

Shear Bond Strength of Veneering Porcelain to Different Core Materials like Zirconia, Lithium Disilicate and Nickel Chromium Alloy - An Invitro Comparative Study

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Abstract

Objective: To evaluate the shear bond strength of veneering porcelain to zirconia, Lithium disilicate and Nickel chromium alloy cores.

Materials and Methods: Rectangular samples of dimension 9x4x4mm were made in three different core materials viz. Zirconia, Lithium disilicate and Nickel chromium alloy. The bonding surface of the specimens were sandblasted. Feldspathic porcelain (cerinate) was applied on the cores and fired to form a 3mm thick veneer. The specimens were embedded in an acrylic block. These specimens were subjected to shear force in a universal testing machine. Load was applied at a cross head speed of 0.5mm per minute until the specimens fractured. The force required for fracturing was recorded. The broken specimens were examined under scanning electron microscope. Data analysis was done with factorial ANOVA.

Results: The maximum bond strength was seen between lithium disilicate core and the veneering ceramic (409.4N). The Least bond strength was between Zirconia core and the veneering ceramic (284.8N). The bond strength between Nickel chromium base metal alloy core specimens and the veneering ceramic was 310.2N. SEM images of lithium disilicate specimens showed predominantly cohesive failure. SEM of Zirconia specimens showed mixed adhesive and cohesive failures. SEM of Nickel Chromium specimens showed cohesive failure. Energy dispersive microanalysis of the Lithium disilicate, metal ceramic and zirconia specimens showed mainly oxygen and silica on the surface.

Conclusions: All ceramic restorations with lithium disilicate core and feldspathic veneer can resist clinical failure due to fracture of the veneering ceramic. Delamination of the veneering ceramic can be frequent with Zirconia core.

Scanning electron microscopic analysis showed both adhesive and cohesive failures. Lithium Disilicate specimens showed predominantly cohesive failures. Multiple cracks and many pores were seen in the zirconia specimens and the failure was both adhesive and cohesive.

Electron Dispersive microanalysis showed mainly oxygen and silica as the chemical constituents present on the fractured surface.

Keywords: Zirconia; Lithium Disilicate; Nickel Chromium Alloys; Shear Bond Test; Elemental Analysis; SEM

Introduction

Porcelain received wide acceptance as a restorative material when it was successfully combined with alloys and thereby an improvement in the mechanical properties. There after porcelain fused to metal (PFM) ruled the scene for the subsequent four decades. PFM systems intelligently combined the fracture resistance of the metal substructure with the aesthetic property of the porcelain. However, failures also have occurred with PFM fixed dental prosthesis at the rate of 4% after five years, 12% after 10 years, and 32% after 15 years [1].

As time went on, patients felt that the aesthetics was not up to the mark. PFM restorations have an inherent weakness such as the inability to transmit light, making the restorations appear dark in colour and limiting the aesthetics [2,3]. It was found that some of the alloys had questionable biocompatibility because of the components like Nickel and Beryllium. Demand for aesthetics and biocompatibility, fuelled the development of metal free restorations [4]. In an all-ceramic restoration, the ceramic material may be monolithic or it may consist of a ceramic core and a covering ceramic veneer and is known as a bi-layered, all-ceramic restoration [5]. In the bi-layered, all-ceramic restoration, the ceramic core supports the restoration and is mainly responsible for its strength.

Pressable lithium disilicate and zirconia - yttrium oxide partially stabilized tetragonal

zirconia polycrystal (Y-TZP) were introduced as core materials in layered all ceramic restorations. As such these materials did not match the aesthetic requirements of a dental restorative, however it could be compensated by veneering with feldspathic porcelains and the success story of all ceramic restorations started there. Unlike ceramics used with PFM restorations, ceramics used in fabricating all-ceramic restorations are made up of more crystalline particles. As such, the percentage of crystalline particles may range from 40% to 70%. Furthermore, the ceramic may be of polycrystalline nature. Due to the transformation toughening mechanism, Y-TZP has been shown to have superior mechanical properties compared to other all-ceramic systems [6].

