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EVALUATION OF EFFECT OF ACID ETCH ON SHEAR BOND STRENGTH OF TWO CERAMIC REPAIR TECHNIQUES

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ABSTRACT

Background: Metal ceramic crown and fixed partial dentures could expose to fracture of ceramic veneer with and without metal exposure. The replacement of this restoration is costly, time consuming and removal of restoration could expose abutment teeth to fracture and this lead to generation of new repair systems with different surface treatment. **Objective:** The main objective of present study was to evaluate the effect of two type of acid etch with two type of ceramic repair technique on the shear bond strength of two different failures of metal ceramic restoration. **Subject and methods:** Eighty metal discs (20 mm in diameter × 0.7 mm thick) were fabricated from nickel-chromium alloy (Kera, USA). Feldspathic porcelain (VITA, Germany) were applied over one test surface of the discs in the thickness of 1.8 mm followed by conventional firing. The defect, was created in 1/4th area of the metal-ceramic discs (40 samples exposure metal and 40 sample with exposure ceramic). Then all samples were sandblasted with (50µ AL2O3) and beveled subdivided according to type of repair technique into 40 samples repaired with Ivoclar vivadent repair technique (with and without 37% phosphoric acid), and 40 samples repaired with Pulpdent repair technique (with and without 9.6% hydrofluoric acid). After that all the repaired samples were stored in distilled water at (37°c) for 24h. After thermocycling at(6-60°c), all the samples were stored at (37°c) for additional

7 days then shear bond strength of all the samples were measured with Instron Universal testing machine. **Results:** One way ANOVA showed non-significant effect through application 37% phosphoric acid and sandblasting with Ivoclar repair technique for exposure metal and exposure ceramic. Also one way ANOVA showed non-significant effect through application 9.6% hydrofluoric acid and sandblasting with Pulpdent repair technique for exposure metal and exposure ceramic of MCR. Comparison between two techniques using student s t - test showed, Pulpdent repair technique with application 9.6% hydrofluoric acid and sandblasting for exposure metal and exposure ceramic of metal ceramic restoration resulted in higher significant shear bond strength in comparison to Ivoclar repair technique with application of 37% phosphoric acid for exposure metal and exposure ceramic of metal ceramic restoration. Pulpdent repair technique and Ivoclar repair technique without acid resulted in similar effect on shear bond strength of exposure metal and exposure ceramic of MCR.

KEYWORDS: Metal ceramic repair, Acid etch, sandblasting, shear bond strength.

INTRODUCTION

Ceramic fused to metal restoration is regarded as the gold standard in fixed prosthodontics treatment.[1] On times, fractures do occur in ceramic as a result of trauma, metal flexure or ceramic fatigue, and a decision on how to repair the resultant defect need to be made. Reconstruction of the prosthesis is a costly affair and time consuming and it is therefore worthy to attempt repair with composite resin, especially in less severe cases.[2,3] An easy alternative is to repair the deficiency using one of the many proprietary porcelain repair system. However, for the repair to withstand functional loads, the bond between the repair material and remaining restoration must be strong and durable.[4] The establishment of durable and reliable bond between a dental and resin composite is important in dental practice because it is extensive use in resin bounded restoration and porcelain repair systems.[5] The situation are mentioned for repair of metal porcelain restoration; fractures in porcelain only with no metal exposure, fractures with some exposure of metal and fractures with complete deveneering of porcelain exposure with exposure all metal.[4] Repair of fractured metal-ceramic crowns aims to reestablish the function and esthetics of restorations by using various repair materials.[6,7] With the goal of promoting a satisfactory adhesion between the repair materials and the surfaces to be repaired, specific treatment of the substrate must be performed.[8] These may be treatments that promote mechanical retention, such as airborne-particle abrasion with aluminum oxide, diamond bur and etching with hydrofluoric acid[9] or phosphoric acid.[10] In addition, treatments may be used that promote chemical adhesion, such as silanization[11,12] and the application of adhesive primers.[13] Finally, treatments that promote mechanical retention as well as chemical adhesion may also be considered, such as the deposition of silica by conventional airborne-particle abrasion[14] or by the use of specific equipment.[15] The association of these types of treatments is frequently used to improve the bond strength between the repair material and the surface of a fractured prosthesis.[16] Until recently, due to lack of materials with a defined and specific protocol for repair of metal-ceramic restorations, it was a common practice to use different combinations of the available adhesive systems and composite resins in conjunction with a variety of surface treatments. However, with the emergence of different intraoral ceramic repair systems in current time there is a need for establishing an optimum bond strength value and a standardized technique for repair of metal-ceramic restoration.[17]

METHODOLOGY

Samples preparation: A silicon (Oomoo-USA) mold was prepared from machined stainless steel, disk which was fabricated to be 20mm in diameter and 0.7mm height;[17] this will aided waxing procedure of samples. Eighty disk of casting inlay wax was prepared from silicon mold. Each ten wax patterns were sprued, invested, casted, finished and polished according to manufacture instruction of **(Bego, Germany)**. The metal disc samples were prepared for ceramic application according to manufacture instruction **(Vita, Germany)**. Ceramic was applied in thickness of (1.8mm) over all surfaces of metal discs (0.2mm opaque, 0.8mm dentine, and 0.8mm enamel) with aid of custom made jig. Three different heights of custom made metal jigs Fig (1) have been made for standardization the porcelain layering application, the custom made jig have the same shapes and diameter of metal disc but with three different heights. Brass metal was used to fabricate custom made jig with stainless steel spring in center of jig to facilitate working procedures. Three different heights custom made jig were named as (opaque custom made jig with stander height 0.9mm), (dentine custom made jig with stander height 1.7mm), (enamel custom made jig with stander height 2.5mm). Finally the metal ceramic samples finished and glazed with a firing temperature (880˚c) without vacuum according to manufactures instruction of **(Vita, Germany)** to achieve a uniform thickness of (2.5mm).

