

Shear bond strength of ceramic brackets with various base designs bonded to aluminous and fluorapatite ceramics

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SUMMARY This study was conducted to evaluate the shear bond strength (SBS) of various ceramic bracket base designs bonded to glazed aluminous (Vitadur Alpha) and fluorapatite (IPS e.max Ceram) ceramics, to examine the mode of failure, and to determine the debonding characteristics of the brackets and the ceramic surfaces after bond failure.

Forty ceramic discs (15 mm in diameter and 1.5 mm thick) of each ceramic were prepared and divided into four equal groups. Ten pieces of each group of different bracket bases (beads, Inspire Ice; large round pits, Crystalline IV; and irregular base, Clarity) and one group of stainless steel brackets (Optimesh XRT, control) were bonded to glazed ceramics under a 200 g load. All specimens were then subjected to SBS evaluation using a universal testing machine at a crosshead speed of 0.2 mm per minute. The data were analysed using analysis of variance and Tukey's test at a significance level of 0.05. The mode of failure was examined under a stereomicroscope.

The results demonstrated that for Vitadur Alpha and IPS e.max Ceram, the highest SBS were found with Inspire Ice (25.1 ± 2.6 and 24.9 ± 2.1 MPa) and were significantly different than Crystalline IV (21.6 ± 1.1 and 20.9 ± 1.5 MPa), Clarity (19.6 ± 1.5 and 19.3 ± 2.3 MPa), and Optimesh XRT (14.9 ± 1.3 and 15.3 ± 2.2 MPa; $P < 0.05$). Inspire Ice and Crystalline IV had 100 per cent adhesive failure while Clarity and Optimesh XRT had combination failure. The various base designs gave different SBS, but the SBS of all base designs could withstand normal orthodontic force.

Introduction

Ceramic orthodontic brackets have been available for clinical use since 1987. They were designed to combine aesthetics with the reliability of stainless steel brackets (Birnie, 1990). All currently available ceramic orthodontic brackets are composed of aluminium oxides (Harris *et al.*, 1992; Karamouzos *et al.*, 1997) which have many advantages such as biocompatibility, good aesthetics, resistance to temperature and chemical changes, and good bond strength that is higher or equal to that of stainless steel brackets (Odegaard and Segner, 1988; Swartz, 1988; Flores *et al.*, 1990; Viazis *et al.*, 1990). There are two types of ceramic brackets which are classified according to their distinct differences during fabrication, namely, polycrystalline and monocrystalline (single crystal) aluminas (Bordeaux *et al.*, 1994; Bishara and Fehr, 1997; Gautam and Valiathan, 2007).

Polycrystalline aluminas are made of sintered or fused aluminium oxide particles. The aluminium oxide particles are blended with a binder and the mixture is formed into a shape from which a bracket can be machined. Temperatures above 1800°C are used to 'burn out' the binder and fuse the particles of the moulded mixture together. This firing process is called 'sintering'. They are then heat treated to remove surface imperfections and stresses created by the curing process. These slight imperfections and impurities

can serve as foci for crack propagation under stress and compromise a bracket during clinical use (Swartz, 1988).

Monocrystalline aluminas are also manufactured from aluminium oxides. Aluminium oxides are heat treated to temperatures in excess of 2100°C and then cooled slowly to permit complete crystallization. This process minimizes the stress-inducing impurities and imperfections found in polycrystalline aluminas (Swartz, 1988).

Both poly- and monocrystalline ceramic brackets come with various base designs such as beads, grooves, or round pits for the purpose of mechanical interlocking between the brackets and the teeth. In addition, they provide chemical bonding with silanes. Silanes (gamma-methacryloxypropyltrimethoxysilane) are coupling agents developed for bonding glass fillers into polymers, which increase the wettability of the ceramic surface (Bowen and Rodriguez, 1962). In most studies, silanes have been found to successfully increase the adhesion of the resin composite to the ceramic surface (Newman *et al.*, 1984; Kao *et al.*, 1988; Lu *et al.*, 1992; Whitlock *et al.*, 1994; Major *et al.*, 1995; Kocadereli *et al.*, 2001; Harari *et al.*, 2003; Türkkahraman and Küçükeşümen, 2006). However, there are contradictory reports regarding the efficacy of silane-coupling treatment in the long-term adhesion between resin composite and ceramic (Bailey, 1989; Diaz-Arnold and Aquilino, 1989; Wolf *et al.*, 1992;

Ozcan *et al.*, 2008). The efficiency of silane-coupling agents can be influenced by several factors. Single-bottle products have a limited shelf life because of rapid solvent evaporation and hydrolyzation. Silanes might have different chemical structures; this makes it important to use one bonding system and not interchange components that might not be compatible (Blatz *et al.*, 2003). Another factor of concern is that all silanes are sensitive to humidity. In humid conditions, silanized interfaces seem to be unstable; the silane bond was found to deteriorate under atmospheric moisture (Nergiz *et al.*, 2000). Additionally, it is recommended that only fresh silanes are used since aged silanes can compromise bond strength (Robbins, 1998).