The veneering material provides the morphology of the restoration, shade, translucency and aesthetics. Though not very significant, the core may also contribute to the development of the shade of the restoration. However, the veneer-core bond strength is considered to be one of the weakest links of the bi-layered all-ceramic

restorations [7,8]. They are prone to delamination and fracture [9]. The major drawback of the layered all ceramic restoration is the fracture of the veneers.

The bonding of metal and ceramic has been studied extensively and the bond strength between ceramic and the alloy is considered to be optimum at 25Mpa. The bonding mechanism between lithium disilicate and veneering porcelains, as well as zirconia and veneering porcelains has not been understood clearly. There is a wide variation in the flexural strength of the materials used in metal ceramic and all ceramic restorations. While zirconia has a flexural strength of 900-1200 MPa, Lithium disilicate has 407 MPa, Nickel Chromium alloys 620 MPa and Feldspathic porcelain 149 MPa. This should be viewed against the frequent complaint of delamination that occur in layered all ceramic restorations¹⁰. The study on comparing the different substrates viz. Lithium disilicate, Zirconia and Nickel Chromium alloy against feldspathic veneering porcelains was taken up in this context with the objective to evaluate the shear bond strength of veneering porcelain to zirconia, lithium disilicate and nickel chromium alloy cores.

Methodology

Three core materials viz. 1. Zirconia (Noritake TZR, Japan), 2. Lithium disilicate (emax, Ivoclar, Vivadent) 3. Nickel Chromium alloy (Cera Bond, Germany) and one Porcelain veneering material (Cerinate, Germany) were used to make specimens of three core veneer combinations (N = 30, n = 10/group)

Preparation of zirconia specimens

Ten wax blocks of dimension 4 × 4 × 9mm were made (Figure 1) and these wax blocks were scanned using Sirona optical scanner. Ten specimens of dimension 4 × 4 × 9mm were milled using Inlab MCXL Sirona machine (CEREC) from the zirconia blank (Noritake TZR). The bonding surface (4 × 4mm) of the specimens was sand-blasted with 110µm aluminium oxide particles at 2.5 bar pressure and were steam cleaned and air dried.

Preparation of lithium disilicate specimens

Ten wax blocks of dimension 4 × 4 × 9mm were made, sprued and invested in phosphate bonded investment. The mould was prepared using lost wax technique. For burnout, the ring was placed in a furnace and heated from 200°C to 900°C in one hour and maintained at that temperature for 30 min. Meanwhile the pressing furnace was preheated to 700°C for 45 minutes. High leucite ceramic

Figure 1: Schematic diagram of specimen consisting of core and veneer.

ingots and the plunger were then heated in the burnout furnace for 5-10 minutes. The heated ingots and plunger were introduced in the heated mould and was placed in the pressing furnace. The temperature increased at the rate of 60°C per minute till it reached 1075°C and was maintained for 20 minutes. At the end of this cycle the plunger pushed the ingots into the mould which was completed in 6-7 minutes. The ring was then cooled for a period of 45 minutes. The casting was divested and the sprues were removed. The bonding surface (4 × 4mm) of the specimens was sandblasted as in the case of the other specimens and air dried.

Preparation of Ni-Cr alloy specimens

Ten specimens of Nickel Chromium alloy of dimension 4 × 4 × 9mm were made by induction casting. The bonding surface (4 × 4mm) of specimens was sandblasted with 110µm aluminium oxide particles at 2.5 bar pressure and were steam cleaned and air dried. The metal surface was oxidised by heating at 600°C for 1min. A layer of opaque porcelain was added onto the bonding surface and fired at 980°C for 1 min.

Preparation of the veneer

Feldspathic porcelain was added on to the bonding surface of the previously made core specimens for a thickness of 3mm (Figure 2). The building up was done in layers and the firing temperature was limited to 930° C. Dimensions of the completed specimens were verified using an index measuring 12 × 4 × 4 mm

Embedding the specimens in acrylic block

The specimens were embedded in an acrylic block measuring 2.5 × 1.5 × 1.5cm. The core veneer interface of the specimen was placed at the level of the top surface of the acrylic block (Figure 3,4).

Figure 2: Specimens prepared.

Figure 3: Specimen embedded in acrylic (Schematic diagram).

Figure 4: Specimen embedded in acrylic with veneered portion exposed.

