

*Adhesion of veneering porcelain to alumina ceramic
as determined by the strain energy release rate*

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Abstract:

Purpose: *To study the interface adhesion between alumina and porcelain using a fracture-mechanics approach, and to determine the basis of this adhesion from observations under the scanning electron microscope of the fracture path after loading the specimens.*

Methods: *The specimens used for the study were 5 bilayered rectangular blocks of Alumina (VITA In-Ceram 2000 AL Cubes) bonded to veneering porcelain (VITA VM 7). A notch was prepared using a diamond saw to the interface of the bilayered specimen. The specimens then underwent a four-point bending interfacial delaminating test on a universal testing machine. The strain energy release rate (G , J/m^2) was calculated from the critical load to induce stable crack extension along the interface during loading of the pre-cracked specimens. The interface of these specimens was then investigated under the scanning electron microscope to identify the crack path and determine the behaviour of the adhesion between alumina and porcelain.*

Results: *The mean G value of the 4 specimens was calculated as $12.09 \pm 3.02 J/m^2$. It was observed under the scanning electron microscope that the 4 specimens exhibited irregular interface cracking behaviour. In the majority of specimens, cracks started from the tip of the pre-crack and extended along the alumina and porcelain interface in an almost straight line. However, some specimens demonstrated a wave like fracture behaviour between the porcelain and the alumina.*

Conclusion: *Clinical failures of alumina restorations are reported to occur due to fracture of the alumina core and/or chipping of the veneering ceramic. In this study, it was found that complete delamination occurred from the pre-crack in most of the specimens with low G values ($12.09 \pm 3.02 J/m^2$) indicating a relatively weak adhesion between alumina and veneering porcelain as compared to gold and porcelain ($72.7 \pm 10 J/m^2$). Failure primarily occurred near the interface; however some specimens exhibited fracture of the alumina with veneering porcelain still bonded to the core.*

Introduction:

Dental ceramics were first introduced in dentistry in the late 1700s. Porcelain jacket crowns were developed in the early 1900s and they consisted of feldspathic or aluminous porcelain baked on a thin platinum foil and can be considered the ancestors of all-ceramic crowns. The end of 20th century saw the introduction of several all-ceramic dental restorations and since then new materials for all-ceramic restorations are introduced every year and attest to the popularity of ceramics in dentistry (Craig 2006)

Metal ceramic restorations are the most popular materials for crowns and fixed dental prostheses. The reason for this is, high strength and long term survival in the oral cavity, but the metallic background can present an aesthetic problem in some clinical situations. All-ceramic systems were developed as a response to the increasing concerns in dentistry for superior aesthetics and biocompatibility. However, if the metallic understructure is eliminated, the strength of the restoration becomes an important issue.

Several processing techniques are available for fabricating all-ceramic restorations, for example sintering, heat pressing, slip casting and machining. In an effort to improve strength and increase the versatility of all-ceramic restorations, several core materials have been developed, for example, IPS Empress, In-Ceram Alumina, In-Ceram Zirconia, and Procera alumina. These new materials and techniques have widened the range of applications of all-ceramic materials.

Reinforced all-ceramic crowns typically consist of a high strength non-porcelain ceramic core material, laminated with dentin and incisal porcelain. However, successful performance and reliability of veneered ceramic prostheses may be limited by mechanical integrity and adhesion of the veneering porcelain to the ceramic substrates. The thermal expansion properties of core materials and veneering porcelains should match to reduce residual stresses and ensure good adhesion and thereby achieve a durable bond between the core and the veneering porcelain.

According to Criag (2002), ceramics are strong in compression but at the same time they can be inherently brittle and weak when placed under tensile and torsional stresses.

There are several factors that are associated with the stress state created in dental ceramic restorations including;

- (1) Thickness of ceramic layers
- (2) Mechanical properties of each ceramic
- (3) Elastic modulus of the supporting substrate material
- (4) Direction, magnitude and frequency of applied load
- (5) Residual stresses induced by processing
- (6) Size and location of occlusal contact areas
- (7) Restoration-cement interfacial defects
- (8) Environmental effects

In addition to the above, Kelly et al in 1996 suggested that in the case of all-ceramic fixed partial dental prostheses the cause of fracture between veneering porcelain and ceramic core could be due to intra-ceramic defects and/or lack of adequate framework support.

