedded in the self cure clear acrylic (DPI-RR polymer and monomer – Fig.27) which was confined within a GI pipe mold of dimension 5mm width and 20mm diameter (Fig. 2a). The level of the core- veneer interface of test samples were positioned to enable evaluation of shear bond strength with the Universal testing machine(Model LR 100 K, Lloyd instrument,UK)(Fig.30). In this manner all the forty test samples were mounted for the evaluation of shear bond strength.

Test for shear bond strength :

A total of forty test samples (Group I, II, III & IV) were tested for shear bond strength in a Universal testing machine. Test sample was fixed to the sample fixture at the bench vice of the machine with the monobevelled chisel blade placed adjacent to and directly to the bonding interface(Fig.2b, Fig 31). Force was applied to the sample so that shear load was exerted adjacent to and directly to the bonding interface at a cross head speed of 0.5mm/min until fracture occurred. Load deflection curves and ultimate load to failure were recorded automatically and displayed by the computer software of the testing machine. Shear bond force was recorded in Newton, and shear bond strength (MPa) was calculated through dividing the load (N) at which failure occurred by the bonding area (mm²)

Bond strength (MPa) = load (N) \div surface area (mm²)

The basic values of shear bond strength of all samples in four groups were tabulated. The mean shear bond strength for each group was calculated and tabulated for statistical analysis.

Statistical Analysis :

The data was analyzed using the software SPSS 10.0 . Descriptive statistics was used to find the mean and standard deviation of variables. Independent student T-test was used to compare the bond strength between groups. P<0.05 was considered as the level of significance.

SEM Analysis and EDX analysis :

To determine the mode of failure , the fractured samples were examined under scanning electron microscopy (Jeol, JSM-6390LA)(Fig.33) under 30x and 250x magnification. (Fig. 34 to Fig. 37). Surface chemistry was analyzed using Energy dispersive X-ray microanalysis (EDX analysis). The failure modes were presented along with the results.

METHODOLOGY



- Group I Porcelain veneered base metal alloy samples before aging
- Group II Porcelain veneered base metal alloy samples after aging
- Group III Porcelain veneered zirconia samples before aging
- Group IV Porcelain veneered zirconia samples after aging



Fig. 1 : Schematic representation of dimension of sample



Fig. 2a : Schematic representation of sample embedded in the mold Fig. 2b : Schematic representation of Shear bond strength testing of sample



Fig 3 : Materials used in the laboratory for the fabrication of base metal alloy substructure

a. Inlay wax, b. Scale, c. Metal Caliper, d. Sprue Wax, e. Silicon casting ring and Crucible former, f. Surfactant spray, g. Phosphate- bonded investment material, h.Investment liquid, i. Base metal alloy pellets



Fig.4: Burnout Furnace



Fig. 5: Induction Casting Machine



Fig. 6. Aluminium Oxide Powder (50 µ)



Fig. 7 :Sand Blaster



Fig. 8: Alloy Grinder



Fig.9 : Steam Cleaner



Fig. 10a: Feldspathic Porcelain 10b: Opaque Porcelain 10c : Universal buildup liquid



Fig.11 : Laboratory tools required for porcelain veneering a. Ceramic Slab, b. Ceramic holder, c. Ceramic honeycomb tray, d.Ceramic brush, e. Tissue



Fig.12: Dental Porcelain Furnace



Fig. 13: Zirconia Block



Fig. 14: CAD/CAM Wax



Fig. 15: Scan powder



Fig. 16 : Cercon Wax Stick



Fig. 17: Rectangular Block of CAD//CAM Wax attached to the rectangular scanning frame



Fig.18: CAD/CAM Milling Machine



Fig. 19 : CAD/CAM Sintering Machine


Fig.20: Cercon – Ceram Kiss Liner



Fig.21: Cercon Ceram Kiss veneering Ceramics



Fig.22 : Dental Ceramic Furnace (**Programat P – 500, Ivoclar Vivadent**)



а

Fig. 23a: Porcelain veneered base metal alloy sample



b

Fig. 23b :Twenty samples of porcelain veneered base metal alloy



Fig. 24a: Porcelain veneered Zirconia sample



Fig. 24b : Twenty samples of porcelain veneered Zirconia



a b Fig. 25: Samples immersed in distilled water for aging a. Porcelain veneered base metal alloy Samples, b. Porcelain veneered Zirconia Samples



Fig.26: Incubator



Fig 27 : Self Cure Clear Acrylic



Fig.28: Porcelain veneered base metal alloy Sample embedded in the mold





Fig.29: Porcelain veneered Zirconia Sample embedded in the mold



Fig 30 : Universal Testing Machine



Fig. 31: Sample testing in Universal Testing Machine



Fig.32 : Samples with fractured piece a. Porcelain veneered base metal alloy sample with fractured piece b. Porcelain veneered Zirconia sample with fractured piece



Fig. 33: Scanning Electron Microscope

RESULTS

The present in vitro study was conducted to evaluate and compare the shear bond strength of veneering porcelain to base metal alloy and zirconia substructures before and after aging.

A total of forty test samples were prepared and were randomly divided into four test groups of ten samples each (Group I,II,III,&IV). Twenty base metal alloy core - porcelain veneer samples were prepared and divided into two groups (Group I and Group II). Each group contained 10 samples. The Group I test samples were used to determine the shear bond strength before aging. The Group II test samples were used to determine the shear bond strength after aging. Twenty zirconia core - porcelain veneer samples were prepared and divided into two groups (Group III and Group IV). Each group contained 10 samples. The Group III test samples were used to determine the shear bond strength before aging. The Group IV test samples were used to determine the shear bond strength after aging. All samples were tested for shear bond strength in Universal testing machine. The basic values of shear bond strength of all test samples in four groups were tabulated. The mean shear bond strength for each group was calculated and tabulated. The results were subjected for statistical analysis . Tested samples were subjected to qualitative analysis using scanning electron microscopy and energy dispersive X-ray microanalysis.