Figure 1: Three different heights of custom made metal jigs.
(A) Custom made metal jig for opaque buildup with stander height 0.9mm.
(B) Custom made metal jig for dentine buildup with stander height 1.7mm.
(C) Custom made metal jig for enamel buildup with stander height 2.5mm.

Failures creation: The failures were created to exposed metal surface in 40 samples and exposed ceramic surface in 40 samples by making depth orientation grooves to remove quarter of the total area of the sample. These depth orientation grooves were reduced with the help of straight diamond fissure bur of (2mm) diameter until metal was exposed Fig (2) and to achieve (1.2mm) uniform thickness in ceramic samples Fig (2).[17] A straight fissure bur fixed on lubrication free air hand- piece with water spray coolant and dimmable LED, hand-piece can rotate 360° and water spry coolant reduce heat generated on working materials to prevent micro-cracks and helps removal of cutting debris. All the failure area of samples of two major groups was roughening with aluminum oxide (50µm) for (10 sec.) with pressure of (35-60psi) at distance (10mm) from the sample surface and was cleaned with ultrasonic steamer. The failure ceramic margins were beveled at 45degree of (1-2mm) width with flame shaped diamond bur under water irrigation. Then area subsequently was dried with oil-free compressed air.[17,18] After roughening and beveling the failure area, all of samples were repaired as per manufacture's instruction with one of the respected repair techniques, which are: Ivoclar vivadent repair technique **(Germany)**, Pulpdent porcelain prep technique **(USA)**.

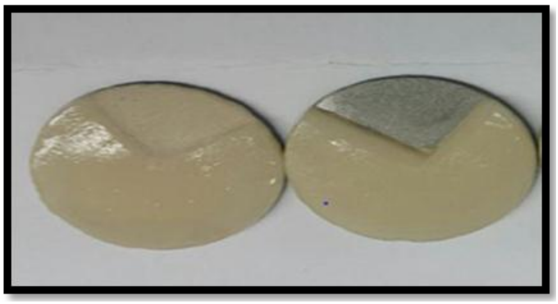

Fig 2: Failure formation (exposure ceramic and exposure metal).

Sample grouping: All the samples were divided into two major groups according to repair technique used. Two types of repair technique (Ivoclar vivadent repair technique as in table (1) [(A=40 sample), Pulpdent porcelain prep technique as in table (2) (B=40 sample)].

Table 1: Ivoclar vivadent repair technique (A).

Ivoclar repair technique	40 samples	With/ without
Group: AA1M	10 samples	Exposure metal with 37% phosphoric acid
Group: AA1C	10 samples	Exposure ceramic with 37% phosphoric acid
Group: AA2M	10 samples	Exposure metal without 37% phosphoric acid
Group: AA2C	10 samples	Exposure metal without 37% phosphoric acid

Ivoclar repair procedures with acid: Each 10 samples of (AA1M) and (AA1C) repaired with application of 37% phosphoric acid for (1 min) and then the sample cleaned with water to remove the acid gel and dried it with oil-free compressed air. then steps of Ivoclar repair procedure were continued according to manufacture instruction of (Ivoclar vivadent repair technique, Germany) which was as follow: Monobond was applied with disposable brush on the exposed ceramic and allowed to reacting for (60sec.), samples dried with oil-free air, followed that a thin layer of Heliobond was applied to the entire surface area which needs to repair and then all excess was removed with compressed air and cured for (10sec.). The sample of exposure metal and exposure ceramic fixed to the lubricated custom made metal jig and repaired with layer of ceramic composite (Tetric N- Ceram M2) that adapted with suitable instrument until (1.2mm) of missing ceramic was restore and cured it for (10sec.), except for exposure metal samples direct opaque composite was applied to prepared metal surface with 0.5mm as maximum thickness and cured it for (20sec.) and then ceramic composite it is applied and cured. After complete the composite buildup and cured it, the sample was removed from lubricated jig and any excess was removed with suitable fine grained diamond grinding instrument.

Ivoclar repair procedures without acid: Each 10 samples of group (AA2M) and group (AA2C) repaired according to manufacture instruction of (Ivoclar vivadent repair technique, Germany) which was as follow: Monobond was applied with disposable brush on the exposed ceramic and allowed to reacting for (60sec.), samples dried with oil-free air, followed that a thin layer of Heliobond was applied to the entire surface area which needs to repair and then all excess was removed with compressed air and cured for (10sec.)**.** The sample of exposure metal and exposure ceramic fixed to the lubricated custom made metal jig and repaired with layer of ceramic composite (Tetric N- Ceram M2) that adapted with suitable instrument until (1.2mm) of missing ceramic was restore and cured it for (10sec.), except for exposure metal samples direct opaque composite was applied to prepared metal surface with (0.5mm) as maximum thickness and cured it for (20sec.) and then ceramic composite it is applied and cured. After complete the composite buildup and cured it, the sample was removed from lubricated jig and any excess was removed with suitable fine grained diamond grinding instrument.

Table 2: Pulpdent porcelain prep technique (B).

pulpdent repair technique	40 samples	With/ without
Group: BB1M	10 samples	Exposure metal with 9.6% HF acid
Group: BB1C	10 samples	Exposure ceramic with 9.6% HF acid
Group: BB2M	10 samples	Exposure metal without 9.6% HF acid
Group: BB2C	10 samples	Exposure metal without 9.6% HF acid

Pulpdent repair procedures with acid: Each 10 samples of group (BB1M) and group (BB1C) repaired According to manufacture instruction of **(Pulpdent porcelain prep technique, USA)** which was with application of 9.6% hydrofluoric acid etchant that applied to exposed surface and leaved for (1min) with evacuating fumes with vacuum. Then etch gel was cleaned with water and dried with oil-free compressed air. After that the Pulpdent Dry-Rite drying agent was applied to the defect area and dried it rapidly with oil-free air. Pulpdent silane Enhancer was applied to the etched metal and ceramic surface and dried it with oil-free air. The sample of exposure metal and exposure ceramic fixed to the lubricated custom made jig and repaired with layer of ceramic composite (Tetric N- Ceram M2) that adapted with suitable instrument until (1.2mm) of missing ceramic was restore and cured it for (10sec.), except for exposure metal group (BB1M), Direct opaque composite was applied to prepared metal surface with (0.5mm) as maximum thickness and cured it for (20sec.) then ceramic composite it is applied and cured.