Etching with hydrofluoric acid is widely recommended and used for ceramic surface modification which shows strong bond strengths (Zachrisson and Buyukyilmaz, 1993; Barbosa *et al.*, 1995). However, it is considered a hazardous agent which can produce a tissue rash, burns, and deep tissue necrosis (Moore and Manor, 1982). During intraoral use of hydrofluoric acid, special precautions should be used. Unlike phosphoric acid, at 37 per cent concentration, it is not toxic or corrosive and results in satisfactory bond strength (Bourke and Rock, 1999).

A more demanding sense of aesthetics has led to an increase in adults requesting orthodontic treatment. Thus, the orthodontist frequently encounters all-ceramic restorations, which are gaining popularity because of their superior biocompatibility and aesthetic appeal (Albakry *et al.*, 2004). These ceramics may be aluminous or fluorapatite. The aluminous ceramic (Vitadur Alpha, Vita Zahnfabrik, Bad Sackingen, Germany) is composed of glass powder and fused alumina crystals, which constitute up to 50 per cent by weight (McLean and Hughes, 1965). The fluorapatite ceramic (IPS e.max Ceram, Ivoclar Vivadent AG, Schaan, Liechtenstein) is used as the veneering ceramic of this system. This is a feldspathic-based ceramic with a microstructure dissimilar to IPS d.SIGN. This glass-ceramic consists of dispersed fluorapatite crystals in a feldspathic glassy matrix. Fluorapatite crystals, 2–5 µm in length and 300 nm in diameter of needle-like morphology, are known to be contained in natural bone and teeth. The very small crystals in dental microstructures result in optical properties such as translucence and opalescence, which is also seen in dental restorations (Holand *et al.*, 2003).

Orthodontic brackets may be bonded to ceramic restorations. Optimal bracket adhesion to a ceramic surface requires that the orthodontic forces be applied without bond failure during treatment and that the ceramic integrity is not jeopardized during debonding. Bond strengths between 6 and 8 MPa are clinically sufficient for successful bonding of brackets to enamel (Reynolds, 1975; Whitlock *et al.*, 1994). Unfortunately, little is known about the bond strength of various ceramic brackets base designs bonded to all-ceramic restorations. Therefore, the objectives of this study were (1)

to evaluate the shear bond strength (SBS) of various ceramic brackets base designs bonded to glazed aluminous and fluorapatite ceramics, (2) to examine the mode of failure of the various bracket base designs and of both ceramics, and (3) to determine the debonding characteristics of the brackets and the ceramic surfaces after bond failure.

Materials and methods

Forty samples of glazed aluminous and fluorapatite ceramic discs were produced according to the manufacturers' instructions. Aluminous (Vitadur Alpha, Vita Zahnfabrik) and fluorapatite (IPS e.max Ceram, Ivoclar Vivadent AG) ceramics are used as veneering ceramics for Vita In-Ceram and the IPS e.max System, respectively. Ceramic powders were mixed with deionized water and condensed into a round shape silicone mould (Provil, Haraeus Kulzer, Wehrheim, Germany), 15 mm in diameter and 1.5 mm thick. The specimens were then fired according to the manufacturers' instructions (Table 1). After firing, sintered ceramic discs with a final diameter of 13.45–14.12 mm (5.87–10.33 per cent shrinkage) were polished (Phoenix 4000, Buehler GmbH, Düsseldorf, Germany) under running water using 600 and 1200 grit silicon carbide paper (3M Espe, St Paul, Minnesota, USA). The specimens were then cleaned in an ultrasonic cleanser for 10 minutes. Finally, the specimens were submitted to self-glazing according to the manufacturers' instructions (Table 1).

Subsequently, the discs were embedded in autopolymerizing clear acrylic resin (Takilon, Rodont srl, Milan, Italy), 20 mm in height and 30 mm in diameter. The specimens for each ceramic were randomly divided into four groups of 10 for bonding with three groups of ceramic brackets which had various base designs (beads, Inspire Ice; large round pits, Crystalline IV; and irregular, Clarity). Stainless steel brackets (Optimesh XRT) served as the control (Figure 1 and Table 2).