- Group I \rightarrow Porcelain veneered base metal alloy samples before aging
- Group II \rightarrow Porcelain veneered base metal alloy samples after aging
- Group III \rightarrow Porcelain veneered zirconia samples before aging
- Group IV \rightarrow Porcelain veneered zirconia samples after aging

Samples	SHEAR BOND STRENGTH (MPa)
1	38.8
2	35.8
3	40.3
4	39.4
5	34.2
6	43.2
7	39.6
8	41.7
9	44.8
10	37.3
Mean Value	39. 51

Table 1 - Basic values of shear bond strength of veneering porcelain tobase metal alloy substructure before aging (Group I)

Table 2 – Basic values of shear bo	ond strength of veneering porcelain to
base metal alloy substructure after	er aging (Group II)

Samples	SHEAR BOND STRENGTH (MPa)
1	35.8
2	40.3
3	41.2
4	36.3
5	31.1
6	38.0
7	34.9
8	35.5
9	42.3
10	36.6
Mean value	37.2

Table 3: Basic values of shear bond strength of veneering porcelain tozirconia substructure before aging (Group III)

Samples	SHEAR BOND STRENGTH (MPa)
1	30.2
2	26.1
3	28.1
4	29.2
5	27.0
6	28.2
7	30.9
8	28.2
9	25.3
10	28.0
Mean value	28.12

Table 4 : Basic values of shear bond strength of veneering porcelain tozirconia substructure after aging (Group IV)

Samples	SHEAR BOND STRENGTH (MPa)
1	26.7
2	25.8
3	24.6
4	26.9
5	25.8
6	28.4
7	27.1
8	24.5
9	26.8
10	25.4
Mean value	26. 2

Table 5- Mean Shear Bond Strength obtained from basic values of four

Groups (Group I, II, III & IV)

	Group I	Group II	Group III	Group IV
Mean (MPa)	39.51	37.2	28.12	26.2

Table 5 shows the mean shear bond strength of veneering porcelain to base metal alloy and zirconia substructure before and after aging obtained from basic values of four groups (Group I, Group II, Group III and Group IV) calculated in MegaPascal (MPa).

Statistical analysis :

The data was analyzed using the software SPSS 10.0. Mean and Standard deviations were estimated from the samples of each study group. Descriptive statistics was used to find the mean and standard deviation of variables. Independent student T – test was used to compare the bond strength between groups. P < 0.05 was considered as the level of significance.

Table 6 - Comparison between mean obtained from Group I and Group II(Independent student T- test)

	No.of samples	Mean	SD	P – Value	
Porcelain veneered base metal alloy samples before aging (Group I)	10	39.51	3.2384		
Porcelain veneered base metal alloy samples after aging (Group II)	10	37.20	3.3463	P = 0.134	
D = 0.124					

P = **0.** 134

Inference : There was no statistically significant difference between shear bond strength of veneering porcelain to base metal alloy substructure before and after aging (Group I and Group II)

Table 7 - Comparison between mean obtained from Group III andGroup IV (Independent student T- test)

	No.of samples	Mean	SD	P – Value
Porcelain veneered ziroconia samples before aging (Group III)	10	28.12	1.717	P = 0.010*
Porcelain veneered zirconia samples after aging (Group IV)	10	26.20	1.2092	1 - 0.010

P = 0. 010

Note : * denotes significance at 5% level.

Inference : Statistically significant difference was evidenced in the shear bond strength of veneering porcelain to zirconia substructure before and after aging (Group III and Group IV)

Table 8 - Comparison between mean obtained from Group I and Group III(Independent student T – Test)

	No.of samples	Mean	SD	P - Value
Porcelain veneered base metal alloy samples before aging (Group I)	10	39.51	3.2384	P – 0.000*
Porcelain veneered zirconia samples before aging (Group III)	10	28.12	1.1717	F = 0.000*

P = **0.000**

Note : * denotes significance at 5% level.

Inference : In before aging groups, statistically significant difference in shear bond strength was evidenced between the materials, porcelain veneered base metal alloy and porcelain veneered zirconia (Group I and Group III)

Table 9 - Comparison between mean obtained from Group II and Group IV(Independent Student T – Test)

	No.of samples	Mean	SD	P – Value
Porcelain veneered base metal alloy samples after aging (Group II)	10	37.20	3.3463	
Porcelain veneered zirconia samples after aging (Group IV)	10	26.20	1.2092	P = 0.000*

P = 0.000

Note : * denotes significance at 5% level.

Inference : In after aging groups, statistically significant difference in shear bond strength was evidenced between the materials, porcelain veneered base metal alloy and porcelain veneered zirconia (Group II and Group IV).

Graph 1,2,3 and 4 shows the basic data of the results obtained in this study for the shear bond strength of samples in the Group I, Group II, Group III and Group IV respectively. Graph 5 shows Comparison of mean shear bond strength obtained from basic values of four Groups.

Graph:1

Basic values of shear bond strength of veneering porcelain to base metal alloy substructure before aging (Group I)



Graph :2

Basic values of shear bond strength of veneering porcelain to base metal alloy substructure after aging (Group II)



Graph :3

Basic values of shear bond strength of veneering porcelain to zirconia substructure before aging (Group III)



Graph :4

Basic values of shear bond strength of veneering porcelain to zirconia substructure after aging (Group IV)





Comparison of mean shear bond strength obtained from basic values of

four groups (Group I, II, III & IV)



Qualitative analysis of Group I test samples by Scanning Electron Microscopy (SEM) under 30x and 250x magnification and Energy Dispersive X-ray microanalysis(EDX analysis) - Fractured interface of the core surface





Fig. 34 b: Group I

- Fig. 34 a : Tested porcelain veneered base metal alloy sample before aging under 30x magnification
- Fig. 34 b : Tested porcelain veneered base metal alloy sample before aging, under 250x magnification
- **Note :** The Arrow indicates the direction of load.