Pulpdent repair procedures without acid: Each 10 samples of group (BB2M) and group (BB2C) repaired According to manufacture instruction of **(Pulpdent porcelain prep technique, USA)** but without application of hydrofluoric acid. Pulpdent Dry-Rite drying agent was applied to the defect area and dried it rapidly with oil-free air. Then Pulpdent silane Enhancer was applied to the metal and ceramic surface and dried it with oil-free air. The sample of exposure metal and exposure ceramic fixed to the lubricated custom made jig and repaired with layer of ceramic composite (Tetric N- Ceram M2) that adapted with suitable instrument until (1.2mm) of missing ceramic was restore and cured it for (10sec.), except for exposure metal group (BB2M), direct opaque composite was applied to prepared metal surface with (0.5mm) as maximum thickness and cured it for (20sec.) then ceramic composite it is applied and cured.

Aging and thermocyclying: After fabrication of repaired samples, they were all stored in distal water at (37˚c) for (24 hours) and were then subjected to 500 thermal cycles between (6-60˚c) with (30 sec) of dwell time and (12 sec) of transfer time. The temperature of baths (6-60 ˚c) was constantly monitored by a thermometer and adjusted using ice and boiling water. All samples were then stored in distal water at (37˚c) for additional 7 days.[19,17]

Sample testing: Shear bond strengths were tested with (Laryee - WDW- 50) Instron universal testing machine using chisel shaped rod with a cross head speed of (0.5mm/min). The tested specimens were placed in the lower member (jaw) of the testing machine in such a way that the long axis of the chisel-shaped rod was parallel and closed as possible to the composite ⁄ defect substrate interface. Specimens were secured tightly in place to ensure that the disc was always parallel to the vertical plane. The specimens were loaded until they failed, and mode of failure was examined visually and was recorded by a single observer as either adhesive (failure at substrate-resin interface), cohesive failure within the substrate or within restorative material) or combination (area of adhesive and cohesive failure).The force was recorded in (Newton), which has been divided by the surface area (mm^2) to obtain shear bond strength to obtain shear bond strength calculated in Mpa.

Mode of failures examination: After completion the testing procedure of shear bond strength. All the samples of all groups were examined visually in an effort to determine the type of bonding failure. The type of bonding failure was recorded by a single observer as either: For metal substrate, adhesive (failure at metal substrate-resin interface), cohesive (failure within metal substrate or within restorative material) or combination (area of adhesive and cohesive failure). For ceramic substrate: adhesive (failure at ceramic substrate-resin interface), cohesive (failure within ceramic substrate or within restorative material) or combination (area of adhesive and cohesive failure)].

A scanning electron microscope (SEM): The specimens were then secured on to an aluminum holder with conductive gold paint and secondary electrons images were observed under Angstron advanced filed- emission scanning electron microscope (USA) at 300X magnifications to examine surface morphology. SEM was made for six samples of exposure metal and exposure ceramic substrates after different surface treatment as in fig (3).

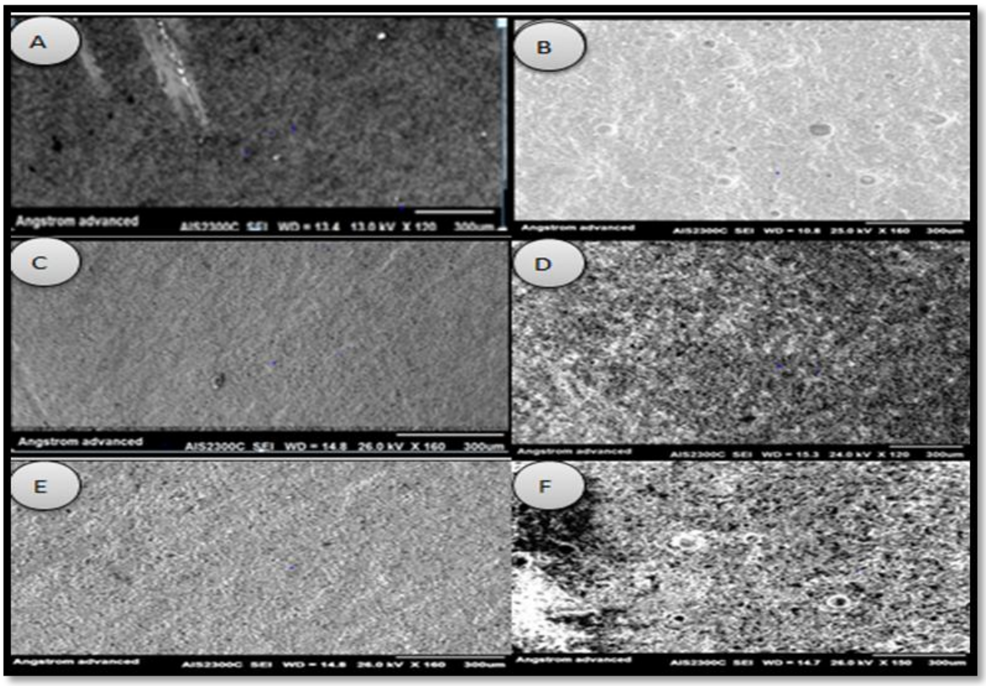

Fig 3: SEM for exposure metal and exposure ceramic of metal ceramic after different surface treatment.

