

## Scanning Electron Microscopic Analysis and Bond Nature of Dual Forms of Resin Repaired and Thermocycled Crystalline Lithium Disilicate Ceramic

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### Abstract

**Aim:** The aim of this in vitro study was to qualitatively assess the mode of failure of dual forms of resin repaired lithium disilicate ceramic.

**Material and Methods:** Twenty two wax patterns were fabricated from customized stainless steel jig. The samples were divided into 2 groups with each group comprising of 11 samples. Group I wax patterns were heat pressed layered Lithium disilicate ceramic (IPS emax press, IPS emax Ceram Powder Dentin, Ivoclarvivadent, Schaan, Liechtenstein) and Group II wax patterns were heat press monolithic Lithium disilicate ceramic. The samples were etched with 5% HF for 20s, and one representative sample from each group was analyzed under SEM for etched surface characteristics. The remaining 20 samples were then silanized and repaired with composite resin, according to the manufacturer's instructions. These samples were then subjected to thermocycling and shear bond testing using universal testing machine and data was obtained. The data was then subjected to statistical analysis using non-parametric Mann-Whitney U test. Mode of failure was analyzed using scanning electron microscope.

**Results:** The mean shear bond value for Group I: 5.34 MPa and for Group II: 13.88 MPa. There was high statistical significant difference between the two test groups ( $P < 0.05$ )

**Conclusion:** The mean shear bond strength of repair composite resin bonded to monolithic lithium disilicate restorations is higher than that of bilayered lithium disilicate restorations. The repair of monolithic lithium disilicate ceramic crowns with repair composite resin is recommended and not for the bilayered lithium disilicate ceramic. Mixed mode of failure was observed in SEM analysis for both groups.

**Keywords:** Lithium disilicate glass ceramic, SEM analysis, Repair composite, 5% Hydrofluoric acid, shear bond.

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### I. Introduction

The ceramo-metal restoration had been the gold standard in crown & bridge procedures for several years. Although durable and time-tested, patient's demand for metal-free restorations mainly for esthetic reasons, and the demand had been met with various all ceramic systems.

In spite of the advantages of All – ceramic restorations including life-like appearance, biocompatibility and durability, there are still disadvantages to their use clinical use. fracture of layering ceramics still remains the primary cause of failure of all – ceramic crowns.<sup>[1, 2, 3, 4]</sup>

The evolution of glass-ceramics in the dental field was profoundly influenced by the increase in crystalline structure of up to 60-70% and reinforcement through lithium-disilicate. This led to a flexural strength two or three times higher and suitable for restorations in the molar region.<sup>[3]</sup>

Studies had shown that the contact damage is induced at the loading, occlusal surface<sup>[1]</sup> for molars and premolars or palatine surfaces for incisors. Wang et al<sup>[5]</sup> showed that all ceramic restorations demonstrated a fracture rate of 4.4% at a 5-yr follow-up in a clinical study.<sup>[2]</sup>

Repair composite restoration was a conservative method that can increase the longevity and durability of restorations while preserving the old restorations.<sup>[2, 3, 6, 7, 8, 9]</sup>

Adequate surface treatment for lithium disilicate glass ceramic was achieved with the HF concentration of 5% applied for 20 seconds.<sup>[10, 11, 12, 13]</sup> and the use of a silane agent to provide a high bond strength.<sup>[14, 15, 16]</sup>

Numerous studies, evaluating the shear bond strength between repair composite and monolithic lithium disilicate ceramics had focused on different repair composite resins and/or different types of surface treatment agents, including different concentrations and duration as their study variables.<sup>[2, 3,17, 18, 19, 20]</sup>

Standalone studies focusing on the SEM analysis between repair composite resin and bilayered lithium disilicate ceramic that had been subjected to suitable surface treatments were lacking in the literature. Moreover such studies comparing the bond strength between the repair composite resins bonded to either bilayered or monolithic lithium disilicate ceramic were lacking.

Therefore, in view of the above, the aim of the present *in vitro* study was to qualitatively assess the mode of failure of dual forms of resin repaired lithium disilicate ceramics. The null hypothesis of the present study was that there will be no significant difference in the SEM analysis and bond strength between the repair composite resin bonded to bilayered and monolithic lithium disilicate ceramic test groups.