Graph:6 Energy Dispersive X– ray microanalysis of fractured interface of the core surface (Group I)

Qualitative analysis of Group I test samples by Scanning Electron Microscopy(SEM) under 30x and 250x magnification and Energy Dispersive X-ray microanalysis (EDX analysis)- Fractured veneer surface



Fig.34c : Group I

Fig. 34 d: Group I

Fig. 34 c : Fractured veneer surface (Group I), under 30x magnification





Graph :7 Energy Dispersive X– ray microanalysis of fractured veneer surface (Group I)

Inference (GroupI) : The arrow indicated the direction of load. The loaded side demonstrated predominantly cohesive failure within the veneering porcelain. 30x magnification of base metal alloy sample before aging (Fig.34a) revealed a combination of cohesive failure of veneering ceramic and cohesive failure of metal oxide. 250x magnification of fractured core surface (fig.34b) showed numerous pores within the veneering porcelain and in the metal oxide layer. 30x magnification (Fig.34c) of fractured veneer surface revealed the presence of metal oxide and the ceramic material. 250x magnification (Fig.34d) of fractured veneer surface revealed numerous pores within the ceramic and also in the metal oxide. Chemical composition of the fractured core surface and the fractured veneer surface were analyzed using energy dispersive X-ray microanalysis (EDX analysis). Surface chemistry of the fractured core surface (Graph:6) explained the elements seen on the surface of fractured core and revealed the presence of silica, alumina, sodium, potassium, chromium, nickel, oxygen and carbon. The total count of silica was found to be higher indicating predominantly cohesive failure of veneering ceramic. Surface chemistry of fractured veneer surface (Graph:7) revealed the presence of silica, alumina, sodium, potassium, chromium, nickel, oxygen and carbon. Since the silica content was higher, the surface chemistry indicated predominantly cohesive failure of veneering porcelain. Graphical representation of surface chemistry was presented along with SEM images of corresponding samples.

Qualitative analysis of Group II test samples by Scanning Electron Microscopy (SEM) under 30x and 250x magnification and Energy Dispersive X-ray microanalysis(EDX analysis) -Fractured interface of the core surface



- Fig. 35 a : Tested porcelain veneered base metal alloy sample after aging, under 30x magnification
- Fig. 35 b : Tested porcelain veneered base metal alloy sample after aging, under 250x magnification
- Note : The Arrow indicates the direction of load.



Graph :8 Energy Dispersive X – ray microanalysis of fractured interface of the core surface (Group II)

Qualitative analysis of Group II test samples by Scanning Electron Microscopy (SEM) under 30x and 250x magnification and Energy Dispersive X-ray microanalysis(EDX analysis) -Fractured veneer surface





Fig.35c : Group II

Fig. 35 d: Group II

Fig. 35 c : Fractured veneer surface (Group II), under 30x magnification

Fig. 35 d : Fractured veneer surface (Group II), under 250x magnification



Graph :9 Energy Dispersive X– ray microanalysis of fractured veneer surface (Group II)

Inference (Group II): The arrow indicated the direction of load. The loaded side demonstrated cohesive failure within the veneering ceramic. 30x magnification of tested base metal alloy samples after aging (Fig.35a) showed a cohesive failure of veneering ceramic. Higher magnification (250x) (Fig.35b) showed small pores in the veneering ceramic layer over the base metal alloy surface. 30x (Fig.35c) and 250x (Fig.35d) magnification of fractured veneer surface showed numerous pores within the veneering ceramic. Chemical composition of the fractured core surface and the fractured veneer surface were analyzed using energy dispersive X-ray microanalysis (EDX analysis). Surface chemistry of the fractured core surface (Graph:8) explained the elements seen on the surface of fractured core and revealed the presence of silica, alumina, sodium, potassium, oxygen and carbon. The elements seen over the fractured core surface indicated cohesive failure of veneering ceramic. Surface chemistry of fractured veneer surface (Graph:9) revealed the presence of silica, alumina, sodium, potassium, oxygen and carbon. Elements which were presented over the fractured veneer surface indicated cohesive failure of veneering ceramic. Graphical representation of surface chemistry was presented along with SEM images of corresponding samples.

Qualitative analysis of Group III test samples by Scanning Electron Microscopy (SEM) under 30x and 250x magnification and Energy Dispersive X-ray microanalysis (EDX analysis) –Fractured interface of the core surface



Fig.36a : Group III

Fig. 36b Group III

- Fig. 36 a : Tested porcelain veneered zirconia sample before aging, under 30x magnification
- Fig. 36 b : Tested porcelain veneered zirconia sample before aging, under 250x magnification



Note : The Arrow indicates the direction of load.

Graph :10 Energy Dispersive X – ray microanalysis of fractured interface of the core surface (Group III)

Qualitative analysis of Group III test samples by Scanning Electron Microscopy (SEM) under 30x and 250x magnification and Energy Dispersive X-ray microanalysis (EDX analysis)-Fractured veneer surface



Fig.36c : Group III

Fig. 36 d: Group III

Fig. 36 c:Fractured veneer surface (Group III), under 30x magnificationFig. 36 d:Fractured veneer surface (Group III), under 250x magnification



Graph :11 Energy Dispersive X– ray microanalysis of fractured veneer surface (Group III)

Inference (Group III) : The arrow indicated the direction of load. The loaded side demonstrated cohesive failure within the veneering ceramic. 30x magnification of tested zirconia samples before aging (Fig.36a) revealed a mixed cohesive and adhesive failure of veneering ceramic, predominantly cohesive failure of veneering ceramic exposing zirconia core in some areas. Higher magnification (250x) (Fig.36b) showed small pores within the veneering ceramic. 30x (Fig.36c) and 250x (Fig36d) magnification of fractured veneering ceramic surface revealed numerous pores within the veneering ceramic. Chemical composition of the fractured core surface and the fractured veneer surface were analyzed using energy dispersive X-ray microanalysis (EDX analysis). Surface chemistry of the fractured core surface (Graph:10) explained the elements seen on the surface of fractured core and revealed the presence of silica, alumina, sodium, potassium, oxygen, carbon and zirconia . The elements seen over the fractured core surface indicated mixed cohesive and adhesive failure of veneering ceramic exposing some areas of zirconia core. Since the silica content was higher, the surface chemistry indicated predominantly cohesive failure of veneering ceramic. Surface chemistry of fractured veneer surface (Graph:11) revealed the presence of silica, alumina, sodium, potassium, oxygen, carbon, zinc and titanium. Elements which were presented over the fractured veneer surface indicated predominantly cohesive failure of veneering ceramic. Graphical representation of surface chemistry was presented along with SEM images of corresponding samples.