The ceramic surfaces were etched with 37 per cent phosphoric acid, (Ormco/Sybron Dental Specialties, Glendora, California, USA) for 60 seconds, and a thin coat of porcelain primer, (Ormco/Sybron) was applied twice with a microbrush for 10 and 60 seconds, respectively. The discs were then rinsed with a water spray for 15 seconds and

Table 1 Firing schedules for the ceramics used in the present study.

| Ceramic | Type of firing | Starting temperature (°C) | Heating rate (°C/min) | Firing temperature (°C) | Holding time (min) |
|-----------------|----------------|---------------------------|-----------------------|-------------------------|--------------------|
| Vitadur Alpha | Dentine | 600 | 60 | 960 | 1 |
| | Glaze | 600 | 85 | 940 | 1 |
| IPS e.max Ceram | Dentine | 403 | 50 | 850 | 0 |
| | Glaze | 403 | 50 | 800 | 0 |

thoroughly air-dried. System 1+ liquid activator (Ormco/Sybron) was then applied to both the ceramic surfaces and the bracket bases, and System 1+ paste (Ormco/Sybron) was applied to the activated bracket bases. The brackets were then positioned on the ceramic discs and 200 g of

pressure was applied to the brackets. Excess adhesive was carefully removed from the bracket base with a sharp scaler and allowed to completely polymerize for 10 minutes. Finally, all specimens were stored in an incubator (Memmert, model BE500, Memmert GmbH, Schwabach, Germany) at 37°C and 100 per cent humidity for 24 hours before testing.

SBS testing of the ceramic brackets on the ceramic specimens was performed using a single-bladed Instron machine (model 5583, Instron, Norwood, Massachusetts, USA) at a crosshead speed of 0.2 mm/minute. The load at failure was recorded in newtons and converted to megapascals to determine SBS (force per surface area of the bracket base). Bracket bond area was determined by measuring the width and length of the bracket base with a digital calliper (Mitutoyo, Tokyo, Japan) and the area calculated (Table 2). After debonding, the surfaces of the specimens were examined by one observer (BK) under a stereomicroscope (SMZ 1500m, Nikon Instech, Kanagawa, Japan) to determine the mode of failure. For determination of the mode of failure, each sample was recorded according to a modification of the method of Bordeaux *et al.* (1994; Table 3).

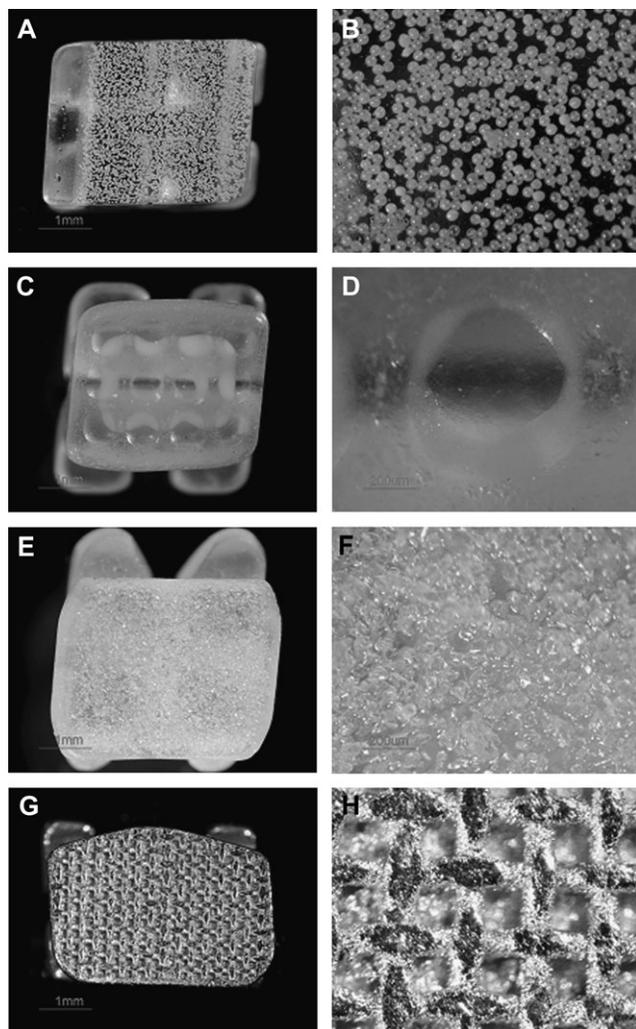


Figure 1 Stereophotomicrographs of base designs of ceramic bracket ($\times 20$ magnification, bar = 1 mm and $\times 100$ magnification, bar = 200 μm , respectively). (A) and (B), beads base (Inspire Ice); (C) and (D), large round pits base (Crystalline IV); (E) and (F), irregular base (Clarity); (G) and (H), mesh base (Optimesh XRT).

