To Evaluate the Effect of Various Surface Treatments on the Shear Bond Strength of Three Different Intraoral Ceramic Repair Systems: An In Vitro Study

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Abstract Fractures of metal-ceramic restoration pose an esthetic and functional dilemma both for patient and the dentist. Intraoral repair systems eliminate the remake and removal of restoration. Many intraoral repair materials and surface treatments are available to repair intraorally fractured metal-ceramic restoration. Bond strength data of various materials and specific technique used for repair are necessary for predicting the success of a given repair system. This study evaluated the shear bond strength of three different intraoral repair systems for metal-ceramic restorations applied on exposed metal and porcelain surface. One hundred and twenty metal discs (20 mm in diameter × 0.7 mm thick) were fabricated with nickel–chromium alloy (Mealloy, Dentsply, USA). Feldspathic porcelain (Duceram, Degudent, Germany) were applied over one test surface of the discs in the thickness of 1.8 mm followed by conventional firing. The defect, which simulates clinical failures were created in 1/4th area of the metal-ceramic discs. The metal-ceramic discs samples were divided into ceramic substrate (Group I, \(n = 60\)) and metal substrate (Group II, \(n = 60\)), according to the defect location. Then, samples of ceramic substrate (Group I) and metal substrate (Group II) were subdivided into A, B according to the surface treatments (A; roughening with diamond bur and B; abraded with 50 µm Al₂O₃) and repaired with one of the intraoral repair systems tested (a. Ceramic repair system, Ivoclar Vivadent; b. Clearfil repair system, Kurary, c; Porcelain repair system, 3M ESPE). All the repaired samples were stored in distilled water at 37 °C for 24 h. After thermocycling at 6–60°C, all the samples were stored at 37 °C for additional 7 days. Shear bond strength of all the samples were calculated by using Universal testing machine.

The mean shear bond strength values for the group I (A/B) were as follows: Ceramic repair system (9.47 ± 1.41/14.03 ± 2.54 MPa), Clearfil repair system (14.03 ± 2.32/14.64 ± 2.28 MPa), and Porcelain repair system (14.41 ± 3.96/14.86 ± 3.10 MPa). The mean shear bond strength values for the group II (A/B) were as follows: Ceramic repair system (9.42 ± 1.44/18.61 ± 2.60 MPa), Clearfil repair system (14.44 ± 3.23/14.98 ± 2.73 MPa), and Porcelain repair system (11.86 ± 2.24/13.24 ± 2.72 MPa). Air abrasion with 50 µm aluminum oxide particles is the preferred surface treatment. Porcelain repair system showed the highest shear bond with air abrasion for ceramic substrate and for metal substrate Ceramic repair system showed the highest bond strength with air abrasion as a surface treatment. This study suggest that the three repair systems tested are adequate for intraoral chairside repair of metal-ceramic restoration when air abrasion is used for surface treatment of the substrate.

Keywords Repair of metal-ceramic restoration · Intraoral repair · Chair side repair of metal-ceramic restoration

Introduction

Metal-ceramic restorations are always considered as a gold standard for fixed partial restoration due to their ability to fulfill the functional as well as esthetic demands [1]. However, they occasionally show fracture of the veneered ceramic which have been reported to range from 2.3 to 8%...
The cause of clinical fracture of veneering ceramic on metal-ceramic restorations is multifactorial including clinical factors (inadequate tooth reduction during dental preparation or parafunctional occlusion), laboratory factors (lack of proper framework support for porcelain, intraceramic defects) or trauma [4].

Repair of fractured metal-ceramic restorations aims to reestablish the function and esthetics of restorations by using various components of intraoral repair systems. To achieve optimum adhesion between the composite resin and the fractured metal-ceramic restorations, combination of mechanical and chemical retentive system should be used [4].

Intraoral repair system enhances the mechano-chemical bond between resin and metal or ceramic substrate by mechanically increasing the surface area, decreasing the surface tension and creating very fine surface roughness, and chemically by selective dissolution of glassy matrix causing physical alteration to promote adhesion of resin to the porous surface of the fractured metal-ceramic restoration [5].

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.

This study was undertaken to evaluate the effect of different surface treatments (roughening with diamond bur and air abrasion with 50 μm aluminum oxide particles) on shear bond strength of three commonly available intraoral ceramic repair systems namely (Ceramic repair system, Ivoclar Vivadent, Germany; Clearfil repair system, Kurary, Japan; Porcelain repair system, 3M ESPE, Germany) to metal and ceramic substrate.