Shear bond test

These blocks were placed in the mounting jig of the universal testing machine and were subjected to shear force. Load was applied at a cross head speed of 0.5mm per minute until the fracture of the specimens occurred. The force required for fracturing was recorded. (Figure 5,6).

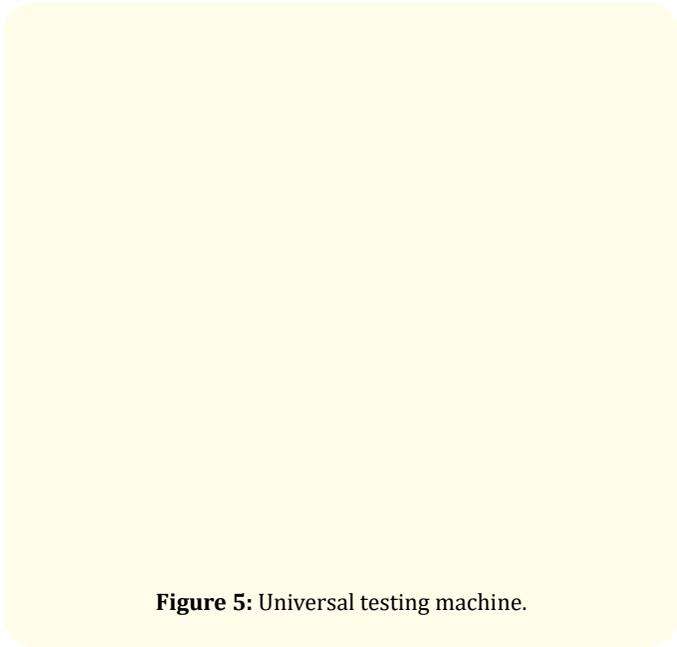


Figure 5: Universal testing machine.

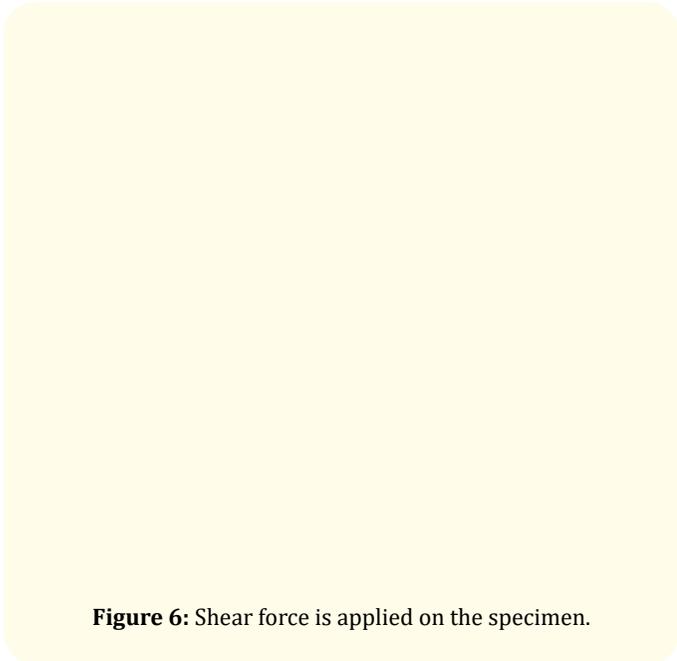


Figure 6: Shear force is applied on the specimen.

Surface Analysis

Scanning Electron microscopy and Energy Dispersive x-ray microanalysis were done using Zeiss Scanning electron microscope. The fractured surface of the specimens was used for this purpose (Figure 7).

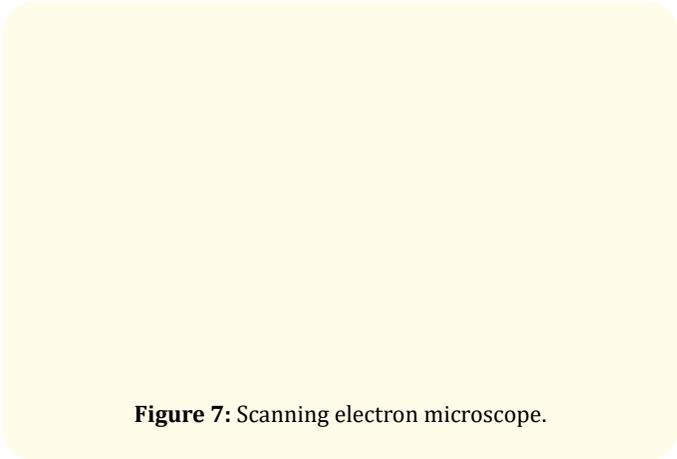


Figure 7: Scanning electron microscope.