## II. Materials And Methods

A total of 11 wax patterns with height 3mm, diameter 10mm, with central defect of diameter 4 mm which simulates the fractured site and debonded ceramic site of the restorations were prepared for bilayered lithium disilicate ceramic test samples as Group I, another 11 wax patterns with height 5mm, diameter 10 mm with central defect well of diameter 4mm which simulates the fractured site and debonded ceramic site of the restorations were prepared for monolithic lithium disilicate ceramic test samples as Group II were fabricated using customized stainless steel jig. (Fig. 1a,1b).

**Fig1a Group I wax pattern Bilayered**



**Fig 1b Monolithic Group**



11 wax patterns for Group I were sprued and invested with phosphate bonded investment (Bellavest® SH, Germany). The burn out process was carried out at 900°C after soaking time of half an hour. Lithium Disilicate monolithic ceramic press (IPS emax press, Ivoclarvivadent, Schaan, Liechtenstein) was carried out. Divesting was done using glass beads followed by ultrasonic cleaning with 1% HF acid. Each sample was finished with water emery paper of 220, 320 and 400 grit sizes, respectively.

For addition of layering ceramic, surface of the test samples were sandblasted with 50µm aluminum oxide particles at low pressure of 50 psi to create roughness. After sandblasting, opaque ceramic powder was added and fired, then dentin ceramic powder was added to the thickness of 2mm and fired under 750°C vacuum pressure. Final trimming and polishing was done using sintered diamond (Vijay dental products, Chennai, India).

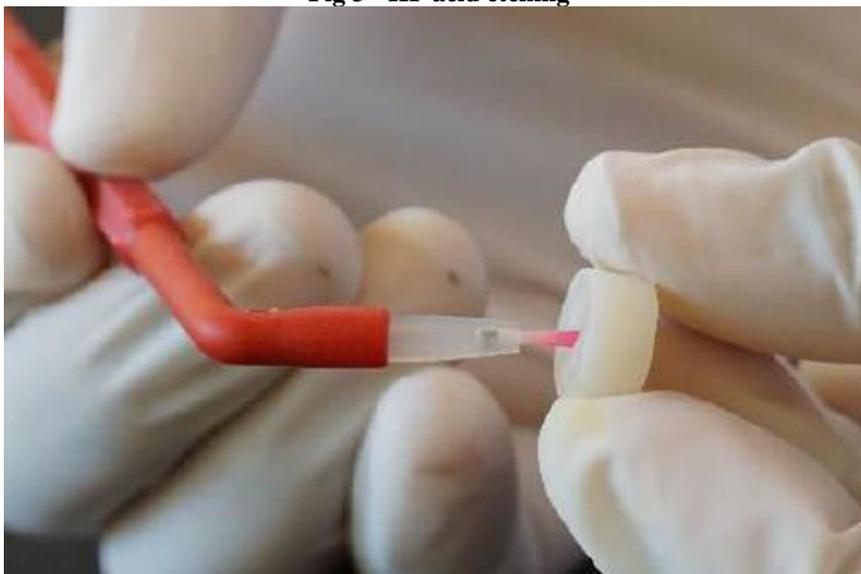
11 wax patterns for Group II were sprued and invested, pressed and finished following all necessary protocol as followed for Group I except the layering procedure which is not needed for Group II. Thus all 20 test samples with their specified dimensions were obtained. (Fig. 2a,2b)

**Fig 2a,2b -Group I and Group II – ceramic samples( Bilayered& Monolithic )**



The surface of defect well of test samples from groups I &II were etched with 5% HF for 20 seconds. The samples were then rinsed with distilled water for 1 minute and air dried. (Fig. 3 & 4)

**Fig 3 - HF acid etching**





**Fig 4 Distilled water rinsing**

One representative test sample was randomly taken from each test group for qualitative analysis of etched surface using scanning electron microscope (S- 3400N, Hitachi High Technologies Corporation, Japan). The sample was coated with a layer of gold using gold sputtering system and was examined at 2000X magnification to assess the surface characteristics (**Fig. 5**)