Qualitative analysis of Group IV test samples by Scanning Electron Microscopy (SEM) under 30x and 250x magnification and Energy Dispersive X-ray microanalysis (EDX analysis)- Fractured interface of the core surface



Fig.37a:Group IV

Fig.37b: Group IV

- Fig. 37 a : Tested porcelain veneered zirconia sample after aging, under 30x magnification
- Fig. 37 b Tested porcelain veneered zirconia sample after aging, under : 250x magnification



Note : The Arrow indicates the direction of load.

> Graph :12 Energy Dispersive X-ray microanalysis of fractured interface of the core surface (Group IV)

Qualitative analysis of Group IV test samples by Scanning Electron Microscopy (SEM) under 30 x and 250x magnification and Energy Dispersive X-ray microanalysis(EDX analysis)-Fractured veneer surface



Fig.37 c : Group IV

Fig. 37 d: Group IV



Fig. 37 d : Fractured veneer surface (Group IV), under 250x magnification



Graph :13 Energy Dispersive X– ray microanalysis of fractured veneer surface (Group IV)

Inference (Group IV): The arrow indicated the direction of load. The loaded side demonstrated cohesive failure within the veneering ceramic. 30x magnification of tested zirconia samples after aging (Fig.37a) revealed a mixed cohesive and adhesive failure of veneering ceramic, predominantly cohesive failure of veneering ceramic exposing zirconia core in some areas. Higher magnification (250x) (Fig.37b) showed small pores within the veneering ceramic. 30x (Fig37c) and 250x (Fig37d) magnification of fractured veneering ceramic surface revealed numerous pores within the veneering ceramic. Chemical composition of the fractured core surface and the fractured veneer surface were analyzed using energy dispersive Xray microanalysis (EDX analysis). Surface chemistry of the fractured core surface (Graph:12) explained the elements seen on the surface of fractured core and revealed the presence of silica, alumina, sodium, potassium, oxygen, carbon and zirconia . The elements seen over the fractured core surface indicated mixed cohesive and adhesive failure of veneering ceramic exposing some areas of zirconia core. Since the silica content was more, the surface chemistry indicated predominantly cohesive failure of veneering ceramic. Surface chemistry of fractured veneer surface (Graph:13) revealed the presence of silica, alumina, sodium, potassium, magnesium, iron, oxygen, carbon and calcium. Elements which were presented over the fractured veneer surface indicated predominantly cohesive failure of veneering ceramic. Graphical representation of surface chemistry was presented along with SEM images of corresponding samples.

DISCUSSION

Core veneered restorations are the cornerstone for prosthetic dentistry, and combination of a strong core and an esthetic veneer ceramic has proven successful for many decades. Porcelain-fused-to-metal restorations have been in use for more than five decades due to their improved mechanical properties, esthetics and biocompatibility. However, the need for the superior esthetic and biocompatibility led to a material shift, as all ceramic core materials are currently replacing dental casting alloys, but the principle itself remains the same.⁴⁴

Due to strength limitations, application of all-ceramic core material was limited to three-or four-unit fixed partial denture restoration and where gnathologic conditions, like the occlusal relation and functional stresses, are optimal^{4,44}. The introduction of tetragonal Zirconia polycrystals (TZP) as a restorative core material opened the design limits of all ceramic restoration to extensive multiunit reconstructions with high confidence and success rate. The unique chemical stability, the superior mechanical properties, and the esthetic color combined with CAD/CAM technology, makes zirconia the core material of choice^{3,4,44}.

The flexural strength of Zirconia ranges from 900- 1200 MPa and fracture toughness of 9-10 MPa.m^{1/2} which is very high compared to other dental ceramics⁷. In addition to that, Y-TZP presents a stress induced phase

transformation mechanism that make this material more resistant to crack propagations as a result of tetragonal to monoclinic (t-m) transformation which is accompanied by a volumetric expansion that closes crack tips and superimposes compressive stresses on the existing stress⁴⁸. Current processing technologies unfortunately cannot make zirconia frameworks as translucent as natural teeth, so they have to be veneered with porcelain to achieve acceptable esthetics .¹⁸

In contrast to metal ceramic restoration, the use of all-ceramic restoration may be limited in certain clinical situation, for instance, when treatments involve short clinical crowns, patients with parafunctional habits, fixed /removable combination prosthesis, and long span fixed partial denture. The use of all-ceramic fixed partial dentures is limited by the dimensional requirement of the connectors. However, metal ceramic restoration demonstrate higher versatility in terms of margin an frame work design which may facilitate their use in demanding clinical scenarios, such as situations requiring long-span fixed partial dentures with non-rigid connectors^{3,11,25,55}.

The success of metal ceramic restoration has been evaluated clinically. It was reported that the percentage failure for crowns and fixed partial denture at the 15- year follow-up was 4.9% and 4.0% respectively and the relative risk of restorative failures for each was 0.859 and 0.606 respectively. Walton

reported 85% survival of metal ceramic fixed partial dentures followed for 15 years^{25,55}.

There were few short term clinical studies addressing the clinical performance of ZrO_2 based restorative systems. These prospective evaluation of the clinical performance of ZrO_2 - based FPDs were performed for a maximum of 5 yrs. Most of the restorations failed biologically because of recurrent caries and endodontic complications. Mechanical issues were related to minor porcelain chipping which did not require replacement of the restorations.²⁵Raigrodski et al in a study of posterior 3 unit fixed partial dentures, observed minor veneer chipping in 25% of cases after a mean follow – up of 31.2 months⁵. Irena Sailer et al in a clinical study reported the success rate of 3 to 5 unit zirconia frame works for posterior fixed partial denture after 5 years of clinical observation. The success rate of zirconia framework was 97.8% . However the survival rate was 73.9% due to other complications. Secondary caries was found in 27.7% of the fixed partial denture, and chipping of the veneering ceramic in 15.2%..²⁹

According to clinical studies the Y-TZP core ceramic exhibited high stability as a framework material. Fractures of the Zirconia framework have not been reported so far⁷. However, delamination or minor chip-off fracture of veneering porcelain was described as the most frequent reason for the failures of Zirconia fixed partial dentures. Therefore, the bond between core and veneer or the veneer material itself is one of the weaknesses in layered

ziroconia based restorations and plays a significant role in their long term success²⁹.