Scanning Electron microscopic analysis

For Scanning Electron microscopic analysis, the specimens were first gold sputtered to increase the conductivity. The fractured surfaces of the veneer specimens were examined under scanning electron microscope at a magnification of 1000x. Zeiss (Germany) microscope was used.

Energy dispersive x-ray microanalysis

The fractured surfaces of the veneer specimens were analysed under the energy dispersive microanalysis to determine the elemental composition.

Results and statistical analysis

The results were tabulated and subjected to Factorial ANOVA test to detect statistically significant differences (Figure 8).

Results

- **Null hypothesis:** There is no significant difference in the mean Shear Bond Strength of the three materials i.e., Zirconia = Lithium disilicate = Nickel chromium
- **Alternate hypothesis:** There is a significant difference in the mean Shear Bond Strength of the three materials i.e., Zirconia/Lithium disilicate/Nickel chromium
- **Level of significance:** $\alpha = 0.05$
- **Statistical technique used:** Analysis of Variance (ANOVA).

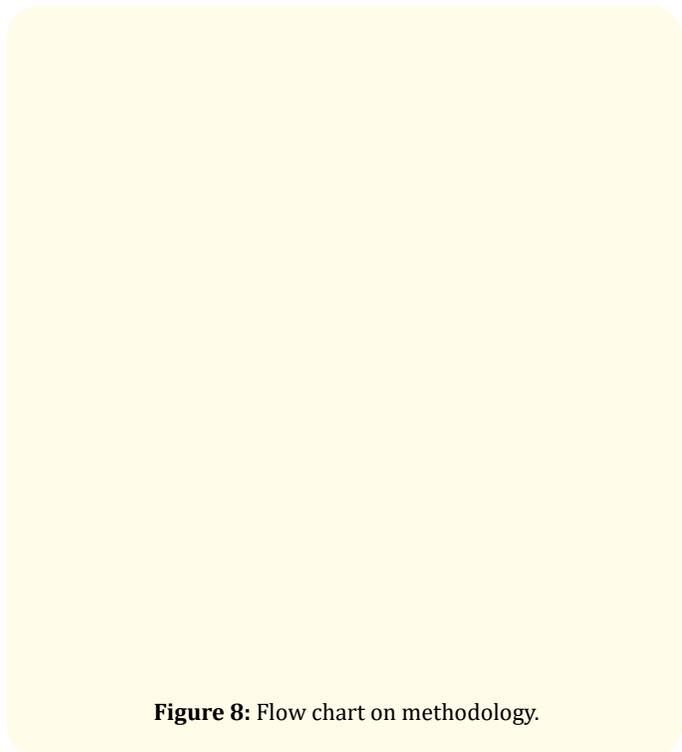


Figure 8: Flow chart on methodology.

- **Decision criterion:** The decision criterion was to reject the null hypothesis if the p-value was less than 0.05. Otherwise, the null hypothesis was accepted. If there was a significant difference between the groups, we carried multiple comparisons (post-hoc test) using Bonferroni test.

Shear bond strength (SBS)

The higher mean shear bond strength was seen between lithium disilicate core and the veneering ceramic followed by Zirconia core and the veneering ceramic and Nickel chromium base metal alloy core and the veneering ceramic (Table 1). The difference in mean SBS among the three materials was found to be statistically significant (P < 0.001) (Table 2). The difference in mean SBS was found to be statistically significant between Zirconia and Lithium Disilicate (P < 0.001) as well as between Lithium Disilicate and Nickel Chromium (P < 0.001). No significant difference was observed between Zirconia and Nickel Chromium (P > 0.05). (Table 3).