**Fig 5 Quality analysis of the surface characteristics of the etched surface of test samples by SEM**

Following etching of the test sample with 5% HF for 20 seconds, it was rinsed with distilled water for 1 minute and air dried. Next, Silanization was done (Monobond N, Ivoclar Vivadent, Schaan, Liechtenstein) The

silane coupling agent was applied to the defect well and allowed to react for 60s (**Fig. 6**) After Silanization, bonding agent (Heliobond, IvoclarVivadent, Schaan, Liechtenstein) was applied in a thin layer to the entire surface of the defect well (**Fig. 7**). The bonding agent was then cured for 10 seconds using a light cure unit (**Fig. 8**). Repair composite resin (Tetric N-Ceram, IvoclarVivadent, Schaan, Liechtenstein) was filled in individual increments of 2mm + 2mm + 1mm using a customized incremental jig and light cured for 10 seconds respectively(**Fig.9 & 10**). In this manner the defect well was filled until the 5mm depth was achieved.



**Fig 6 Silanization of etched surface**



**Fig 7 Application of bonding agent**



**Fig 8 Light curing of the bonding agent**



**Fig 9 Placement of the composite resin**



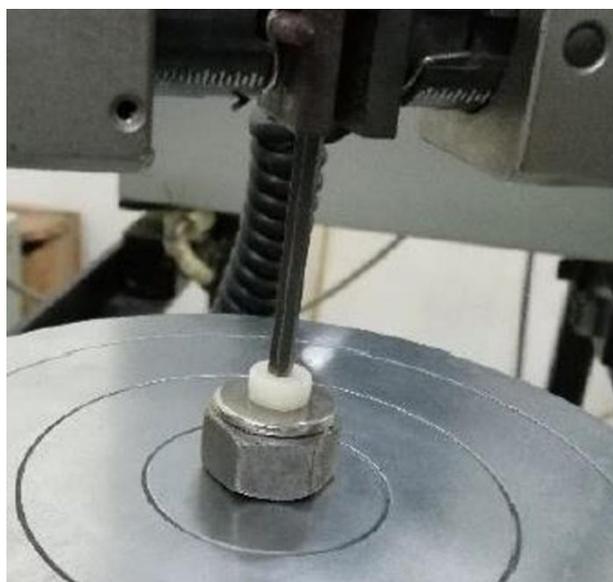
**Fig 10 - Light curing of the composite resin**

The test samples were stored in distilled water in individual containers for 24 hours at 37°C. Then the test samples were subjected to thermocycling for a total of 250 cycles in a distilled water bath between 5°C and 55°C with a dwell time of 60 seconds and a dry time of 10 seconds at 27°C using a thermocycling apparatus (Haake, W15, Germany) to simulate three months of clinical use. (Fig. 11). Upon completion of thermocycling, the test samples were again stored in distilled water in their respective containers till they were subjected to shear bond testing.



**Fig 11- Thermocycling of the test samples**

The test samples were subjected to shear bond testing individually in the universal testing machine (Instron 3382 100 KN, UK). The test samples were placed on the fixture with a ball ended pin of 4mm diameter positioned perpendicular to samples (Fig. 12). Force was applied to the test sample in such a way that the shear load was exerted directly on the whole of the repair composite surface at a cross head speed of 2mm/min until failure of the bond occurred. Shear bond force at which the bond failed was recorded in Newton (N) and shear bond strength (MPa) was calculated by dividing the force (N) at which failure of the bond occurred by the surface area of adhesion ( $\text{mm}^2$ ). The tested samples were stored in distilled water again.



**Fig 12 Shear testing of the test sample**

**Bond Strength (MPa) = Force (N) / surface area (mm<sup>2</sup>)**

Surface analysis of the mode of failure was carried out individually on one representative test sample per group using Scanning Electron Microscope (S- 3400N, Hitachi High Technologies Corporation, Japan) and the images obtained were compared between two groups and interpretation of results were drawn.