In order to gain the strength benefits of the core material, the coreveneer bond strength must be of adequate strength and toughness to transmit functional stresses from esthetic veneer to underlying framework⁴⁴. The success of a metal-ceramic restoration depends primarily on strong adhesion between the porcelain and alloy. The adhesion mechanism between metal and porcelain is believed to be the micro-mechanical bond, compatible coefficient of thermal expansion match, van der Waals force and mainly the suitable oxidation of metal and interdiffusion of ions between the metal and porcelains^{3,11,57,62}.

However, the bonding mechanisms for veneering ceramic to the zirconia are upto now unclear. According to investigation on the wettability of the zirconia core with the veneering ceramic, micromechanical interactions were merely regarded⁷. Many variable may affect the Zirconia core-veneer bond strength; such as the surface finish of the cores, which can affect mechanical retention; residual stress generated by mismatch in coefficient of thermal expansion; development of flaws and structure defects at core-veneer interface; and wetting properties and volumetric shrinkage of the veneer²⁴.

It has long been documented that presence of water will degrade the strength of silicate glasses and many other ceramic materials (Shand 1958, Mould 1959). Exposure to an aqueous environment has also been found to

affect the mechanical properties of dental ceramics. Sherril and O'Brion in 1974 demonstrated that fracture stress of aluminous and feldspathic porcelain decreased by nearly 30% when samples were broken in water. Southan and Jorgensen (1974) showed that the ability of a dental porcelain to sustain a static load in water decreased as the duration of load application increased. The effect of aqueous exposure and other aspects of dental porcelain mechanical behavior have been the subject of an extensive review by Jones (1983) who also lists decreasing strength with decreasing stress/strain rate as further evidence for the detrimental role played by water^{16,42}.

The process of strength degradation of ceramic in aqueous environment is believed to be caused by a stress-corrosion process involving the stable growth of small, pre-existing flaws (Hillig and Charles,1965; Michalske and Frieman,1983). The effect to water is so pronounced that stress corrosion has been observed in silicate glasses at moisture levels as low as 0.017% relative humidity (Wiederhorn, 1967). The oral environment would appear to have all the factors necessary for fatigue failure to occur in ceramicbased dental prosthesis. Water is, of course, the primary chemical species in saliva. A dental restoration would also be exposed to water from a cementing agent as well as from the dentin tubules. Stresses, both masticatory- related and also associated with thermal expansion mismatches between the various components of the restoration would be present to provide the driving force for fatigue⁴².

In a survey of the literature, few articles utilized various bond strength test methods for all-ceramic core and veneering ceramic, such as the shear bond strength test²⁶, three and four point loading test ⁶¹, biaxial flexural strength test, and the microtensile bond strength test^{43,44} However, each test has a common limitation which is the difficulty in determining the core-veneer bond strength from applied force at failure on the sample in the specific test setup. Many authors in the literature suggested the use of shear bond strength test as one of the most reliable method to evaluate the bond strength because it concentrates the applied tension on the interface between two materials.⁵⁴ However , shear bond strength test has some disadvantages such as high standard deviation, occurrence of non-uniform interfacial stresses, and influence from specimen geometry.⁷

For improving the clinical usefulness of shear bond strength test, the standardization of specimen preparation, cross-sectional surface area, rate of loading application are important .The specimens tested in this study were fabricated in rectangular forms (9mm length x 4mm height x 4mm width) so as to standardize the cross-sectional area easily.

In the view of above considerations, the present invitro study was conducted to evaluate and compare the shear bond strength of veneering porcelain to base metal alloy and zirconia substructure before and after aging. Total of forty samples were prepared and were randomly divided into four test groups of ten samples each. Twenty base metal alloy core – porcelain veneer samples were prepared and divided into two groups (Group I and Group II). Each group contained 10 samples. The Group I test samples were used to determine the shear bond strength before aging. The Group II test samples were used to determine the shear bond strength after aging. Twenty zirconia core – porcelain veneer samples were prepared and divided into two groups (Group III and Group IV). Each group contained 10 samples. The Group III test samples were used to determine the shear bond strength before aging. The Group IV test samples were used to determine the shear bond strength after aging.

All the samples were mounted in GI pipe using self cure clear acrylic. The samples were tested for shear bond strength in Universal testing machine . Load was applied at cross head speed of 0.5mm per minute until fracture occured .The basic values of shear bond strength in MegaPascal were obtained with the help of computer attached to the testing machine. Data obtained were subjected to statistical analysis. Tested samples were subjected to qualitative analysis using scanning electron microscopy and interface chemistry was evaluated using energy dispersive X-ray microanalysis.

In this study the mean shear bond strength value of veneering ceramic to base metal alloy before aging (Group I) was 39.51Mpa and after aging (Group II) was 37.2 MPa. The mean shear bond strength value of porcelain

veneered zirconia sample before aging (Group III) was 28.12MPa. and after aging was 26.2 MPa.

On comparison between the mean shear bond strength obtained from porcelain veneered base metal alloy samples before aging (Group I)and after aging (Group II), the shear bond strength value was found to be decreasing after aging and the difference was found to be statistically insignificant (P=0.134). On comparison between the mean shear bond strength obtained from porcelain veneered zirconia samples before aging (GroupIII) and after aging (GroupIV), the shear bond strength value decreased after aging. The difference was found to be statistically significant (P=0.010).

In this study, porcelain veneered base metal alloy group before aging (GroupI) and porcelain veneered zirconia group before aging (GroupIII) showed a statistically significant difference in shear bond strength (P=0.000). The result revealed a higher bond strength value of porcelain veneered base metal alloy group before aging than porcelain veneered zirconia group before aging.