Scanning electron microscopy (SEM)

SEM images of lithium disilicate specimens showed predominantly cohesive failure. Higher magnification exhibited an opaque

Table 1: Mean Shear bond strength with Zirconia, Lithium disilicate, Nickel Chromium (N).

Material	Mean	Std	SE of	95% CI for Mean		Min	Max
		Dev	Mean	Lower	Upper		
Zirconia	284.87	31.66	10.01	262.23	307.51	230.60	316.40
Lithium Disilicate	409.40	33.40	10.56	385.51	433.29	366.40	488.60
Nickel Chromium	310.21	32.52	10.29	286.94	333.48	261.70	365.20

Table 2: ANOVA.

Source of Variation	df	Sum of Squares (SS)	Mean SS	F	P-Value
Between Groups	2	86628.309	43314.154	40.921	< 0.001 *
Within Groups	27	28578.990	1058.481	---	---
Total	29	115207.299	---	---	---

*Denotes significant difference.

Table 3: Multiple comparisons using Bonferroni method.

(I) Group	(J) Group	Mean	P-Value	95% CI for Mean Difference	
		Difference (I-J)		Lower Bound	Upper Bound
Zirconia	Lithium Disilicate	-124.530	< 0.001*	-161.668	-87.392
	Nickel Chromium	-25.340	0.279	-62.478	11.798
Lithium Disilicate	Zirconia	124.530	< 0.001*	87.392	161.668
	Nickel Chromium	99.190	< 0.001*	62.052	136328
Nickel Chromium	Zirconia	25.340	0279	-11.798	62.478
	Lithium Disilicate	-99.190	< 0.001*	-136.328	-62.052

*Denotes significant difference.

layer and oxide layer (Figure 9). SEM images of Zirconia specimens showed mixed adhesive and cohesive failures. Higher magnification exhibited many small pores from where the fracture originated and propagated (Figure 10). SEM images of Nickel Chromium specimens showed cohesive failure. Higher magnification exhibited an opaque layer and oxide layer (Figure 11).

Figure 9: SEM image of lithium disilicate-feldspathic porcelain specimen.

Figure 11: SEM image of Nickel chromium-feldspathic porcelain specimen.

Energy dispersive microanalysis (EDM)

EDM of the Lithium disilicate, metal ceramic, and zirconia specimens showed the chemical components present on the surface obtained after the SBS test and which were mainly oxygen and silica (Table IV). There were traces of sodium, zirconia and potassium too (Figure 12-14). The elemental composition could not be correlated to the bond strength.

Figure 10: SEM image of Zirconia-feldspathic porcelain specimen.

Figure 12: EDM of lithium disilicate-feldspathic porcelain specimen.

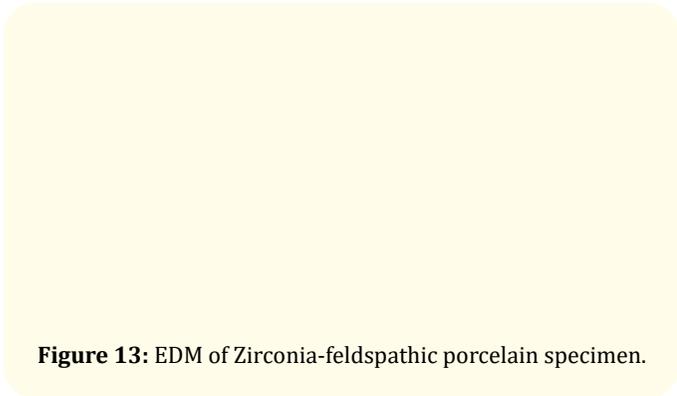


Figure 13: EDM of Zirconia-feldspathic porcelain specimen.

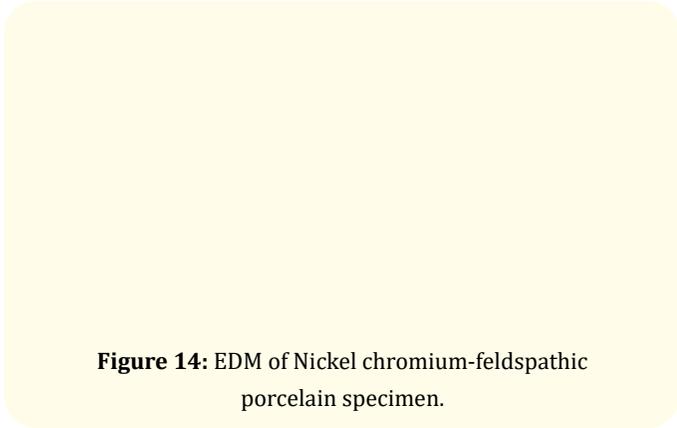


Figure 14: EDM of Nickel chromium-feldspathic porcelain specimen.