Materials and Methods

One hundred and twenty metal-ceramic discs were fabricated (20 mm in diameter and 2.5 mm thick) with nickel-chromium base metal alloy of 0.7 mm thick (Mealloy; Dentsply, USA) and ceramic (Duceram, Degudent, Germany) in the thickness of 1.8.

Fabrications of samples were accomplished by waxing the disc patterns using a circular metal matrix (custom made) with an opening of 20 mm in diameter and 0.7 mm in thickness. The wax patterns were invested and casted as per manufacturer’s instructions. The metal discs were finished with the help of carborundum discs, high-speed lathe, metal trimmer and were sandblasted to achieve a uniform thickness with a final dimensions (20 mm in diameter × 0.7 mm thickness).

The metal discs samples were prepared for ceramic application. Ceramic was applied in the thickness of 1.8 mm over one test surface of metal discs (0.2 mm opaque, 0.8 mm dentine, and 0.8 mm enamel) with an aid of a custom made metallic jig. Finally, the metal-ceramic discs samples were finished and glazed to achieve a uniform thickness of 2.5 mm.

The one hundred and twenty metal-ceramic discs were divided into ceramic substrate (Group I, n = 60) & metal substrate (Group II, n = 60, according to the defect location. The defect in the Group I (n = 60) discs were created to exposed body ceramic by making depth orientation grooves. These depth orientation grooves were reduced to achieve a uniform thickness of 1.2 mm (Fig. 1) with the help of straight diamond fissure bur of 2 mm diameter (ISO shape 152, Bestell-Nr, Order-No. 531535C, DFS, Landenstrabe, Riedenburg). The defect in the Group II (n = 60) discs were created until metal was exposed in 1/4th of the total area of the sample (Fig. 2).

The samples of ceramic substrate (Group I) and metal substrate (Group II) were further subdivided into A, B containing thirty samples each, according to the surface treatments (A; roughening with diamond bur, B; abraded with 50 μm Al₂O₃). The surface of Group IA, IIA samples were pretreated with sintered diamond bur with low speed rotary instrument and were cleaned with ultrasonic steamer whereas, the surface of Group IB, IIB samples were abraded with 50 μm aluminum oxide (Danville Engineering, San Ramon, CA, USA) using Microetcher ERC intraoral sandblaster (Danville Engineering, San Ramon, CA, USA).
for 10 s with a pressure of 35–60 psi at a distance of 10.0 mm from the sample surface and were cleaned with ultrasonic steamer. After surface treatment, all the samples were repaired with one of the respected intraoral repair systems (a. Ceramic repair system, Ivoclar Vivadent; b. Clearfil repair system, Kurary; c. Porcelain repair system, 3M ESPE) as per manufacturer’s instructions (Table 1).

The defective ceramic margins were beveled at 45 degree of 1–2 mm width with a flame shaped diamond bur under water irrigation. Subsequently, the area was dried with oil-free compressed air.

A 37 % phosphoric acid etchant was applied to all the groups’ samples except subgroup b (Clearfil repair system) in which 40 % phosphoric acid etchant was used, according to manufacturer instruction and then cleaned with water and dried with oil-free compressed air. After post etching, ceramic primer was applied only in Group II (metal substrate) samples, in which metal surface were exposed and left for 180 s. Then, silane coupling agents was applied in all the samples according to manufacturer instruction. After silane application, opaque layer of 0.3 mm thickness was applied on the conditioned metal surface of group II samples. Subsequently, samples were cured for 20 s.

The bonding agent was applied to each of the samples and polymerized with a light curing unit (QHL75 curing light, Dentsply) for 20 s. Then, the defect area was repaired with a layer of ceramic composites (Nano-hybrid composites) of thickness 1.2 mm in Group I samples and 1.5 mm layer in Group II samples using custom made jig and cured for 40 s, as per the manufacturer’s instructions.

All the repaired samples were then stored in distilled water for 24 h before thermocycling. Thermocycling was done 6–60 °C for 500 cycles with a 30-second dwell time. After thermocycling, the samples were stored in distilled water for additional 7 days. Shear bond strength testing was performed using Universal testing machine (WDW-SE, Serial—20070802, Times Shijin Group), with a 10-kN load cell and 0.5 mm/min crosshead speed. A chisel load applicator was used to direct a parallel shearing force as close as possible to the composite/defect substrate interface. The shear bond strength values were recorded in MPa.