Porcelain veneered base metal alloy group after aging (Group II) and porcelain veneered zirconia group after aging (Group IV) showed a statistically significant difference in shear bond strength (P=0.000). The result revealed higher bond strength value of porcelain veneered base metal alloy group after aging than porcelain veneered zirconia group after aging.
The highest shear bond strength value was obtained in porcelain veneered base metal alloy before aging group (Mean value -39.51MPa) followed by porcelain veneered base metal alloy after aging group (Mean value – 37.2MPa), porcelain veneered zirconia before aging group (Mean value – 28.12MPa) and porcelain veneered zirconia after aging group (Mean value – 26.2MPa).(Group I > Group II > Group II > Group IV)

A study done by Bu-Kyung Choi et al (2009) to evaluate the shear bond strength of veneering ceramic to base metal group was found as 35.87±4.23 Mpa⁷. Daniel M. Schweitzer et al (2005) reported the mean shear bond strength of porcelain fused to base metal alloy as 30.98MPa. . Al- Dohan et al (2004) reported the shear bond strength of porcelain- fused- to - metal as 30.16±5.89 MPa. Haralambos Petridis et al(1999) reported the mean shear bond strength value for porcelain fused to metal as 29.66MPa. J.L.Drummond et al (1984) reported the shear bond strength of veneering ceramic to nonprecious alloy as 31.83±3.65MPa. The bond strength measurement of metal ceramic system was standardized by the International Organization of Standardization through the Schwickerath crack initiation test (three point bending test), and the mean debonding strength / crack initiation strength should be greater than 25MPa to meet the ISO requirement.^{7,11} In this study, the mean shear bond strength of porcelain veneered base metal alloy before aging group (Mean value - 39.51 MPa) was in favour of ISO requirements and previous studies.

Haralambos Petridis et al (1999) reported the mean shear bond strength of porcelain –fused-to- metal after wet storage and thermocycling as 22.91MPa . J L. Drummond et al (1984) reported the shear bond strength of non-precious alloy after 4months of aging as 25.07 ± 5.23 MPa , after 12 months of aging as 25.01 ± 7.06 MPa. In this study, the mean shear bond strength obtained from the porcelain veneered base metal alloy samples after aging (Mean value – 37.2 MPa) was in agreement with previous studies.

Bu-Kyung Choi et al (2009) evaluated the shear bond strength of veneering ceramic to zirconia substructure and the value was found as 25.43 ± 3.12 MPa. Petra C Guess et al (2008) reported the shear bond strength of porcelain veneered zirconia as 27.9 ± 4.79 MPa. Hamid M . Ashkanani et al (2008) reported the shear bond strength of porcelain veneered zirconia as 42.45 ± 12.63 MPa. Al- Dohan et al (2004) reported the shear bond strength of porcelain veneered zirconia as 27.90 ± 4.79 MPa.. In this study , the mean shear bond strength value obtained from porcelain veneered zirconia samples before aging (Mean value – 28.12 MPa) was in agreement with previous study results. However, unlike in Al-Dohan's study, this study results indicated a significant difference in mean shear bond strength values between metal groups and zirconia groups. This difference in findings could be attributed to many factors , such as study design, methodology, skill and experience of the operator, and different properties of different materials.

M. Erhan Comlekoglu et al (2008) evaluated the shear bond strength of porcelain veneered zirconia after aging and reported as 25.4 ± 4.5 MPa. R. Morena et al (1986) studied about the fatigue of dental ceramics in a simulated oral environment and found the mean dynamic fatigue result for feldspathic porcelain as 44MPa . In this study, the mean shear bond strength obtained from porcelain veneered zirconia samples after aging (Mean value – 26.2MPa) was in favour of previous studies.

The results of this study showed that aging had an influence on shear bond strength. The shear bond strength of porcelain veneered base metal alloy and porcelain veneered zirconia was found to be decreasing after aging. The strength degradation of ceramic in aqueous environment was believed to be caused by a stress- corrosion process involving the stable growth of small, preexisting flaws.

The mode of failure of samples were examined using scanning electron microscopy under 30x and 250x magnification. Interface chemistry was evaluated using energy dispersive X-ray microanalysis. Group I samples under 30x magnification revealed a combination of predominantly cohesive failure of veneering ceramic and cohesive failure of metal oxide. 250x magnification of fractured core surface showed numerous pores within the veneering porcelain and in metal oxide layer. 30x magnification of fractured veneer surface of same group revealed the presence of metal oxide and the ceramic material. 250x magnification of fractured

veneer surface revealed numerous pores within the ceramic and also in the metal oxide. Surface chemistry of the fractured core surface explained the elements seen on the surface of fractured core and revealed the presence of silica, alumina, sodium, potassium, chromium, nickel, oxygen and carbon . The total count of silica was found to be higher indicating predominantly cohesive failure of veneering ceramic. Surface chemistry of fractured veneer surface revealed the presence of silica, alumina, sodium, potassium, chromium, nickel, oxygen and carbon. High silica content indicated predominantly cohesive failure of veneering ceramic.

Group II samples under 30x magnification showed a cohesive failure of veneering ceramic. 250x magnification of fractured core surface showed small pores on the veneering ceramic layer over the base metal alloy surface. 30x and 250x magnification of fractured veneer surface of the same group showed numerous pores within the veneering ceramic. Surface chemistry of the fractured core surface revealed the presence of silica, alumina, sodium, potassium, oxygen and carbon. The elements seen over the fractured core surface indicated cohesive failure of veneering ceramic. Surface chemistry of fractured veneer surface revealed the presence of silica, alumina, sodium, potassium, oxygen and carbon. Elements which were presented over the fractured veneer surface indicated cohesive failure of veneering ceramic.