### III. Results

The basic data obtained from shear bond testing were tabulated using Microsoft Excel 10 (Microsoft, USA) and the mean and standard deviation were calculated. Basic and mean shear bond values of repair composite resin bonded to bilayered lithium disilicate ceramic restoration (Group I) and to monolithic lithium disilicate ceramic restoration (Group II) were tabulated. ( **Table 1 and 2**). The data were subjected to statistical analysis. The SPSS software for Windows 10.0.05 (SPSS Software Corp., Munich Germany) was used for statistical analysis. Non-parametric Mann-Whitney U test (**Table 3**) was used for statistical analysis to compare the respective overall mean shear bond strength values, between the two test groups. P value <0.05 was considered as significant.

### IV. Tables

**Table I: Basic and mean shear bond strength (MPa) of repair composite resin bonded to bilayered lithium disilicate ceramic restoration (Group I)**

Sample no	Shear bond strength in MPa
1	6.40
2	5.32
3	4.70
4	5.59
5	5.70
6	4.03
7	8.32
8	3.19
9	5.20
10	4.93
Mean (MPa)	5.34

#### Inference

Group I exhibited maximum shear bond strength value of 8.32MPa and minimum shear bond strength value of 3.19MPa. The mean shear bond strength was 5.34MPa.

**Table II: Basic and mean shear bond strength (MPa) of repair resin composite bonded to monolithic lithium disilicate ceramic restoration (Group II)**

Sample no	shear bond strength in MPa
1	14.23
2	12.66
3	11.61
4	16.72
5	16.64
6	19.60
7	13.47
8	11.69
9	12.77
10	9.43
Mean (MPa)	13.88

#### Inference

Group II exhibited maximum shear bond strength value of 19.60MPa and minimum shear bond strength value of 9.43MPa. The mean shear bond strength was 13.88MPa

**Table III: Comparative evaluation of the mean shear bond strength between repair composite resin bonded to bilayered lithium disilicate ceramic restoration (Group I) and repair composite resin bonded to monolithic lithium disilicate ceramic restoration (Group II) using Mann-Whitney U non- parametric test**

Groups	Mean shear bond strength (MPa)	SD	P value
I	5.34	1.379	0.000*
II	13.88	3.003	

## Inference

Mann-Whitney U test showed overall statistically significant difference between the two test groups with the 'p' value 0.000\*. Group II showed higher mean shear bond strength as compared Group I and this difference was found to be highly significant.

## Note :

\* 'p' value <0.05 denotes statistical significance.

S- Statistically significant

SD- Standard deviation

CI- 95%

DF -1

MPa-Megapascals

## V. Discussion

It is a known fact that, glass ceramic materials have been widely used in dentistry, studies with glass ceramic reinforced by lithium disilicate crystals had shown excellent clinical outcomes with great optical/mechanical properties<sup>[21, 22, 23]</sup> and high survival rate overtime.<sup>[3, 11, 19, 20, 24, 25,]</sup>

In the present study we had used lithium disilicate glass ceramic fabricated by hot press method in its two forms as monolithic and as core with fluorapatite dentin layering glass ceramic.<sup>[4]</sup>

Intraoral ceramic-repair system for chipped/fractured layering ceramic rely on strong resin bonds and adequate surface treatments.<sup>[11, 12]</sup> These systems may increase the longevity of a failing restoration and may be a provisional, cost effective alternative to immediate replacement.<sup>[10, 26, 27, 28, 29, 30, 31]</sup>

The clinical success of either a repaired ceramic restoration or a resin cemented ceramic restoration depends on the quality and durability of the bond between ceramic and the resin.<sup>[11, 32]</sup>

A desirable porous surface for repairing lithium disilicate ceramics was achieved by etching for 20 seconds<sup>[12, 13]</sup> which had been followed in the present study.

Application of a silane coupling agent to the pre-treated ceramic surface provides a chemical covalent and hydrogen bond and was a major factor for a sufficient resin bond to silica based ceramics.<sup>[32, 33]</sup> Silanization also increases wettability of the ceramic surface.<sup>[28, 34, 35, 36]</sup> Also, the use of a thin layer of unfilled resin prior to the composite resin improved bond strength and the interfacial quality between lithium disilicate glass ceramic and composite resin as it promotes a better infiltration to the superficial irregularities of the etched ceramic surfaces on application.<sup>[15, 18, 25, 37, 38]</sup>

Özcan<sup>[10]</sup> stated that thermocycling is more effective method for simulation of aging of composites and creates more challenging conditions for composite restorations. Thermocycling was performed aiming to create thermal strains at the bonding interface by thermal changes in water baths between 5-55°C.<sup>[3, 31]</sup> Thus, in this study, samples were subjected to a short thermocycling exposure simulating 3 months of clinical use and this was employed before shear bond testing.