Each sample was examined visually and mode of failure 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). Data were analyzed using one-way ANOVA test followed with Post-Hoc test by Bonferroni method.

**Results**

Tables 2 and 3 shows the ANOVA tests performed for ceramic and metal groups, respectively. Tables 4 and 5 present the shear bond strength mean values for ceramic and metal groups, respectively. The mean shear bond strength values for Ceramic repair system (9.47 ± 1.41 MPa), Clearfil repair system (14.03 ± 2.32 MPa), and Porcelain repair system (14.41 ± 3.96 MPa) were observed for ceramic substrate with roughening with diamond bur as a surface treatment (Table 4). However, when the ceramic surface was abraded with air abrasion (50 micron aluminum oxide particles), the mean shear bond strength of Ceramic repair system (14.03 ± 2.54 MPa), Clearfil repair system (14.64 ± 2.28 MPa), and Porcelain repair system (14.86 ± 3.10 MPa) were observed (Table 4). This result shows that the shear bond strength values for Ceramic repair system with air abrasion is statistically highly significant then roughening with diamond bur as a surface pretreatment. Whereas, the shear bond strength values for Clearfil repair system and Porcelain repair system with air abrasion and roughening with diamond bur were statistically insignificant, although the mean shear bond strength values with air abrasion obtained is higher.

The mean shear bond strength values for the metal substrate treated with diamond bur and repaired with three intraoral repair systems used in the study were: Ceramic repair system (9.42 ± 1.44 MPa), Clearfil repair system (14.44 ± 3.23 MPa), and Porcelain repair system (11.86 ± 2.24 MPa) (Table 5). Whereas, when metal surface was treated with air abrasion (50 micron aluminum oxide particles) and repaired with three intraoral repair system, the mean shear bond strength of Ceramic repair system (18.61 ± 2.60 MPa), Clearfil repair system (14.98 ± 2.73 MPa), and Porcelain repair system (13.24 ± 2.72 MPa) were observed (Table 5). These results shows that the shear bond strength values for Ceramic repair
The system with air abrasion were highly significant statistically as compared to roughening with diamond bur. The mode of failures of all the samples were examined by single observer using visual examination and classified as adhesive (failure at substrate-resin interface), cohesive (failure within the substrate or within restorative material) or combination (area of adhesive and cohesive failure). For ceramic substrates, all the three intraoral repair systems presented with 100 % cohesive failure. In ceramic substrate, ceramic repair system and porcelain repair system showed 100 % adhesive failure whereas, Clearfil repair systems showed 60 % cohesive, 30 % adhesive failure and 10 % combination.

### Table 1: Technique used for ceramic and metal specimens

| Repair system and manufacturer                                           | Sequence of material application (ceramic specimens)          | Sequence of material application (metal specimens)          |
|--------------------------------------------------------------------------|----------------------------------------------------------------|----------------------------------------------------------------|
| Ceramic repair system, Ivoclar Vivadent, Liechtenstein, Germany          | Total etch (37 % phosphoric acid)                              | Total etch (37 % phosphoric acid)                            |
|                                                                          | Monobond-S                                                      | Metal/zirconia primer                                        |
|                                                                          | Heliobond                                                      | Monobond-S                                                   |
|                                                                          | Tetritic EvoCeram                                               | Monopaque                                                    |
| Clearfil repair system, Kurary, Tokyo, Japan                              | K etchant Gel                                                  | K etchant Gel                                                |
|                                                                          | Clearfil SE bond primer                                         | Alloy primer                                                 |
|                                                                          | Clearfil porcelain bond                                         | Clearfil SE bond primer                                      |
|                                                                          | Activator                                                      | Clearfil porcelain bond activator                            |
|                                                                          | Clearfil SE bond bond                                          | Clearfil SE bond bond                                         |
|                                                                          | Clearfil APX composite                                          | Clearfil St Opaque                                           |
| Porcelain repair system, 3 M, ESPE, Seefeld, Germany                     | Scothbond etchant                                              | Scothbond etchant                                            |
|                                                                          | Rely X ceramic primer                                           | Rely X ceramic primer                                         |
|                                                                          | Single Bond 2                                                  | Single bond 2                                                 |
|                                                                          | Filtek Z350 composite                                           | Filtek Z350 opaquer                                           |

### Table 2: One way ANOVA results for ceramic groups

| Source of variance | Sum of squares | Df | Mean square | F   | P     |
|--------------------|----------------|----|-------------|-----|-------|
| Between groups     | 207.6          | 5  | 41.5        | 5.6 | 0.0001|
| Within groups      | 399.4          | 54 | 7.4         |     |       |
| Total              | 607.1          | 59 |             |     |       |