A: Exposure metal roughening with 50µm Al2O3.

B: Exposure ceramic roughening with 50µm Al2O3.

C: Exposure metal roughening with 50µm Al2O3 and 37%H3po4.

D: Exposure ceramic roughening with 50µm Al2O3 and 37%H3po4.

E: Exposure metal roughening with 50µm Al2O3 and 9.6% HF.

F: Exposure ceramic roughening with 50µm Al2O3 and 9.6% HF.

Statistical Analysis: IBM SPSS statistics program version 21 used for statistical analysis of current study and Microsoft Excel 2010 for graphics presentation. Descriptive statistical including mean and stander deviation were calculated for shear bond strength in (Mpa) for Ivoclar repair technique and Pulpdent repair technique, for each main group individually. Analysis of one way ANOVA test was performed to determine whether there is significant difference with and without acid for exposure ceramic or exposure metal for each repair technique used. Student's t-test was used for multiple comparisons between the two major repair techniques (Ivoclar and Pulpdent). In the above tests, P- values more than 0.05 were considered as statically insignificant, whereas P-values equal to 0.05 were regarded as statically significant.

RESULTS

Shear bond strength of ivoclar repair technique: The descriptive statistics (mean values, stander deviation, maximum and minimum values) of shear bond strength of the first major group (Ivoclar repair technique group AA1&AA2 are presented in table (3), figure (4).

Table 3: Descriptive Statistics of group AA1 and AA2.

G	N	Min.	Max.	Mean± Std
AA1M /Exposure Metal With Acid	10	10.446	12.739	11.108±0.823
AA2M /Exposure Metal Without Acid	10	11.065	19.108	15.312±3.046
AA1C/Exposure Ceramic With Acid	10	10.446	16.178	12.357±1.638
AA2C/Exposure Ceramic Without Acid	10	7.988	14.661	11.847±2.529

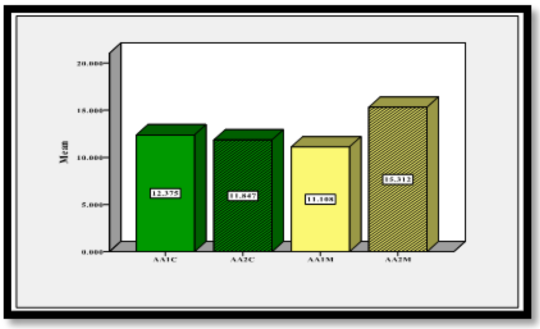

Figure 4: Bar chart showed mean value of Ivoclar repair technique groups.

From table (3) it's clear that the higher mean value for shear bond strength has been recorded for the group of exposure metal repaired without acid (AA2M) , which was with mean value **(Mean=15.312±3.046Mpa)** and this was higher than the mean value of exposure metal repaired with acid (AA1M) **(Mean =11.108±0.823 Mpa)**. The data was analysis statistically by using analysis of one way **(ANOVA)**. The result showed that there is no significant difference **(P-value >0.05)** between the two subgroup (AA1M), (AA2M); P- value was **(P-value=0.159)**. And also from table (3); it’s obvious that the second highest shear bond mean value for exposure ceramic has been recorded for the group of exposure ceramic with acid (AA1C), which was **(Mean=12.357±1.638Mpa)** and this was higher than the mean value of exposure ceramic repaired without acid **(Mean=11.847±2.529Mpa)**. Then the statistical analysis of the data was performed by using analysis of one way **(ANOVA)** between the two subgroup (AA1C), (AA2C) .The result showed that there is no significant difference **(P-value > 0.05)**; the **(P-value=0.703)**.

Shear bond strength of pulpdent repair technique: The summery of descriptive statics (mean values, stander deviation, maximum and minimum values of shear bond strength) of the second major group Pulpdent repair technique groups (BB1&BB2) are presented in table (4), fig (5).

Table 4: Descriptive Statistics of group BB1 and BB2.

G	N	Min.	Max.	Mean± Std
BB1M /Exposure Metal With Acid	10	11.975	21.656	14.561±3.009
BB2M /Exposure Metal Without Acid	10	11.210	19.751	15.745±2.661
BB1C/Exposure Ceramic With Acid	10	14.503	28.080	20.360±4.406
BB2C/Exposure Ceramic Without Acid	10	8.280	16.999	11.211±2.425

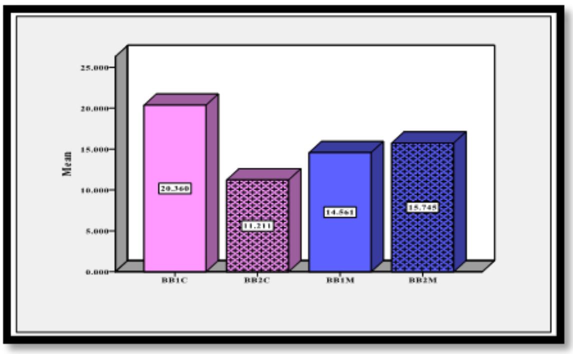

Figure 5: Bar chart showed the mean value of Pulpdent repair technique groups.

And from table (4), it's clear that the higher mean value for shear bond strength register for exposure ceramic repaired with acid (BB1C), **(Mean =20.360±4.406Mpa)** and this was higher than the mean value of exposure ceramic repaired without acid (BB2C) **(Mean =11.211±2.425Mpa)**. Statistical analysis of the data by using of one way ANOVA was performed. The result showed that there is no significant difference **(P-value > 0.05)** between the two subgroup (BB1C), (BB2C); **(P value = 0.402)**. And also from table (4) it's obvious that the second highest shear bond strength mean value for exposure metal without acid (BB2M), was **(Mean =15.745±2.661Mpa)** and this was higher than the mean value of exposure metal repaired with acid (BB1M) was **(Mean =14.561±3.009Mpa)**. And when analyzing this data statistically by using one way **(ANOVA)** between two subgroup (BB1M), (BB2M) ,the results showed that there is no significant difference **(P-value > 0.05)**; **(P-value=0. 174)**.