The mechanism of fracture of all-ceramic restorations and metal ceramic restorations differs significantly due to the differences in their internal structures, mechanism of their bonding and the brittle nature of the ceramic. There are larger inter-atomic forces in all-ceramic restorations associated with covalent and ionic bonds, which can lead to greater resistance to plastic deformation as compared to metal components of metal-ceramic restorations.

In order to achieve the best clinical outcome, measurements of the adhesion between various all-ceramic restorations are needed for selection of the appropriate core-veneer combination in day to day clinical decision making. There are reports on the adhesion between the ceramics and metal substrates using interfacial fracture mechanics (Suansuwan and Swain 1999, Yamada et al 2004, Tholey et al 2007); however there is a dearth of literature on such evaluations between all-ceramic restoration cores and veneering porcelain.

There have been a few other studies conducted that have evaluated the clinical adhesion of porcelain to alumina. Odman and Anderson 2001 in a prospective multicenter study reported a cumulative survival rate of 93.5 % after an observation period of 10 years for alumina framework all-ceramic crowns. Out of 50 patients enrolled in the study, 74 % of the crowns were placed in the posterior region. The authors reported cumulative success and survival rates of more than 90 %. However, the study had a small sample size and there was a loss of follow up of 18 % mainly from the group of patients who had crowns placed in the posterior region.

In another study, by Fradeani et al 2005 with a comparatively bigger sample size of 205 patients, clinical performance of Procera all-ceramic crowns over a period of 5 years was evaluated at three different dental practices. 155 crowns were placed in the posterior region and the 5 year survival rate was 95 %. Out of the four failed molar crowns, two crowns had delamination of the veneering porcelain and the other two failed crowns had fracture of both veneering porcelain and alumina coping. It was suggested that in order to avoid risk of ceramic delamination especially in the posterior region, a double scanning procedure should be preferred. This would better support the layering material on the core and thus reduce the possibility of interproximal breakage. The double scan procedure involves two scans, first scan is done for the die and the second scan involves scanning of cut back of the wax pattern with the desired veneering porcelain thickness. The data of these two scans is merged and sent electronically to the milling facility for the fabrication of alumina/zirconia coping.

The shortcomings of this study were that there was no calibration between the three clinicians and the dental technicians in those practices, and the mean follow up of this study was only 23.52 months.

Many limitations have been observed in the reporting of the clinical studies regarding mechanical failure of all-ceramic restorations. Patient selection and technique sensitivity are found to be more critical in all ceramic restorations versus metal ceramic restorations. Most of the studies on the outcomes of all ceramic restorations have very strict inclusion and exclusion criteria and ambiguous definitions of survival and failures, indicating a lack of standardization of the terminology in reporting treatment outcomes of all ceramic restorations.

Also, there is very limited data on the clinical performance of the success of all-ceramic fixed partial dental prostheses. The use of all ceramic restorations replacing missing teeth with tooth borne fixed dental prostheses is mainly limited to anterior and premolar teeth, and may require the use of controversial and technique sensitive clinical procedure such as adhesive cementation. (Riagrodski 2004)

Literature review:

The most commonly used methods to measure the bonds strength between two dissimilar materials are shear bond strength tests, flexure mode tests (3-4 point bending tests) and micro tensile bond strength test.

Data available in the scientific literature present a range of bond strengths between the different all-ceramic materials available.

Al Dohan et al 2004 in an in-vitro study evaluated the strength of the substructure and veneering porcelain interface in 4 all ceramic systems (IPS Empress 2, Procera AllCeram, Procera AllZircon and DC-Zircon) with metal ceramic combination as control group.

12 specimens from each all-ceramic system and metal ceramic system were made from 1 master die. All specimens were subjected to shear force on a universal testing machine. The failed specimens were examined microscopically to classify the mode of failure as cohesive in the core, cohesive in veneer or adhesive at the interface.