The present study was to comparatively evaluate the SEM pictures and bond strength between repair composite resin bonded to bilayered lithium disilicate ceramic with layering and repair composite resin bonded to monolithic lithium disilicate ceramic.

In terms of evaluation method, shear bond strength test was chosen because it is the most common method for investigating the bond strength between various surfaces, luting agents, and ceramics.<sup>[9, 12, 39, 40, 41, 42, 43]</sup> The shear test was the commonly used test for evaluating the composite repair bonding.<sup>[8, 44,]</sup>

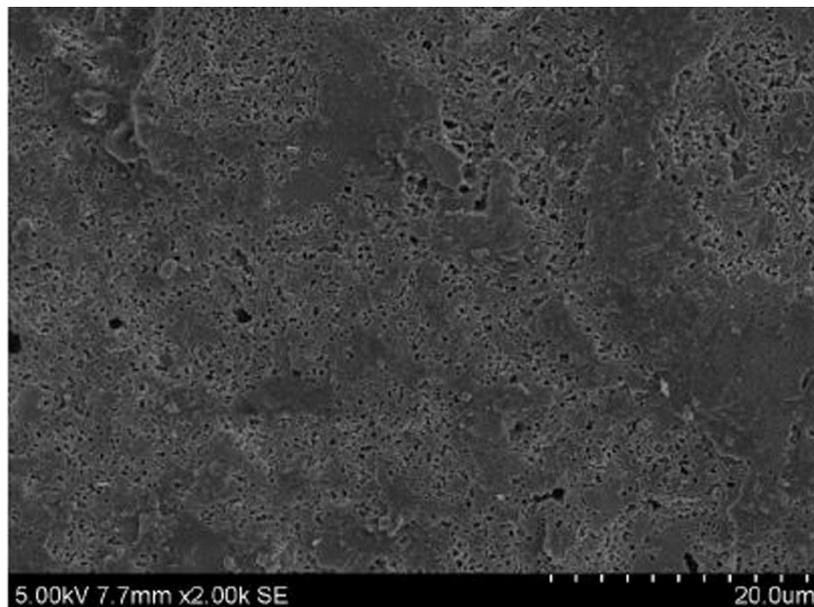
Shear bond strength test was performed by applying the force parallel to the bonding interface<sup>[21, 44, 45]</sup> and the shear bond strength was calculated by dividing the maximum load (in N) to the surface area (in mm<sup>2</sup>) of the composite resin. Shear bond strength value (in MPa) is the stress on the unit of area.<sup>[44]</sup>

For clinical applications, usually 15-25 MPa bond strength for direct composite resin has been reported as an optimal value depending on the composite material and repair method.<sup>[13, 44, 46,]</sup> In the present study, Group II showed a mean bond strength of 13.88 ± 3.00 MPa (ranging between 9.43-19.60 MPa), the results which are in agreement with previous studies.<sup>[1, 13, 25, 39, 47]</sup>

After shear bond testing, tested samples were subjected to SEM analysis to assess the mode of failure. SEM analysis at 2000X magnification was done on the repair composite resin surface that got sheared from the defect well of test sample during testing.

Shear bond strength values of Group I, showed low bond strength value when compared to Group II. Studies focusing on shear bond strength between repair resins bonded to lithium disilicate with layering are lacking, hence, shear bond strength values of Group I cannot be compared with the previous studies. Also, values obtained in this study were below the clinically accepted limits (5.34 ± 1.37 MPa). SEM photomicrograph (**Fig 13**) of pre-repaired etched surface (after 20s etching) of Group I representative sample (nanofluorapatite) sample, under 2000X magnification, revealed presence of both undissolved and dissolved surface topography.