Comparison of shear bond strength between Ivoclar and pulpdent repair techniques

Student s t-test was used to evaluate the significance of difference between each pair of two major groups Ivoclar repair technique (A) and Pulpdent repair technique (B). The results are listed in table (9). It showed the following:-

1) Ivoclar repair technique with acid (37% phosphoric acid) for exposure metal showed highly significant difference **(P<0.01)** compared to Pulpdent with acid (9.6% hydrofluoric acid) for exposure metal. Also when comparing the two techniques for exposure ceramic.
2) No significant difference **(P>0.05)** was found between Ivoclar repair technique without acid compared to pulpdent repair technique without acid for exposure metal and exposure ceramic.

Table 9: Student S t- test between ivoclar (A) and pulpdent (B) repair techniques.

Pair of groups	t	P-Value	C.S
AA1M / Exposure Metal With 37% phosphoric acid & BB1M /Exposure Metal With 9.6% hydrofluoric acid	**3.311**	**0.009**	**P<0.01 (HS)**
AA2M / Exposure Metal Without 37% phosphoric acid & BB2M / Exposure Metal Without 9.6% hydrofluoric acid	**0.346**	**0.737**	**P>0.05 (NS)**
AA1C / Exposure ceramic With 37% phosphoric acid & BB1C/ Exposure ceramic With 9.6% hydrofluoric acid	**5.770**	**0.000**	**P<0.01 (HS)**
AA2C / Exposure ceramic Without 37% phosphoric acid & BB2C / Exposure ceramic Without 9.6% hydrofluoric acid	**0.507**	**0.624**	**P>0.05 (NS)**

DISCUSSION

The technology of fabrication metal ceramic crown or fixed partial dentures involving many stages which can lead to errors and failure of the restoration.[20] The repair system has distinct role in repairing and addressing these errors and failures by enhance the mechanical chemical bond between resin and metal or ceramic substrate by mechanically increasing the surface area and decreasing the surface tension and by causing physical alteration which promoted adhesion of resin to porous surface of the metal ceramic restoration; physical alteration achieved by selectively dissolving the glassy matrix chemically.[21]

The first major group Ivoclar repair technique (group A)

1: Groups of exposure metal with and without phosphoric acid etchant (AA1M&AA2M): The present study showed no significant difference when comparing between subgroups of exposure metal repaired with Ivoclar repair technique with use of 37% phosphoric acid (AA1M) and exposure metal repaired with Ivoclar repair technique without 37% phosphoric acid (AA2M) {P>0.05}. This result may be due to etched exposed metal surface after air- born particle abrasion with phosphoric acid would remove the alumina layer that impacted on this surface. Thus chemical bonding of the silane to the silica deposited on the metal substrate may not have occurred and, hence, the bond strength was due to only the mechanical surface treatment resulting from airborne-particle abrasion with aluminum oxide.[1] The role of silane coupling in metal surface treatment has been studied by Cobb, Vargas et al in 2000,[16] they founded at the first and prior to the bonding procedures, silane works on protect the metal surface from the contamination. And it has been recommended for use in enhancing the strength of adhesion in resin to metal through different mechanisms. Dental silanes made on increasing wettability of metal surface with resin materials; and this increase resulted from the bonding that occur between the absorbed water and acidic oxides on the metal surface and to the methacrylate groups inn resin adhesive. Then this eases the penetration and interlocking of resin into the rough metal surface which had increased of surface area and energy after treated it with sandblast. A coating that provided by silane and over it the adhesive resin can freely flow to achieve intimate contact with subsequent micromechanical retention. Despite of the non-statistical difference, the variation between the bond strength mean values of evaluated systems for both groups (AA1M=[11.108±0.823] & AA2M=[15.312±3.046]) explained the integrated relationship between the sandblasting and alloy primer in improve the shear bond strength of repair technique that used these materials in repair of exposure metal surface of metal ceramic restoration. This finding in agreement with study results of dos Santos, Fonseca et al in 2006[1] that said ceramic repair system which used in repair of exposure metal showed higher bond strength after air abrasion of metal exposure surface and this due to existence of alloy primer containing MDP (10methacryloyloxydecyl dihydrogen phosphate). MDP contains an ester phosphate group which forms a strong chemical bonding with oxide layer on surface of alloy for reliable bond of resin to alloy. Similar result was concluding by Mansoor, Aljorani et al. 2014[22] he concluded that the effect of chemical bonding agent (metal primer) alone with a non-abrasion metal surface on shear bond strength value is insignificant and it require air abrasion of metal surface to act in a perfect form. The metal or alloy primers are generally phosphate for base metal alloys and thione or thiol for noble metal alloys.[23] The Ni-Cr alloy used in this study as a metal substructure[24] is a hard non -amorphous materials difficult to finishing and polishing and this alloy was difficult to etched with 37% phosphoric acid to produce any increase in the rough surface (mechanical roughening) and in consequence shear bond strength. This showed by the electron scanning (SEM) fig (3-C). SEM for exposure metal surface roughened with sandblasting without acid fig (3-A) showed uniformly frosted surface, but fig (3-C) when exposure metal samples scanned after sandblasting and etching with 37% phosphoric acid, the frosted surface of the metal like to be more prominent with less lightness and this may be because of cleaning function of phosphoric acid to air abrasion particle of the sandblasting that impacted on the uniform frosted abrade metal surface.