It was seen that complete delamination did not occur between the compatible ceramic core and veneering materials. Failure primarily occurred near the interface with residual veneering porcelain remaining on the core indicating a cohesive type of fracture.

Bond strength of 3 of the tested all ceramic systems (IPS Empress 2, Procera AllZircon and DC Zircon) were not significantly different from the metal ceramic control group. However, the veneering porcelain of AllCeram applied to the Procera AllCeram alumina core showed a significantly weaker bond compared to other systems.

Dundar et al 2007 investigated the bond strength for core and veneering ceramics of four all-ceramic systems using two different bond strength testing methodologies (shear bond strength test and micro tensile bond strength test) and by scanning electron microscopy. Significant differences were found in the shear bond strengths of the different all ceramic specimens with lithium disilicate based ceramic system being the highest (41 ± 8 MPa) and glass-infiltrated alumina (26 ± 4 MPa) and leucite reinforced ceramic(23 ± 3 MPa) being the lowest.

The values for the microtensile bond strengths were higher for low leucite reinforced ceramics (15 ± 2 MPa) and even lower for glass infiltrated alumina. The authors concluded that the testing method and the type of ceramic material used influenced the bond strengths of the bilayered all-ceramic system. Cohesive failures and considerable scatter was observed in the shear bond strength measurements with higher standard deviations making the testing methodology not reliable to quantify the bond strength of bilayered ceramics.

Wakabayashi and Anusavice (2000) investigated the influence of alumina core material thickness and the supporting substrate stiffness on the strength and failure mode of bilayered ceramics discs.

140 densely sintered alumina core discs were randomly divided into 5 groups.

Each group consisted of 35 alumina core disc of 18 mm diameter x 2 mm thickness. After storage in water at 37° C for 96 hours each bonded discs was loaded in a universal testing instrument. Optical scanning electron microscopic examinations were performed for detection of cracks within each surface. Although the crack initiation sites shifted from veneer to the core as the core/veneer thickness ratio increased, no shift occurred as the elastic modulus of the substrate increased.

The results of this study suggested that the thickness of the core to the veneer is the dominant factor that controls the failure initiation site in bilayered ceramics with a relatively strong core and weak ceramic veneer.

However in another study, Webber et al 2003 concluded that changing the veneer porcelain thickness did not alter the load to fracture of the crown specimen.

They investigated the effect of different thickness of veneer porcelain on the compressive load at fracture of Procera AllCeram crowns. 60 brass dies were fabricated with a crown like preparation and chamfer margin and 60 crowns were fabricated with 0.6 mm thick core and different thickness of the veneering porcelain. After storage in distilled water at 37 ° C for 24 hours, the specimens were tested in a universal testing machine.

The limitation of this study was that the suggested effect of silination for improved bonding and thus increasing the overall strength of the restorations is questionable due to the high alumina content in the core.

Dundar et al 2005 evaluated the shear bond strength of alumina and leucite reinforced ceramics to their corresponding veneering porcelain. Test specimens were stored in dry and thermocycled conditions and tested in a universal testing machine. The authors did not find statistically significant difference in the shear bond strengths measurements of alumina specimens kept in dry and thermocycled conditions. The core-veneer interface failure was mainly adhesive in nature for alumina and cohesive for leucite reinforced ceramics. It was suggested that bilayered specimens exhibited complex failure modes and the information on the best combination of the reinforced core and veneering ceramic could assist the clinician to predict possible chipping at the core-veneer interface.

Van Noort et al 1989 critiqued the in-vitro bond strength measurement tests and analysed the variations in bond strength measurements in shear and tension using finite element analysis. They emphasized that the concept of average stress for the measurement of bond strength cannot be applied in practice as there exists a very complex stress situation at the interface. Various studies measuring the parameters using different experimental procedures usually fail to report on the relative elastic moduli, loading geometry, shape and size of adherent. Thus the

absolute value of the stress at the interface needs to be studied when a direct comparison with the clinical situation is to be undertaken.