### Table 3: One way ANOVA results for metal groups

| Source of variance | Sum of squares | Df | Mean square | F   | P     |
|--------------------|----------------|----|-------------|-----|-------|
| Between groups     | 482.3          | 5  | 96.5        | 14.8| 0.0001|
| Within groups      | 353.2          | 54 | 6.5         |     |       |
| Total              | 835.5          | 59 |             |     |       |

### Discussion

Air abrasion with 50 µm aluminum oxide particles is more effective then roughening with diamond bur for enhancing shear bond strength of all the intraoral repair system to both metal and ceramic. Appeldoorn et al. [6], Thurmond et al. [7], Cobb et al. [8], Tulunoglu et al. [9] and, Petridis et al.[10], Ozcan et al. [11] who reported that higher bond strengths of intraoral repair systems to ceramic and metal were achieved with air abrasion (23.5 ± 5.3 MPa) than the roughening with diamond bur (12.0 ± 2.3 MPa). However, these results are in disagreement with the Suliman et al. [12], who stated that higher bond strength of intraoral repair systems were obtained with roughening with diamond bur and etching with hydrofluoric acid (16.98 MPa) than air abrasion alone (16.86 MPa); although the values were statistically non-significant. Air abrasion with 50 µm aluminum oxide particles enhances the bond strength of intraoral repair systems as it promotes micromechanical retention by creating very fine obtuse angular roughness on the surface, thereby increasing the total surface area, decreasing the surface tension and enhancing wetting by the resin [13]. Whereas, roughening with diamond bur creates sharp surface irregularities and microcracks within the ceramic surface causing stress concentration and subsequent fracture. Air abrasion when used for metal substrate produces uniformly frosted surface having shallow interconnected furrows. These furrows draw primer and adhesive agents onto the abraded surface through capillary
Table 4 Mean (+SD) values of shear bond strength (MPa) for ceramic substrate

| Materials               | Mean values for intraoral repair systems with roughening with diamond bur (surface treatment) | Mean values for intraoral repair systems with air abrasion (surface treatment) |
|-------------------------|--------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------|
| Ceramic repair system   | 9.47 ± 1.41 MPa                                                                                   | 14.03 ± 2.54 MPa                                                             |
| Clearfil repair system  | 14.03 ± 2.32 MPa                                                                                   | 14.64 ± 2.28 MPa                                                             |
| Porcelain repair system | 14.41 ± 3.96 MPa                                                                                   | 14.86 ± 3.10 MPa                                                             |

Table 5 Mean (+SD) values of shear bond strength (MPa) for metal substrate

| Materials               | Mean values for intraoral repair systems with roughening with diamond bur (surface treatment) | Mean values for intraoral repair systems with air abrasion (surface treatment) |
|-------------------------|--------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------|
| Ceramic repair system   | 9.42 ± 1.44                                                                                     | 18.61 ± 2.60                                                                 |
| Clearfil repair system  | 14.44 ± 3.23                                                                                     | 14.98 ± 2.73                                                                 |
| Porcelain repair system | 11.86 ± 2.24                                                                                     | 13.24 ± 2.72                                                                 |

action enhancing the shear bond strength of intraoral repair systems to metal [14]. Thus it is evident from the above results that surface treatments of ceramic and metal substrate is one of the key factors for enhancing the shear bond strength of intraoral repair systems, besides the active role of other components of intraoral repair systems like metal primer, silane coupling agent, and restorative material.

The results of the study for three different intraroral ceramic repair systems suggests that, when repair was performed on ceramic with air abrasion as surface treatment, shear bond strength of Porcelain repair system (14.86 ± 3.10 MPa), Clearfil repair system (14.64 ± 2.28 MPa), and Ceramic repair system (14.03 ± 2.54 MPa) were not statistically significant. However, Porcelain repair system showed higher bond strength as compared to other repair systems for ceramic substrate. Tulunoglu et al. [9], Santos et al. [4] analyzed the shear bond strength of Porcelain repair system (18.04 ± 3.23 MPa) to ceramic was higher than Clearfil repair system (16.91 ± 2.22 MPa) however, the values were statistically insignificant. They also found that for bonding of composite resin to ceramic, silane coupling agent was the key link and also all the three systems used in this study contained silane coupling agent. Silane coupling agent contains 3-methacryloxypropyl-trimethosilane (MPS) which forms a dual covalent bond (hydrophobic siloxane bond) with organic (composite) and inorganic substances (ceramic), also promotes the wetting of ceramic surface and enhanced the flow of the low-viscosity resins thereby, improving the bond strength of intraoral repair systems to ceramic [15].