Discussion

Use of ceramics in the dental restorations has a long history. Though ceramics had desirable aesthetics, its brittle nature was considered as a limitation. Hence the later attempts were to integrate metals and ceramics so that there will be a desirable combination of strength of metals and aesthetics of ceramics. The initial attempts were based on mechanical interlocking. It had limitations and the ceramic was quite often chipped off the metal. Once the metal non-metal diffusion was found out, the profile of metal ceramic restorations underwent a sea change. The commonly used alloys consisting of cobalt, nickel, chromium and tin got diffused into the structure of ceramic and the ceramic elements got diffused in the reverse direction. Presence of alloys as a substructure improved the mechanical properties of the fixed restoration, but very often challenged the aesthetic achievements. Substituting metallic substructure has happened in this context, with the introduction of pressable ceramics (Lithium Disilicate). The mechanical properties of Lithium disilicate was not adequate to meet the masticatory loads especially when long span restorations were made. Zirconia was considered because it had superior strength (Flexural Strength of 900-1200 MPa and fracture toughness of 9-10 MPa) and at the same time possessed reasonably matching shades. Manipulation of zirconia was always a tough task but the precision brought in by CAD/CAM technology and the machinability of pre sintered zirconia has opened up newer avenues.

Table 4: Elemental values of specimens obtained from energy dispersive microanalysis.

	Oxygen (%)	Silica (%)	Zirconia (%)	Sodium (%)	Potassium (%)
Zirconia Specimens	77.32	39.43	3.55	6.43	6.38
Metal					
Ceramic Specimens	39.28	20.28	4.16	4.48	5.54
Lithium Disilicate Specimens	99.39	36.89	0	6.63	3.80

In order to obtain life like appearance and translucency the substrates have to be veneered with dentin and enamel porcelains. The binding mechanism between alloys and the veneering porcelains have been explored in detail, and it was almost optimised.

In the current study, bond strength between veneering feldspathic porcelain and three different substrate materials i.e., Zirconia, Lithium disilicate and Nickel Chromium alloy was evaluated.

The specimens tested were fabricated in rectangular forms of dimension 9 x 4 x 4mm so as to standardise the cross-sectional area. Rectangular specimens of all the three substrates were prepared and veneering porcelain (feldspathic porcelain) was layered at one end of the rectangular specimens to a dimension of 3 x 4 x 4mm. Shear test was considered adequate to measure the bond between two materials because of its validity and its ability to induce tension directly at the interface of core and veneer. The specimens were

embedded in standardised polymethyl methacrylate resin blocks of dimension 25 x 15 x 15mm such that core veneer interface was placed at the level of the surface of the block. Force required for fracturing the veneered portion was recorded. Fractured surface of specimens was subjected to Scanning electron microscopy to study the surface structure characteristics and energy dispersive microanalysis was done to know the elemental distribution of the surface layer.

Maximum bond strength was seen between lithium disilicate and veneering porcelain (409 N/25.58 MPa) and the minimum bond strength was observed between zirconia and the veneering porcelain (284 N/17.8 MPa). The bond strength between nickel chromium and veneering porcelain was 310 N/19.38 MPa. The difference in mean shear bond strength was found to be statistically significant between zirconia, lithium disilicate as well as nickel chromium. No significant difference was observed between zirconia and nickel chromium. The bonding mechanism between zirconia and veneer ceramic is still not fully understood. Dundar, *et al.* [11] reported shear bond strength in the range of 23-41 MPa and Al-Dohan [12] reported a shear bond strength in the range of 22-31 MPa for commercially available e max (lithium disilicate) all ceramic system which are similar to the results found in the present study. Pretti, *et al.* [13] evaluated the shear bond strength of metal ceramic bond of two Co-Cr alloys to be 48.39Mpa and 55.96 MPa. De Melo, *et al.* [14] reported the shear bond strength between porcelain and four alloys (two Ni-Cr and two Co-Cr alloys) as follows; 54 MPa and 63 MPa for Ni-Cr alloys and 71.7 MPa and 55.2 MPa for Co-Cr alloys. The difference in findings could be attributed to factors like the study design, and the properties of the materials used.