Dental ceramics are susceptible to fracture under low impact stress at defects that are either inherent to the material and/or introduced during processing (Kelly et al 1996)

Since it is impossible to predetermine the size, location and distribution of the most critical defects, the strength of a number of identical standard ceramic specimens cannot be determined reliably from average fracture strengths.

As a result, it is important that laboratory load to failure mechanical tests simulate the failure mechanisms normally encountered in the clinical situation.

Suansuwan and Swain 1999 evaluated the bonding characteristics of PFM systems by determining the strain energy release rate associated with interface fracture of porcelain and metals.

20 rectangular plates made up of 3 metal alloys (Gold, Palladium and Nickel chromium) and commercially pure titanium metal plates were veneered with comparable thickness of veneering porcelain. Porcelain side of the specimen was notched to the interface with a thin diamond saw and a small pre-crack was initiated at the metal porcelain interface.

Samples were subjected to a limited number of load-unload cycles under 4 point bending and the associated stable crack extension were recorded and examined under scanning electron microscopy.

Strain energy released rates were then calculated and it was observed that the mean strain energy release rates were highest for gold, i.e, greatest adhesion and least for titanium (poor adhesion).

The study has several advantages in terms of studying the bond strength (tensile and shear) between two dissimilar materials;

1. Test was found to be highly reproducible and simple to perform

2. More intrinsic estimation of interface crack resistance properties is possible.
3. As there was a pre-crack made before testing the specimen, there was an observed stability of crack extension along the interface instead of the crack propagating in an unstable manner within or through the porcelain.

The bimaterial interfacial bend test was introduced by Charalambides et al 1989 which allows direct measurement of the interface fracture energy as a sharp crack grows from the notch upon reaching the interface will either deflect or penetrate the interface upon controlled loading and unloading cycles.

Cazzato and Faber 1997 examined the glass alumina interface and measured the interface fracture energy by loading specimens in such a four point bending test. All the specimens were notched with a diamond blade and beams were positioned with the alumina surface in tension and the glass surface in compression. Load and displacement data were recorded and the interface examined under a microscope.

The experiment showed that the higher the glassy phase content in the alumina and the lower moisture content of the environment increased the measured toughness of the interface.

Carrier and Kelly 1995 evaluated the failure behaviour of InCeram alumina structures with or without thin layers of excess infiltration glass left on the core. Two groups of specimens were prepared in the shape of a disk and a crown and were subsequently loaded to biaxial flexure test. It was seen that the excess infiltration glass on the core surface did not affect the failure of specimens in the shape of the crowns. Failure loads were significantly higher for this group. Microscopic evaluation showed core-veneer interfaces with less porosity in the presence of excess of the glass infiltrant.

This pilot study aimed to evaluate the interface between alumina and porcelain using a fracture-mechanics approach, and to determine the behaviour of this adhesion after loading the specimens under the scanning electron microscope.

Materials and methods:

Determination of adhesion between alumina core and veneering porcelain

The approach to study the interface of two dissimilar materials was used by Suansuwan and Swain 1999 to study the adhesion between metals and porcelain. In this study the strain energy release rate (G value) was calculated as described by Charalambides et al (1989).

Specimen preparation:

Five bilayered rectangular plates (8 x 33 x 2 mm) were prepared from alumina (VITA In-Ceram 2000 AL Cubes) and bonded to veneering porcelain (VITA VM 7).

The alumina core ceramic (VITA In-Ceram 2000 AL Cubes) were cut from the blocks using a diamond grinding tool sufficiently oversize and then sintered at 1530 C according to the manufactures protocol (8x 33 mm and thickness 1mm) and subsequently veneering porcelain (VITA VM 7) was applied in 2- 3 layers to achieve a total thickness of the test specimen of approximately 2 mm.

The firing protocol used for the veneering porcelain was according the manufacturers recommendations.

After firing the specimens were mounted on a metal jig and placed perpendicular to a rotary polishing machine to achieve smooth and even surface of veneering porcelain.

The specimens were then evaluated for any visible cracks or deformities and defective specimens were excluded from the study.