2: Groups of exposure ceramic with and without phosphoric acid etchant (AA1C&AA2C): Non-significant difference was found between subgroups of exposure ceramic repaired with Ivoclar repair technique with 37% phosphoric acid (AA1C) and exposure ceramic repaired with Ivoclar repair technique without 37% phosphoric acid (AA2C) {P>0.05}. And this may be due to the phosphoric acid was not enough to create any benefit in creating a strong reliable bond strength between ceramic and composite. And this in agreement with studies of Ajlouni, Bishara et al in 2005[25] that cited on 37% phosphoric acid didn't produce any type of alteration to the ceramic morphology. The acid used for cleaning the surface after mechanical roughening. Aida, Hayakawa et al in 1995,[12] they stated that etching porcelain surface with phosphoric acid not enough to produce required mechanical interlocking to composite resin of repair system. In dental laboratories, the typical procedure is blasting the surface with alumina oxides particles of an average size of (50µm) under an air pressure of (380kpa) for around (10-15s) at a perpendicular short distance (10mm) from the nozzle to the surface.[26] While Darvell, Samman et al in 1995[27] founded in their study, when silane coupling agent has been applied; ═AL─O─Si≡ bonds may be formed between the alumina layer and silane which is necessary to give higher shear bond strength**.** But when the phosphoric acid that have a recognized role in surface cleaning was used,[25] the alumina layer may be removed from the ceramic surface and this may lead to inhibition to the chemical reaction of silane coupling agent which depended on this layer to create better shear bond strength. Horn in 1983[28] found in his study, the silane treatment didn't require phosphoric acid etching for porcelain. Mechanical roughening by sandblasting with silane coupling agent is sufficient for repair metal ceramic restoration. The SEM of samples with exposure ceramic and sandblasted without etching with phosphoric acid fig (3-B) showed amorphous surface with numerous different size of the micro-porosities and multiple fisher like as white fisher lines, while the SEM of samples with exposure ceramic and sandblasted with etching by application 37% phosphoric acid for (1minute) fig (3-D) showed that etching with phosphoric acid was effective for cleaning and clearing the numerous different sizes of micro-porosities that impacted with 50µ of alumina oxide layer of sand blasting which is important for provide durable shear bond strength with silane coupling agent.

The second major group Pulpdent repair technique (group B)

1: Groups of exposure metal with and without hydrofluoric acid etchant (BB1M&BB2M): For Pulpdent groups with exposure metal repaired with and without hydrofluoric acid (BB1M&B2M) one way (ANOVA) showed no-significant difference {P>0.05}. Aida, Hayakawa et al in 1995[12] stated that development of silane agents in repair systems don't require etching with hydrofluoric acid. Also etching porcelain surface without ultrasonic cleaning affect the bond strengths of composite to porcelain. The present study in agreement with[29] and[30] that perform their study on samples that involve part exposed metal with border of ceramic. Chung and Hwang in 1997[29] stated that ceramic surface etched with 9% hydrofluoric acid lead to cracks that might cause ceramic failure. In present study exposure metal samples had border of ceramic that was etch 9% hydrofluoric acid in (BB1M subgroup). De Jager, Feilzer et al. in 2000,[31] also concluded in study of adhesive and roughness of joined surface parts, that excessive rough surface may lead to stress concentration inside the internal porcelain which may consequently weaken the interfacial bonding between ceramic border of exposure metal substrate and composite. The SEM finding of present study for exposure metal sandblasted but without 9.6% hydrofluoric acid fig (3-A) which appears uniformly frosted surface and when comparing it with the SEM finding of exposure metal sandblasted and etched with 9.6% hydrofluoric acid fig (3-E) showed no clear difference in the surface roughness.

2: Group exposure ceramic with and without hydrofluoric acid etchant (B1C&B2C): The results for these groups of exposure ceramic repaired with Pulpdent repair technique with 9.6% hydrofluoric acid (BB1C) and exposure ceramic repaired with Pulpdent repair technique without 9.6% hydrofluoric acid (BB2C) are not statistically significant different {P>0.05}. The non-significant difference between subgroup (BB1C&BB2C) related to greater role of silane coupling agent. Pameijer, LOUW et al in 1996[32] found in their study adequate bond between ceramic and composite resin is attained with a silane coupling agent and an adhesive silanes acting as mediators enhancing adhesion between ceramic and composite resin (inorganic and organic matrices) through double reactivity. The result of presented study in agreement with[12] and[28] who found that this silane agent don't require acid etching of porcelain. And also Uehara and Sakurai in 2002[33] stated in their study on important fact, they cited on theirs no linear relation between increase the rough surface and shear bond strength. However this result in conflict with study of Pameijer, LOUW et al in 1996[32] that conclude the bond between ceramic surface and composite can be made with HF acid etching through creation of micromechanically retentive surface by acid and activity of silane agent. And the present study results disagreed with outcomes of[34] and[35] which they referred to the etching of dental porcelain with hydrofluoric acid, followed by silanzation was reported to give higher bond strength of resin composite to porcelain as compered without acid etching. Also differ from study of Shahverdi, Canay et al in 1998[36] who found the silane coupling agents used in conjunction with HF acid etching after sandblasting increased the bonding strength of resin to porcelain surface under shear like loading. All these disagrees studies have materials and sample design different from the sample design which used in our study which it is more near to the laboratory and clinical condition. The sample that repaired in the present study was a combination of metal and ceramic in the same sample as metal ceramic restoration and this differ from the samples that used in above mentioned studies which repaired the separated metal sample and separated ceramic samples so it is doesn't undergo to the same condition that applied to the samples of present study from the creation of the failure types of exposure metal and exposure ceramic of metal ceramic restoration. The finding of SEM of this study in agreement with study Chung and Hwang in 1997[29] that showed the porcelain surface treated with hydrofluoric acid revealed a surface characterized by deep channels and pores. The SEM explain that the exposure ceramic surface of metal ceramic restoration that roughening with (50μm alumina-oxide) only without 9.6%HF acid etching fig (3-B), showed irregularity and micro-roughness and porosities, while the exposure ceramic surface of metal ceramic restoration repaired with a combination of airborne particle (50μm alumina-oxide) and 9.6%HF acid etching fig (3-F) showed increasing in the irregularity surface with high roughness area like sharp obtuse angularities which is spreading on etched ceramic surface.