Group III samples under 30x magnification revealed a mixed cohesive and adhesive failure of veneering ceramic, predominantly cohesive failure of veneering ceramic exposing zirconia core in some areas. 250x magnification of fractured core surface showed small pores within the veneering ceramic. 30x and 250x magnification of fractured veneering ceramic surface of same group revealed numerous pores within the veneering ceramic. Surface chemistry of the fractured core surface explained the elements seen on the surface of fractured core and revealed the presence of silica, alumina, sodium, carbon and zirconia . The elements seen over the potassium, oxygen, fractured core surface indicated mixed cohesive and adhesive failure of veneering ceramic exposing some areas of zirconia core. High content of silica over the fractured core surface indicated predominantly cohesive failure of veneering ceramic. Surface chemistry of fractured veneer surface revealed the presence of silica, alumina, sodium, potassium, oxygen, carbon and titanium... Elements which were presented over the fractured veneer surface indicated predominantly cohesive failure of veneering ceramic.

Group IV samples under 30x magnification revealed a mixed cohesive and adhesive failure of veneering ceramic, predominantly cohesive failure of veneering ceramic exposing zirconia core in some areas. 250x magnification of fractured core surface showed small pores within the veneering ceramic. 30x and 250x magnification of fractured veneer surface showed numerous pores within the veneering surface. Surface chemistry of the fractured core

surface explained the elements seen on the surface of fractured core and revealed the presence of silica, alumina, sodium, potassium, oxygen, carbon and zirconia . The elements seen over the fractured core surface indicated mixed cohesive and adhesive failure of veneering ceramic exposing some areas of zirconia core. High content of silica over the fractured core surface indicated predominantly cohesive failure of veneering ceramic. Surface chemistry of fractured veneer surface revealed the presence of silica, alumina, sodium, potassium, oxygen , carbon and calcium. Elements which were presented over the fractured veneer surface indicated predominantly cohesive failure of predominantly cohesive failure of predominantly cohesive failure indicated predominantly cohesive failure indicated predominantly cohesive failure of silica over the fractured veneer surface indicated predominantly cohesive failure of predomin

The limitations of this study were that the design of the specimens did not replicate the clinical situation and also a static test was performed without doing thermocycling procedures as in actual oral environment, where there would be repeated changes of temperature and pH. Hence, specimens replicating clinical situations and tested under dynamic load conditions after thermocycling procedures should be included in the subsequent studies.

As the veneering ceramic material is weak compared to the high strength core material, the veneering ceramic is prone to fail at low loads. Thus all tested samples fractured as predominantly cohesive failure within the veneering ceramic. This type of failure mode indicated a sufficient interfacial bond between the core and veneer material. The cohesive failure of veneering ceramic strongly suggests high residual stresses within the veneer layer. This

may be related to the varying thermal diffusivity of core and veneer material. This cooling rate difference may lead to different stress states in the two systems. The effect of coefficient of thermal expansion and the highly deleterious impact on core and veneer ceramics caused by residual stresses has been frequently discussed in the dental literature.⁴⁸

Based on the shear bond strength results of the present study the interceramic bond between zirconia core and veneering ceramics required considerable refinements in order to match the values set by the metal ceramic gold standard. Since the bond strength of the interface was higher than the cohesive strength of the veneering ceramic, it was concluded that the veneering ceramic was the weakest link. Improving the zirconia core-veneer bond strength and the strength of the veneering ceramic may reduce the failure and is paramount to the longevity of the restorations.

CONCLUSION

The following conclusions were drawn from the data obtained in the present in vitro study which was conducted to evaluate and compare the shear bond strength of veneering porcelain to base metal alloy and zirconia substructures before and after aging.

- The mean shear bond strength obtained from the basic values of shear bond strength of veneering porcelain to base metal alloy substructure before aging (Group I) was found to be 39.51MPa.
- 2. The mean shear bond strength obtained from the basic values of shear bond strength of veneering porcelain to base metal alloy substructure after aging (Group II) was found to be 37.2MPa.
- The mean shear bond strength obtained from the basic values of shear bond strength of veneering porcelain to zirconia substructure before aging (Group III) was found to be 28.12MPa.
- The mean shear bond strength obtained from the basic values of shear bond strength of veneering porcelain to zirconia substructure after aging (Group IV) was found to be 26.2MPa.
- On comparison between the mean shear bond strength obtained from porcelain veneered base metal alloy samples before aging(Group I -Mean value 39.51MPa) and after aging (Group II- Mean value

37.2 MPa), the mean shear bond strength value was found to be decreasing after aging and the difference was found to be statistically insignificant (P=0.134).

- 6. On comparison between the mean shear bond strength obtained from porcelain veneered zirconia samples before aging (Group III Mean value 28.12MPa) and after aging (Group IV Mean value 26.2MPa), the mean shear bond strength value was found to be decreasing after aging and the difference was found to be statistically significant (P=0.010)
- 7. On comparison between the mean shear bond strength obtained from porcelain veneered base metal alloy samples before aging (Group I Mean value 39.51MPa) and porcelain veneered zirconia samples before aging (Group III Mean value 28.12MPa), the mean shear bond strength of porcelain veneered base metal alloy samples before aging was found to be higher than the mean shear bond strength of porcelain veneered zirconia samples before aging and the difference was found to be statistically significant (P= 0.000).
 - 8. On comparison between the mean shear bond strength obtained from porcelain veneered base metal alloy samples after aging (Group II Mean value 37.2MPa) and porcelain veneered zirconia samples after aging (Group IV Mean value 26.2MPa), the mean shear bond strength of porcelain veneered base metal alloy samples after aging was found to be higher than the mean shear bond strength of porcelain veneered

zirconia samples after aging and the difference was found to be statistically significant (P=0.000).

9. The highest shear bond strength value was obtained in porcelain veneered base metal alloy before aging group (Group I- Mean value 39.51MPa) followed by porcelain veneered base metal alloy after aging group (Group II- Mean value 37.2MPa), porcelain veneered zirconia before aging group (Group III- Mean value 28.12MPa) and porcelain veneered zirconia after aging group (Group IV- Mean value 26.2 MPa)

 $Group \ I > \ Group \ II > \ Group \ II > \ Group \ IV$

10. To evaluate the mode of failure, the interfaces of fractured core surface and fractured veneer surface were examined under Scanning Electron Microscopy under 30x and 250x magnification. Interface chemistry was evaluated using Energy Dispersive X-ray microanalysis (EDX analysis).