When the repair was performed for metal substrate, Ceramic repair system showed the highest bond strength (18.61 ± 2.60 MPa) which was significantly higher from Clearfil repair system (14.98 ± 2.73 MPa) and Porcelain repair system (13.24 ± 2.72 MPa). Tulunoglu et al. [9] and Santos et al. [4] analyzed the shear bond strength of Clearfil repair system (18.40 ± 2.88 MPa) to metal was although higher than Porcelain repair system (16.26 ± 3.09 MPa) but on statistical comparison the values were non-significant as evident in this study also. Ceramic repair system used in this study showed higher shear bond strength values to metal substrate after air abrasion as a surface treatment due to the presence of alloy primer containing MDP (10-methacryloyloxydecyl dihydrogen phosphate). MDP contains an ester phosphate group which forms a strong chemical bonding with oxide layer on the surface of the alloy for reliable bond of the resin to alloys [4]. However, Porcelain repair system used in this study did not have individual alloy primer containing MDP and hence showed the least shear bond strength values for metal substrate among the three intraoral repair systems studied.

The restorative component also plays a crucial role in ceramic and metal repair. Ceramic repair system consists of a nano-hybrid composite whereas the other repair systems contain micro-hybrid composites. The particle size is larger and filler content is lesser in nano-hybrid composites than the micro-hybrid composite which result in enhanced bond strength of Ceramic repair system to metal when air abrasion was used as a surface treatment [15].

On a comparative note of evaluation between repair to metal substrate and repair to ceramic substrate, the metal surface showed a prevalence of adhesive failure (except Clearfil repair system) which reflects that the bonding of these systems to a metal substrate was not as effective as the inherent strength of the repair materials. The repair to ceramic surface showed a prevalence of cohesive failure for all the three intraoral repair systems. This type of failure is also reported in the literature [4, 11]. This indicates that the bonding of these systems to ceramic was superior to the actual inherent strength of the repair materials.
Unfortunately, there are no reports on the minimum shear bond strength values required for metal-ceramic restoration repair materials. However, the ideal requirement of material should have a bond value similar to reported metal-ceramic bond strength (16–24 MPa) [16]. The average masticatory forces are reported to be between 20 and 830 N in the literature. The masticatory forces between the incisors vary between 155 and 222 N and are higher for molars up to 830 N [17]. Since, the strength is directly proportional to the masticatory forces and inversely proportional to area (Strength = F/A), it may be assumed minimum bond strength required for intraoral repair material is 8–9 MPa.

According to this study, the bond strength values obtained for the three intraoral repair systems were higher than above assumed bond strength value (8–9 MPa). This gives enough justification to recommend all three repair systems in conjunction with air abrasion as a surface pre-treatment used in this study for intraoral chairside repair of metal-ceramic restoration. Further, in vivo studies would definitely give more information and clearer understanding about the clinical performance of these systems.

**Conclusion**

Within the limitations of the study, following conclusions were drawn:

1. Combinations of mechanical and chemical retentive systems enhance the shear bond strength between intraoral repair materials and the surface of fracture metal-ceramic restoration.
2. Air abrasion with 50 μm aluminum oxide particles, when used as surface pretreatment increased the shear bond strength for both ceramic and metal substrate when repaired with three different intraoral repair systems used in the study.
3. When air abrasion was used as a surface treatment for ceramic substrate, Porcelain repair system showed the highest shear bond strength (14.41 ± 3.96 MPa) values as compared to other two systems used in the study, Clearfil repair system (14.03 ± 2.32 MPa) and Ceramic repair system (9.47 ± 1.41 MPa). Whereas, when air abrasion was used as a surface treatment for metal substrate, Ceramic repair system showed the highest bond strength (18.61 ± 2.60 MPa) as compared to two other systems, Clearfil repair system (14.98 ± 2.73 MPa) and Porcelain repair system (13.24 ± 2.72 MPa).
4. The alloy primer which containing MDP (10-methacryloyloxydecyl dihydrogen phosphate) is essential for bonding of composite resin to metal surface of metal-ceramic restorations.
5. Nano-hybrid composites show higher bond strength as compare to micro-hybrid composites when bonded to metal-ceramic restorations.

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