Comparing between two techniques (Ivoclar vivadent and Pulpdent prep technique) for exposure ceramic and exposure metal with acid

1: Comparison between groups of exposure metal repaired with Ivoclar Vivadent technique using 37% phosphoric acid (AA1M) and Pulpdent prep technique with 9.6% hydrofluoric acid (BB1M): The results showed high significant difference between the two techniques with application of acid {P<0.05}. The important finding of this study is cited on the prominent effect of hydrofluoric acid with Pulpdent prep technique on exposure metal of metal ceramic restoration rather than the phosphoric acid with Ivoclar vivadent technique. As previously mentioned in the study of dos Santos, Fonseca et al in 2006[1] on the function of Phosphoric acid with Ivoclar technique in group (AA1M) which was worked as cleaner for both the substrate surface and beveling ceramic borders from the silica layer of air-born abrasion which was necessary to provide durable bond strength with alloy primer and silane coupling agent, and also may be the design of the sample[17] which is clearly affected when treated with HF acid in combination with sandblasting and silane coupling agent that's provide distinct effect on the ceramic boundary which lead to increasing the shear bond strength of repairing exposure metal. And this harmonize with result of study of Gourav, Ariga et al in 2013[4] for repair of metal ceramic restoration, they stated that surface treatment with sandblasting and etching 9.6% hydrofluoric acid produce significantly high shear bond strength than surface treatment with sandblasting and etching with 37% phosphoric acid. Also may be the difference in the type of adhesive system that used in present study with different chemical compositions and concentration of solvents in the silane of two type of repair technique (Ivoclar and Pulpdent repair kits) may have a variable adhesion on the substrate surface acidity.

2: Group exposure ceramic repaired with Ivoclar repair technique with 37% phosphoric acid (AA1C) and Group exposure ceramic repaired with Pulpdent repair technique with 9.6% hydrofluoric acid (BB1C): Under the condition of this study, samples of exposure ceramic surface of metal ceramic restoration (BB1C) repaired with 9.6% hydrofluoric acid showed significantly higher shear bond strength than those samples of metal ceramic restoration repaired with 37% phosphoric acid (AA1C). And this due to the fact that the great part of porcelain structures is consist of silica, the acidic ions of the hydrofluoric acid penetrate into the Si-O framework creating ten thousand micro porosities/mm2 in a honey comb appearance, then the fluoride ions attack the Si-O soluble fluorosilicate[37] And also in another study[38-40] that established when porcelain is treated with further reacts with HF to form a soluble hydrofluoric acid, H2SiF6. The glassy matrix is dissolved and can be rinsed away. A microscopically porous, high energy and micro-retentive surface is thus obtained. And also study of Berry, Barghi et al in 1999[41] said; the values of shear bond strength were higher with silane application after etching. Thus the finding suggests that bonding to porcelain is mostly chemical and not mechanical. And this present study in agreement with outcomes of Stella, Oliveira et al in 2015[42] that explained that etching with 37% phosphoric acid for one minute will not scratch the porcelain, it only clean the surface. They also instituted higher shear bond strength with hydrofluoric acid to the porcelain samples when etched by HF as when compared with phosphoric acid etch that provided low shear bond strength. But this result of present study disagreement with the studies of Leibrock, et al in 1999[10] which did not observe statistically significant differences in bond strength following thermocycling and mechanical overloading among the systems which used phosphoric or hydrofluoric acid. The reason may be due to the difference materials of metal substructure used in study of[10] which was fabricated from (Co-Cr-Mo alloy) and also different sample design compare to design of our present samples study and also may be due to the difference in the type of adhesive systems.

Comparing between two techniques (Ivoclar vivadent and Pulpdent prep technique) for exposure ceramic and exposure metal without acid

1: Group exposure metal repaired with Ivoclar repair technique without 37% phosphoric acid (AA2M) and Group exposure metal repaired with Pulpdent repair technique without 9.6% hydrofluoric acid (BB2M): The result of comparison between the two groups exposure metal repaired with Ivoclar repair technique without 37% phosphoric acid (AA2M) and exposure metal repaired with Pulpdent repair technique without 9.6% hydrofluoric acid (BB2M) was non-significant and this may be due to the two groups treated with the same roughening and beveling surface with sandblast and diamond bur and this mostly give the same roughening surface for both groups. This result in agreement with study of Bagis, Ustaomer et al in 2009[43] that recommended mechanical retention was enhanced through the using of beveled surface on the porcelain and this created by using diamond burs and sandblasting. It has been stated that roughening the surface of exposed metal or ceramic by sandblasting offers good results. Also the current study results in contrast with study of Cobb, Vargas et al in 2000,[16] they concluded that the base metal alloy surface that roughening by grit-blasting, resulted in a metal surface having a significant amount of aluminum oxide particles, which provide a prominent effect on the shear bond strength. Mansoor, Aljorani et al. 2014[22] reported that, the shape of roughness and undercut that produced by abrasion the base metal alloy with (50µm AL2O3) more stable for retention. The study of dos Santos, Fonseca et al in 2006[1] indicated that the abrasion of the metal substrate with aluminum oxide particles would provide a favorable micromechanical retention and this may have preferred by the adhesive, the abrasion aluminum oxide particles made on increasing substrate wetting and, consequently increased the contact between the resin opaque and the treated metal surface. Every groups of Ivoclar repair technique have (alloy primer), and Pulpdent repair technique have (Dry Rite) which applied to it is group of exposure metal before silane coupling agent and after applying the silane coupling agent may be both these materials (alloy primer and Dry Rite) provided the same effect on metal surface to result in non-statically difference on shear bond strength, in spite of different composition and function of this two materials. However, and according to the manufacturing of the Dry Rite (Pulpdent prep porcelain repair kit) that descripted the important role of Dry Rite for [complete chemical drying of restorative surface when residual moisture may affect bonding and cementation] according to manufacture instruction of pulpdent prep technique. While Jain, Parkash et al in 2013[17] established in their study, the alloy primer (Ivoclar vivadent technique) which having MDP (10-methacryloyoxydecyl dihydrogen phosphate) is important for bonding of composite resin to metal surface of metal ceramic restorations. And also air abrasion with (50µm AL2O3) particles, when used on the surface of metal ceramic restoration will increased the shear bond strength for ceramic and metal substrate when repaired with different repair system.