A notch across the entire width of the veneering porcelain of all the specimens was prepared using a low speed water cooled rotary diamond saw using a special jig. The notched specimens were then tested on a universal testing machine placed on a four point bending jig (Figure 1). The distance between the inner and outer rollers of the four-point bending jig was 5 mm on each side.

The specimens were subjected to load at a cross head speed of 0.1mm/min. In addition 2 or 3 unload-reload cycles were conducted to determine whether crack extension occurred.

The specimens were loaded until the crack reached the inner rollers. The load (P) measured in Newton's and the cross head displacement data in millimetres was recorded on the computer for the calculation of the G value (Equation 1).

The testing method for the specimen is described diagrammatically below (Figure 1);

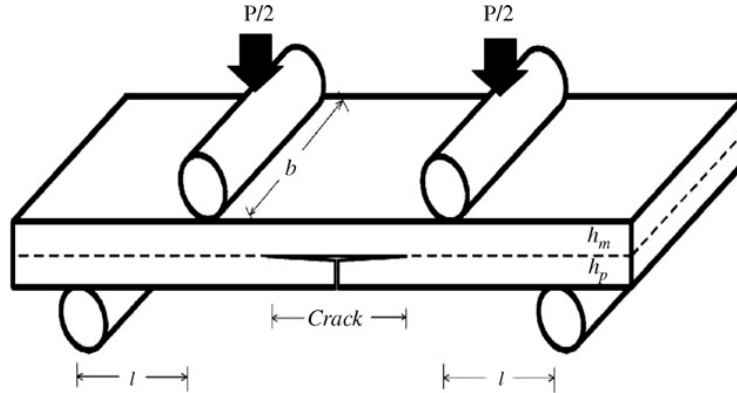


Fig 1: Diagrammatic representation of the testing specimen

The strain energy release rate (G) was calculated by

$$G = \frac{\eta P^2 l^2 (1 - \nu_m^2)}{E_m b^2 h^3}$$

Equation 1

The non dimensional parameter η was calculated by

$$\eta = \left(\frac{3}{2}\right) \left[\frac{1}{(h_m/h)^3} - \frac{\lambda}{\{(h_p/h)^3 + \lambda(h_m/h)^3 + 3\lambda(h_p h_m/h^2)[(h_p/h) - (\lambda h_m/h)]\}^{-1}} \right]$$

Equation 2

and λ was calculated by

$$\lambda = \frac{E_m(1 - \nu_p^2)}{E_p(1 - \nu_m^2)}$$

Equation 3

Where P is the load necessary propagate the crack,
 l distance between inner and outer load line (rollers) on the same side or the moment of the arm,
 ν_m and E_m are Poisson's ratio and elastic modulus of alumina core substructure respectively,
 b and h are the specimen width and total thickness respectively,
 η is a non dimensional parameter,
 ν_p and E_p are Poisson's ratio and elastic modulus of porcelain and
 h_p and h_m are thickness of porcelain and alumina respectively.

After loading the specimens on the universal testing machine the specimens were coated prior to being examined under the scanning electron microscopy.

Results:

The load displacement curve shown in Fig 2 demonstrates a typical load-unload cycle of the specimens tested. The region of the curve representing the plateau-like stable crack extension was used in order to determine the average load for each specimen. A clear feature of the results were that despite the plateau load varying from 38 to 50 N when allowance for the relative thicknesses of the porcelain and alumina is considered, the resultant estimate of the strain energy release rate from the above equations were very consistent. For example, in Specimen 1 with thickness of 2.03mm and plateau load of 38 N, strain energy release rate is 9.35 J/ m² and Specimen 2 with thickness of 1.60 mm and plateau load of 50 N, the strain energy release rate is 10.98 J/ m²; which are fairly consistent.

As illustrated below, Fig 2, the graph a typical load plateau which indicates a debonding of the interface, in cases where the sharp crack either deflects or penetrates the interface depends on the relative toughness of the two adjacent materials. The response to the load was non linear and the crack was deflected along the interface.