There were significant areas of unetched ceramic surface present throughout the observed field, which could be one of the reasons for the low bond strength in first group. SEM photomicrograph of pre-repaired etched surface (after 20s etching) of Group II representative sample (lithium disilicate), under 2000X magnification image revealed significant change in surface microstructure as compared to that observed for Group I etched surface. The etched surface showed, a predominantly irregular surface characterized by numerous micro porosities in the form of pits, grooves and few striations, that were present throughout the observed field. Fewer areas of undissolved glassy phase of lesser dimensions were also visible, interspersed between the predominantly etched surfaces. And this image appeared similar to the images obtained by the previous authors in their respective surface topography study.<sup>[34]</sup>

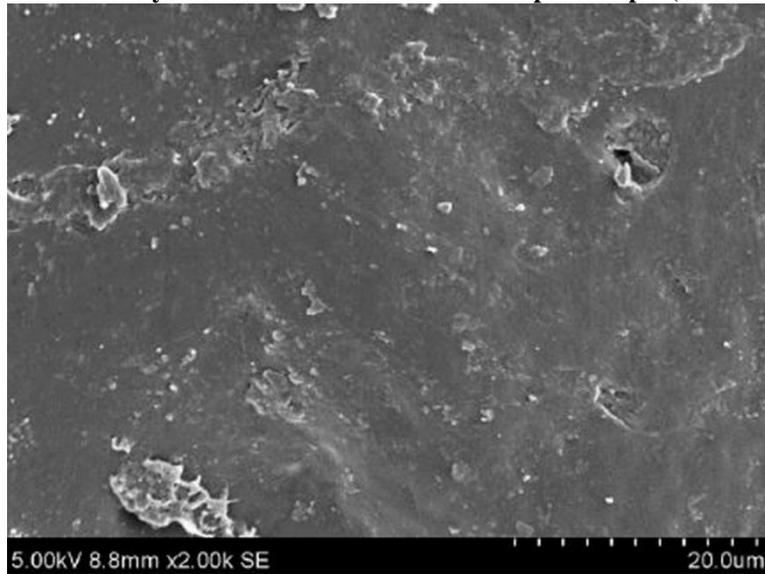


**Fig 13 Qualitative analysis of the etched surface of Group I sample ( Bilayered group)**

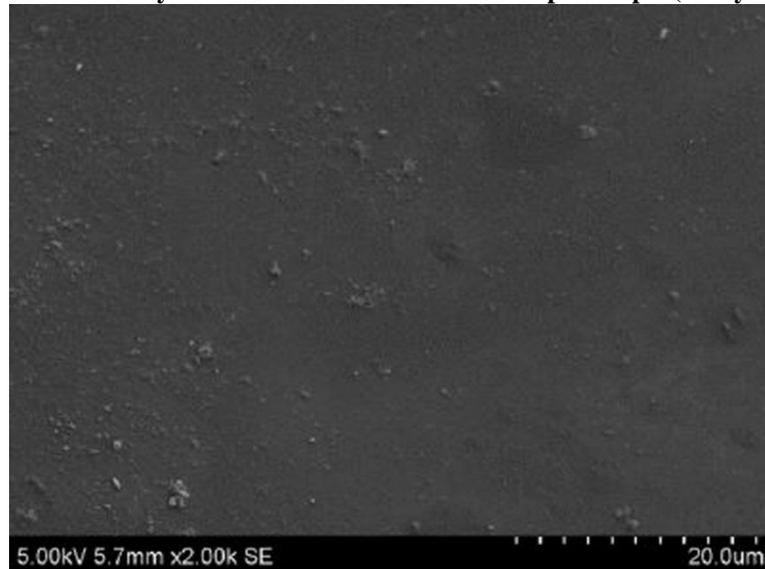
Thermal cycling, an artificial aging method of dental materials, which causes thermal strain on the bonding surface by influence of liquids and thereby thermal change is simulated.<sup>[19, 26]</sup> These could be the reasons for low bond strength for Group I. Shear bond values of both groups tabulated in the present study showed that Group II bond strength value was approximately 15MPa and consequently could be considered sufficient for clinical application. The difference between our results of Group II and those of other studies may be due to several factors such as differences in the concentration of HF used, types of composite resins used, different surface treatment methods in repair process and different testing conditions. Based on the results obtained in this study, the null hypotheses was rejected, because there was high statistical significant difference between the two tested groups ( $p < 0.05$ ). SEM analysis was done on the repair composite resin surface bonded to bilayered and monolithic lithium disilicate ceramic restorations, Group I SEM image at 2000X magnification showed, predominantly smoothen resin surface with sparsely distributed isolated areas of ceramic material, indicative of a mixed mode of failure that was predominantly adhesive in nature between repair resin and ceramic.