After the shear bond testing the fractured specimens were subjected to Scanning electron microscopy to evaluate the surface structure and shear characteristics. Energy dispersive microanalysis was done to find out the elemental composition of the sheared interface. The predominance of cohesive failures in the lithium disilicate groups suggest that adhesive zone had higher strength than the veneering ceramic. In contrast, for the Nickel chromium and zirconia groups adhesive failure was noted. The energy dispersive microanalysis showed mainly the elements such as silica and oxygen at the surface.

Bond strength between the core and the veneer is determined by many factors: the strength of the chemical bonds, mechanical interlocking, the type and concentration of defects at the interface, wetting properties and the degree of compressive stress in the veneer layer due to differences in the coefficients of thermal expansion

between the substrate and the veneering ceramic. The ideal difference in the coefficient of thermal expansion between the metal and ceramic is to be limited to $1 \times 10^{-6} \text{ }^\circ\text{C}$ [15].

Zirconia is used in different contexts: as posts in root canals (1989), as implant abutments (1995) and as all ceramic posterior fixed dental prosthesis (1998) [16,17]. Zirconia is a common choice for long span fixed dental prosthesis mainly because of its superior mechanical properties when compared to other all ceramic systems. The main mechanism of bonding between the zirconia core and the veneering ceramic is said to be by the difference in the coefficient of thermal expansion and the mechanical bonding between the two materials. To ensure maximum survival times, adequate occlusal tooth reduction is essential. Optimal clinical performance of ceramic products requires a minimal occlusal reduction of 2 mm for molar tooth preparations. If the ceramic is supported by a material with a high elastic modulus such as a ceramic or metal post or an amalgam build-up, less occlusal reduction (1.5 mm) need be done. Tinschert *et. al.* [6] reported that the fracture resistance of three-unit ceramic FPDs made of Cercon zirconia core ceramic (Dentsply Ceramco) was 1278 N twice as great as the values reported for In-Ceram Alumina (514 N) and Empress2 (621 N). The commonly used core IPS empress 2 ceramic is composed of 70% lithium disilicate crystals in a glass matrix and veneering ceramic consists of apatite crystals. The primary crystal particles in IPS Empress2 are 0.5 to 4 μm in length. A smaller concentration of lithium orthophosphate crystals ($\text{Li}_2\text{Si}_2\text{O}_7$) approximately 0.1 to 0.3 μm in diameter has also been reported. The bonding mechanism between the lithium disilicate and the veneering ceramic is mainly because of the nanofluoroapatite crystalline structure present in the lithium disilicate. The silica and the oxygen molecules present in veneering ceramic develop a covalent bond with the crystalline structure thereby enhancing the dimensional stability and the mechanical strength of the restorations.

Conclusions

The following conclusions were drawn from the present study

- Among the groups tested shear bond strength was maximum between lithium Disilicate core and the veneering feldspathic ceramic. Least shear bond strength was seen between Zirconia core and the veneering feldspathic ceramic. All ceramic restorations with lithium disilicate core and feldspathic veneer can resist clinical failure due to fracture of the veneering ceramic. Delamination of the veneering ceramic can be frequent with Zirconia core.

- Scanning electron microscopic analysis showed both adhesive and cohesive failures. Lithium Disilicate specimens showed predominantly cohesive failures. Multiple cracks and many pores were seen in the zirconia specimens and the failure was both adhesive and cohesive.
- Electron Dispersive microanalysis showed mainly oxygen and silica as the chemical constituents present on the fractured surface.

Limitations

Knowledge about the bonding between zirconia cores and veneering ceramic is still not understood fully. Improvement in material science might throw light in future towards this area. This can be interpreted as a limitation of the study as well as a future challenge.

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