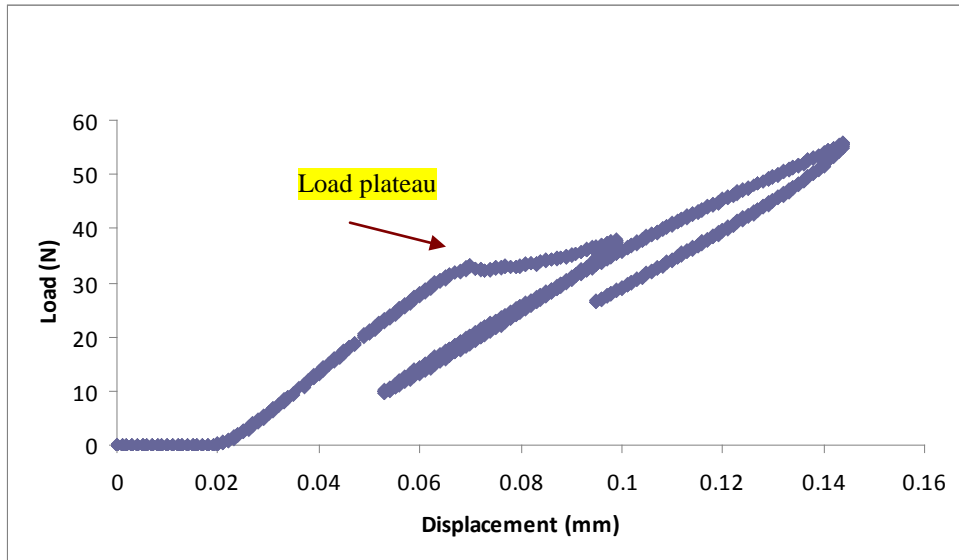


Fig 2: Load displacement curve demonstrating area of stable crack extension

Calculation of the strain energy release rate was done using the formula mentioned in the study by Suansuwan and Swain (1999). The values applied for the calculation are mentioned in the table below for all the specimens except for Specimen # 3.

Mean G value recorded was $12.09 \pm 3.2 \text{ J/m}^2$

	G value(J/m ²)	Load (N)	l (mm)	B(mm)	h(mm)
Specimen # 1	9.35	38	5	7.91	2.03
Specimen # 2	10.98	50	5	7.88	1.60
Specimen # 3	-	-	-	-	-
Specimen # 4	12.94	39	5	7.86	1.71
Specimen # 5	15.12	50	5	7.87	1.90

Please note: Specimen # 3 fractured completely as the load was increased beyond 90 N and hence the data for the calculation of the strain energy release rate (G) was not possible.

Scanning electron microscope results:

The low magnification images of the specimens under the scanning electron microscope show the veneering porcelain layer on the top with the notch and the alumina core layer below

separated by a thin white line extending on both sides of the notch. This phenomenon was seen in all the specimens indicating a typical adhesive type of the fracture running across the interface of alumina core and veneering porcelain in almost a straight line.

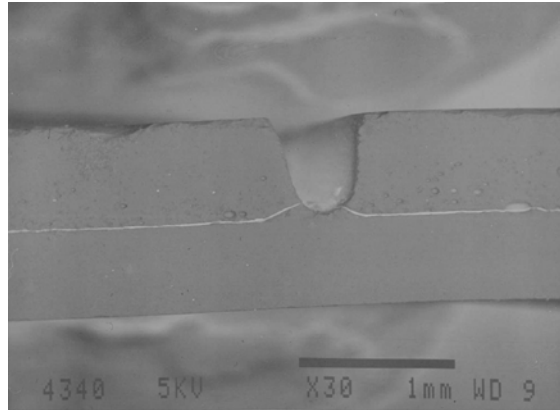


Fig 3: Scanning electron microscope image showing delamination at interface (1)