2: Group exposure ceramic repaired with Ivoclar repair technique without 37% phosphoric acid (AA2C) and Group exposure ceramic repaired with Pulpdent repair technique without 9.6% hydrofluoric acid (BB2C): The non-significant result was registered when comparing the groups of two technique (AA2C&BB2C) and this result was related to fact that the important role is action of silane coupling agent[44] that founded in both repair technique of two technique in spite of different composition of them. And also prior to application of silane coupling agent of each technique for both groups of exposure ceramic, the sandblasting with (50µm AL2O3) to all exposure ceramic samples were made.[26] In addition to all these ceramic surfaces were beveled in order to achieve strong bond strength and esthetics.[43] It is important to provide durable chemical and micromechanical bonds between dental ceramic and composite resin and for this study the mechanical retention may was prompted through the use of beveled surfaces on the ceramic using diamond burs and sandblasting. The study of Berry, Barghi et al in 1999[41] indicate that the silane coupling agents are favorable to covalent bond development between the porcelain surface and the composite, and they also advance the wetting of the porcelain surface for the bonding. In one of the principal studies of Shahverdi, Canay et al in1998[36] on porcelain repair with composite, using a silane agent, it was demonstrated that this technique might be a laboratory and clinical key to fractured porcelain**.** Lacy, Laluz et al. in 1988[45] they observed when silane was not used; relatively a week shear bond strength of the composite to porcelain was produce, unrelatedly of type of surface treatment, with the type of failure that occurs at the interface.

Mode of failure: After visually examination of the mode failure, the results showed in the group of exposure metal surface repaired with Pulpdent technique with 9.6% hydrofluoric acid (BB1M) that the bond mostly cohesively or ⁄ mixed, science this group revealed high shear bond strength value compared with group of exposure metal surface repaired Ivoclar technique with 37% phosphoric acid (AA1M), and this was clearly from the percentage value of failure that account for both groups, which was 70% cohesive and 30% mixed for group of exposure metal surface repaired Pulpdent technique with 9.6% hydrofluoric acid (BB1M). For group exposure metal surface repaired Ivoclar technique with 37% phosphoric acid (AA1M) was 90% adhesive and 10% mixed. And also, the results showed that the group of exposure ceramic repaired with Pulpdent technique with 9.6% hydrofluoric acid (BB1C), the bond failure mostly cohesively, science this group revealed highest mean shear bond strength values compared with group of exposure ceramic surface repaired with Ivoclar technique with 37% phosphoric acid (AA1C). And this indicates that strong bond occurred between the composite resin and ceramic surface and failures occurred in the composite resin rather than the interface of the composite and ceramic. The percentage value of cohesive failure for group (BB1C) was 80% cohesive and 20% mixed rather than group (AA1C) which was 80% adhesive and 20% mixed. The results of both groups of exposure metal surface that repaired with both technique but without two types of acid (AA2M) and (BB2M) ,the bonding failure was mostly cohesively or ⁄ mixed for all samples of both groups of exposure metal , since our study showed high shear bond strength values. The percentage value of failure was 60% cohesive and 40% mixed for (AA2M) and the same it for group (BB2M). At the end, the results showed; both groups of exposure ceramic surface of metal ceramic restoration that repaired with Ivoclar technique (AA2C) and Pulpdent technique (BB2C) but without two types of acid (37% phosphoric acid) and (9.6% hydrofluoric acid), the bond failed adhesively for nearly all samples. Therefore both of these groups showed the lowest shear bond strength values, and tis indicate that weak bond was occurred between the composite resin and the ceramic surface; the failures occurred between them. The percentage value of failure was 90% adhesive and 10% mixed and this was similar for both groups (AA2C) and (BB2C).

CONCLUSION

Within limitation of present study, the following conclusion can be drawn:

1) Application of 37% phosphoric acid and sandblasting with Ivoclar repair technique for exposure metal of metal ceramic restoration showed no significant effect on shear bond strength.

2) Application of 37% phosphoric acid and sandblasting with Ivoclar Viva Dent repair technique for exposure ceramic of metal ceramic restoration showed no significant effect on shear bond strength.
3) Application of 9.6% hydrofluoric acid and sandblasting with Pulpdent repair technique for exposure metal of metal ceramic restoration showed no significant effect on shear bond strength.
4) Application of 9.6% hydrofluoric acid and sandblasting with Pulpdent repair technique for exposure ceramic of metal ceramic restoration showed no significant effect on shear bond strength.
5) Pulpdent repair technique with application 9.6% hydrofluoric acid and sandblasting for exposure metal and exposure ceramic of metal ceramic restoration resulted in significantly higher shear bond strength in comparison to Ivoclar repair technique with application of 37% phosphoric acid for exposure metal and exposure ceramic of metal ceramic restoration.
6) Pulpdent repair technique and Ivoclar repair technique without acid resulted in similar effect on shear bond strength of exposure metal and exposure ceramic of metal ceramic restoration.

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