Group I – SEM analysis under 30x and 250x magnification revealed cohesive failure of metal ceramic bonding, predominantly failure within the veneering ceramic. EDX analysis explained high content of silica on fractured core surface and fractured veneer surface indicated predominantly cohesive failure of veneering ceramic.

Group II – SEM analysis under 30x and 250x magnification revealed cohesive failure of veneering ceramic. EDX analysis explained high

content of silica on fractured core surface and fractured veneer surface indicated cohesive failure of veneering ceramic.

Group III – SEM analysis under 30x and 250x magnification revealed mixed cohesive and adhesive failure of veneering ceramic, predominantly cohesive failure of veneering ceramic. EDX analysis explained high content of silica on fractured core surface and fractured veneer surface indicated predominantly cohesive failure of veneering ceramic.

Group IV – SEM analysis under 30x and 250x magnification revealed mixed cohesive and adhesive failure of veneering ceramic, predominantly cohesive failure of veneering ceramic. EDX analysis explained high content of silica on fractured core surface and fractured veneer surface indicated predominantly cohesive failure of veneering ceramic.

The quantitative results of this study were in correlation with the qualitative results of the study.

SUMMARY

The present in vitro study was conducted to evaluate and compare the shear bond strength of veneering porcelain to base metal alloy and zirconia substructures before and after aging.

Twenty base metal alloy cores and twenty zirconia cores of dimension 9mm length x 4mm width x 4mm height were prepared and veneered with corresponding veneering porcelain to the dimension of 3mm length x 4mm width x 4mm height. Ten porcelain veneered base metal alloy samples were used to determine the shear bond strength before aging (Group I). Ten porcelain veneered base metal alloy samples were used to determine the shear bond strength after aging (Group II). Ten porcelain veneered zirconia samples were used to determine the shear bond strength before aging (Group III). Ten porcelain veneered zirconia samples were used to determine the shear swere used to determine the shear bond strength before aging (Group III). Ten porcelain veneered zirconia samples were used to determine the shear bond strength after aging (Group IV).

All samples were tested for shear bond strength in Universal testing machine. The basic values of shear bond strength of all test samples in four groups were tabulated. The mean shear bond strength for each group was calculated and tabulated. The results were subjected for statistical analysis.

The data obtained in the present study revealed the highest shear bond strength value of porcelain veneered base metal alloy samples before aging (Group I- Mean value 39.51MPa) followed by porcelain veneered base metal alloy samples after aging (Group II- Mean value 37.2MPa), porcelain veneered zirconia samples before aging (Group III- Mean value 28.12MPa) and porcelain veneered zirconia samples after aging (Group IV- Mean value 26.2 MPa). Group I > Group II > Group III > Group IV

On comparison between mean shear bond strength of Group I and Group II, the mean shear bond strength was found to be decreasing after aging and the difference was found to be statistically insignificant. On comparison between mean shear bond strength of Group III and Group IV, the mean shear bond strength was found to be decreasing after aging and the difference was found to be statistically significant.

On comparison between the mean shear bond strength of Group II and Group IV, the mean shear bond strength of Group II was found to be higher than the mean shear bond strength of Group IV.

It was evidenced that aging had an influence on the shear bond strength of porcelain veneered base metal alloy as well as porcelain veneered zirconia samples. Aging facilitate stress corrosion of ceramic materials, resulting in slow crack growth and finally leading to failure of ceramic materials.

On comparison between the mean shear bond strength of Group I and Group III, the mean shear bond strength of Group I was found to be higher than the mean shear bond strength of Group III. The results showed higher shear bond strength of base metal alloy group which were in agreement with the ISO requirements and previous studies.

Tested samples were qualitatively analyzed using scanning electron microscopy and interface chemistry was analyzed using energy dispersive x-ray microanalysis. Group I samples revealed cohesive failure of metal ceramic bonding, predominantly failure within the veneering ceramic. Group II samples revealed cohesive failure of veneering ceramic. Group III samples revealed mixed cohesive and adhesive failure of veneering ceramic, predominantly cohesive failure of veneering ceramic. Group IV samples revealed mixed cohesive and adhesive failure of veneering ceramic, predominantly cohesive failure of veneering ceramic. The quantitative results of this study were in correlation with the qualitative results of the study.

In this study, the shear bond strength of porcelain veneered base metal alloy was found to be statistically significantly higher than the shear bond strength of porcelain veneered zirconia before and after aging. SEM analysis revealed predominantly cohesive failure of veneering ceramic. Since the bond strength of the interface was higher than the cohesive strength of ceramic , it was concluded that the veneering ceramic was the weakest link. Based on the shear bond strength results of the present study the interceramic bond between zirconia core and veneering ceramic requires considerable refinements to match the values set by the porcelain veneered base metal alloy gold standard. Improving the zirconia core-veneer bond strength and the strength of the veneering porcelain may reduce the failure and is paramount to the longevity of the restoration.

The metal ceramic restorations have been extensively used in dentistry for the past five decades. The problem of metal discoloration at the margins and allergic reactions to metals have led to the development of metal free ceramic restorations for superior esthetics and biocompatibility in fixed prosthodontics. Considering all ceramic materials available in dental health care, zirconia offers the best mechanical properties and has the potential to be applied as an alternative support material to alloys for the fabrication of fixed dental prosthesis. The problem involving early fracture of the veneer porcelain of zirconia supported restorations and the unclear effect of the low temperature degradation have led clinicians to question the total substitutution of alloys through zirconia based dental restorations. As zirconia has demonstrated good mechanical and biological performance, future technology is attempting to improve esthetics and minimize veneer fracture, aiming to create confidence in dental community towards this all ceramic system. Zirconia is being widely applied in dentistry starting from oral implant fabrication to the manufacturing of dental crown and bridge work. Zirconia and zirconia - supported ceramics are worthy of being further evaluated particularly with improved production methods.

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