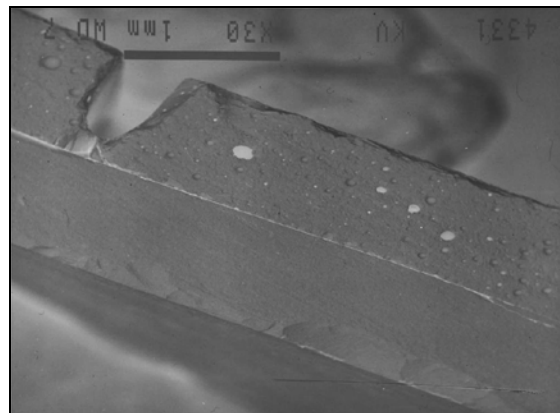


Fig 4: Scanning electron microscope image showing delamination at interface (2)

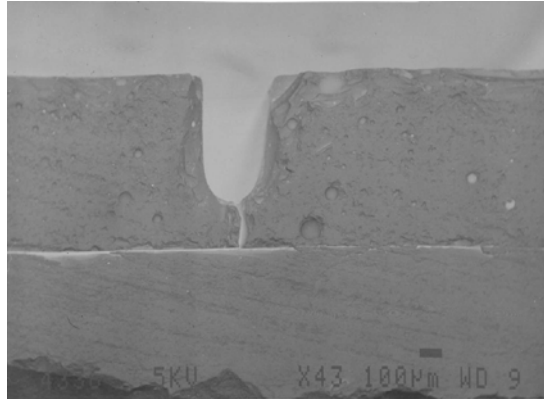


Fig 5: Scanning electron microscope image showing delamination at interface (3)

Discussion:

Clinical performance of alumina crowns has been reported in the literature with a varying range of success and survival rates. Given the brittle nature of ceramic materials, there has been a continuous effort to improve the strength and aesthetics by various means for all-ceramic restorations. One of the reasons of failure is delamination of the veneering porcelain.

There have been various attempts to study the reasons for the mechanical failures with the assumption that the interface between the ceramic core and veneering porcelain is the weakest link. On this premise, some previous studies using the protocol of pre-cracking the specimen and four point bending test for determining the adhesion between metal ceramic combinations have been conducted. This method of determining the adhesion was found to be reproducible and reliable.

Taking it further, this pilot study was done to determine the adhesion or the weakest link between the alumina core and veneering porcelain by the same fracture mechanics approach used by Swansuan and Swain 1999.

The results of the study indicated that the strain energy release rate (G) between alumina and veneering porcelain were $12.09 \pm 3.2 \text{ J/m}^2$. These figures are significantly low as compared to the gold-porcelain G values which were $72.7 \pm 10 \text{ J/m}^2$ in the study by Swansuan and Swain 1999.

The lower values obtained indicate a weak link between the alumina and veneering porcelain and demands further research to evaluate methods in improving the bond between all-ceramic restorations.

It was observed that after loading the pre-cracked specimens, the crack started below the notch and propagated along the interface. However, in cases where the notch did not reach the interface, there was a small vertical crack seen which approached the interface and followed the interface horizontally. Scanning electron microscope observations revealed that failure primarily occurred near the interface. However, in some specimens, higher magnification exhibited fracture of the alumina with veneering porcelain still bonded to the core. Interestingly, on the other side, in some of the specimens the crack diverted in the veneering porcelain indicating a cohesive type of failure as shown in the images below.

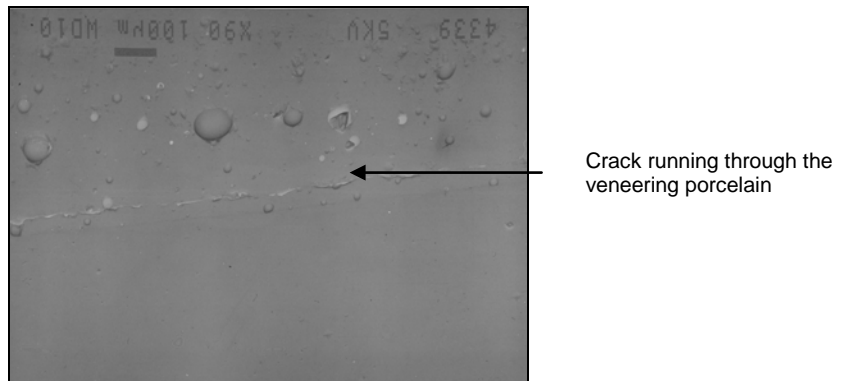


Fig 6: Scanning electron microscope image showing cohesive fracture in veneering porcelain