The mode of failure pattern observed was indicative of a vulnerable bond at the ceramic-repair resin interface. Group II SEM image at 2000X magnification also showed a predominantly irregular surface. There were increased areas of the ceramic material of greater thickness distributed over the resin surface, throughout the observed field, indicative of a mixed mode of failure that was predominantly cohesive in nature within the ceramic. The mode of failure pattern observed was indicative of improved bonding at the ceramic-repair resin interface. It appears from the results of the present study that the repairs of monolithic lithium disilicate ceramic restorations have a better survival rate as compared to bilayered lithium disilicate ceramic restoration. **(Fig14,15,16).**

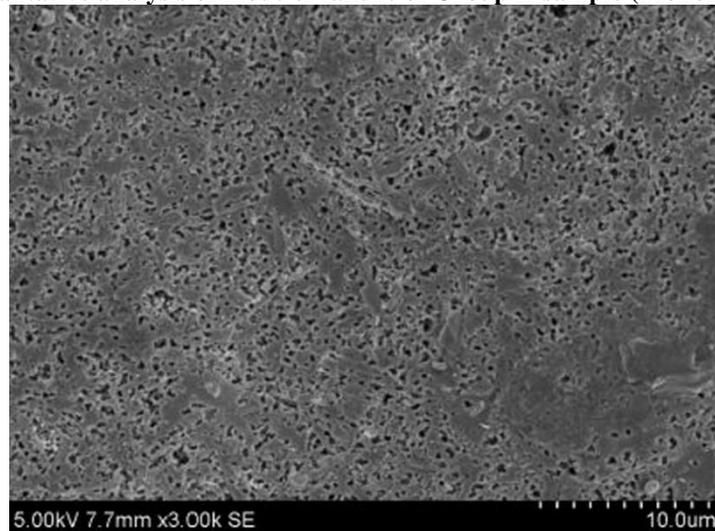
**Fig 14 Qualitative analysis of the etched surface of Group II sample ( Monolithic group)**



**Fig 15 Qualitative analysis of mode of the failure of Group I sample ( Bilayered group)**



**Fig 16 Qualitative analysis of mode of failure of Group II sample (Monolithic group)**



## CLINICAL SIGNANCE

Repair of fractured or chipped monolithic glass ceramic restoration is recommended because of better and superior bond strength values with composite resin when compared with bilayered glass ceramic restorations. Replacement of fractured bilayered glass ceramic restoration is recommended and not repairs due to minimal bond strength values.

Lithium disilicate crowns are expensive compared to porcelain fused to metal crowns. In the event of chipping or fracture, the repaired crown will come in handy especially for those patients who cannot afford a new restoration immediately. Special attention should be given in the shade selection and matching of the resin to glass ceramic.

## VI. Limitations

Samples were subjected to a short thermocycling exposure simulating 3 months of clinical use. The impact of different concentrations of hydrofluoric acid coupled with different durations of application, using different repair resins subjected to longer thermocycling periods may yield different results than those obtained in the present study. It had been proven that the type of composite resin influences its bond strength to ceramic.<sup>[23, 26]</sup> Further studies employing the above parameters coupled are recommended to enhance the results obtained with the present study.

## VII. Conclusions

Within the limitations of the present study, it was concluded that the repair of monolithic lithium disilicate ceramic crowns with repair composite resin had a better bond strength than the bilayered lithium disilicate ceramic crowns and therefore should be widely used in clinical situations. Mixed mode of failure was observed in SEM analysis for both groups.

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