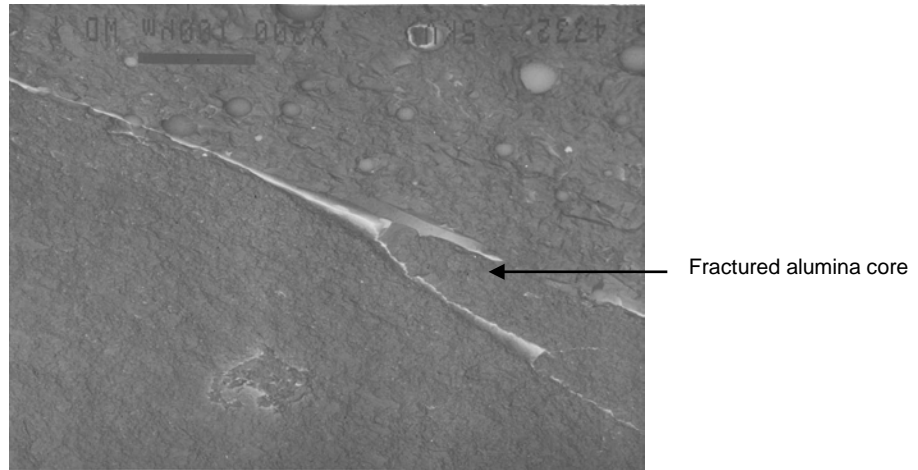


Fig 7: Scanning electron microscope image showing fracture of alumina

Finite element studies of shear bond strength tests have been conducted to study the reasons for mechanical failure of all-ceramic restorations. With the improved strength of contemporary core materials, these finite element analyses indicate that stresses in the core should not greatly influence the longevity of the restoration. According to these tests, the interface area of bilayered materials incorporates stresses of a very complex nature. In many configurations, the most critical sites are still seen in the veneering porcelain near the interface. (Jager et al 2005). Although, finite element analysis was beyond the scope of this study, it would be valuable to evaluate the specimens by this method for future studies.

In vitro studies evaluating the bond strength of all-ceramic restorations measure the critical stress at bond failure, but this does not provide information concerning the energy or the work needed to separate ceramic core from the veneering porcelain. These tests only measure the stress to initiate catastrophic failure of the experimental specimen. They are also very dependent upon the size of the specimen and show a large scatter.

There were some limitations in the present study some of which were encountered during the specimen preparation, as it was challenging to achieve a uniformly thick layer of veneering porcelain on the rectangular alumina plates. It was mainly due to the shrinkage of the porcelain

during the firing cycle. Therefore porcelain was applied in at least 2-3 layers and later ground to achieve a uniform thickness.

There were also some difficulties during the pre-cracking procedure and loading the specimens to allow the crack to propagate along the interface on either side of the notch in a stable way. Due to the rigid and brittle nature of the alumina core, it was difficult to induce a crack in a controlled way and it was found that as the load increased beyond 90 N, one of the specimens broke into several pieces.

Conclusions:

- 1) A simple fracture mechanics approach was used to evaluate the adhesion between alumina and veneering porcelain. A four point bending test that generated stable crack extension along the interface of alumina and veneering porcelain was used in order to measure the strain energy release rate. This was followed by observation of the specimens under scanning electron microscope.
- 2) The mean strain energy release rate $G = 12.09 \pm 3.2 \text{ J/m}^2$ was calculated by the load displacement curve for alumina and veneering porcelain specimens.
- 3) It was observed under the scanning electron microscope that all the specimens exhibited complete delamination along the interface in almost a straight line.
- 4) The lower G values obtained indicates a weak link between alumina and veneering porcelain and demands further research to evaluate methods in improving the bond between core and veneering ceramic of all-ceramic restorations.

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