Statistical analysis

The data were statistically analysed using the Statistical Package for Social Sciences version 16.0 (SPSS Inc., Chicago, Illinois, USA). A two-way analysis of variance (ANOVA) was performed to assess the influence of the different ceramic brackets and the ceramics on SBS. A one-way ANOVA was used to determine differences between the groups. Tukey’s Honestly Significant Differences (HSD) tests were used for *post hoc* comparisons ($\alpha = 0.05$).

Results

Table 4 presents the results of the two-way ANOVA, which revealed statistical differences among the different types of ceramic brackets ($P = 0.01$). However, there were no statistical differences between the different types of ceramics and the interaction between the type of ceramic brackets and the ceramics ($P = 0.57$ and $P = 0.83$, respectively). Therefore, the different types of ceramics did not affect the SBS values.

Table 2 Identification of ceramic brackets used in the present study.

| Name of ceramic bracket | Manufacturer | Type | Base design | Area of surface (mm ²) |
|-------------------------|--------------------------------------|-------------------------|-----------------|------------------------------------|
| Inspire Ice | Ormco/Sybron Dental Specialties | Monocrystalline alumina | Bead | 11.50 |
| Crystalline IV | Tomy, Tokyo, Japan | Polycrystalline alumina | Large round pit | 10.05 |
| Clarity | 3M Unitek, Monrovia, California, USA | Polycrystalline alumina | Irregular | 10.55 |
| Optimesh XRT | Ormco/Sybron Dental Specialties | Stainless steel | Mesh | 11.71 |

The mean SBS values of the ceramic brackets to Vitadur Alpha and IPS e.max Ceram at fracture are presented in Figure 2. For Vitadur Alpha, one-way ANOVA and Tukey's HSD showed a significant difference among the groups ($P = 0.01$). The control group (Optimesh XRT) yielded the lowest mean SBS and standard deviation (SD) values (14.9 ± 1.3 MPa; $P = 0.01$). Inspire Ice produced the highest mean SBS values (25.1 ± 2.6 MPa; $P = 0.01$). There was no significant difference between Crystalline IV (21.6 ± 1.1 MPa) and Clarity (19.6 ± 1.5 MPa; $P = 0.13$). Similar to IPS e.max Ceram, ANOVA and Tukey's HSD showed a significant difference among the groups ($P = 0.01$). Inspire Ice produced the highest mean SBS values (24.9 ± 2.1 MPa; $P = 0.01$) while Optimesh XRT yielded the lowest mean SBS and SD values (15.3 ± 2.2 MPa; $P = 0.01$). The SBS values between Crystalline IV (20.9 ± 1.5 MPa) and Clarity (19.3 ± 2.3 MPa) were not significantly different ($P = 0.14$).

Table 5 and Figure 3 show the predominant site of bond failure after examination of the debonded surface using a stereomicroscope. None of the specimens evaluated in this study was found to display any cracks or fractures of the brackets or ceramic surfaces (type 4 and 5).

Table 3 Type of mode of failure and characteristics [after Bordeaux *et al.* (1994)].

| Type | Characteristics |
|------|--|
| 1 | Failure at the adhesive–bracket base interface. Ninety per cent or greater of the bracket pad exposed and 10 per cent or less of the bonded ceramic free of adhesive. |
| 2 | Combination failure at the adhesive–bracket base interface and the ceramic–adhesive interface. Less than 90 per cent but more than 10 per cent of the bracket pad exposed or more than 10 per cent but less than 90 per cent of the bonded ceramic surface free of adhesive. |
| 3 | Failure at the ceramic–adhesive interface. Ten per cent or less of the bracket pad exposed and 90 per cent or more of the bonded ceramic free of adhesive. |
| 4 | Failure of the bracket itself. Fracture of the bracket during removal with a portion of the bracket still bonded to the ceramic |
| 5 | Failure of the ceramic itself. A portion of the ceramic removed with the bracket base without loss of more than 10 per cent of the adhesive from the bracket pad. |

Table 4 Results of two-way analysis of variance.

| Source | Type III sum of squares | df | Mean square | <i>F</i> | <i>P</i> value |
|---|-------------------------|----|-------------|----------|----------------|
| Corrected model | 1020.17 | 7 | 145.74 | 40.44 | 0.01 |
| Intercept | 32689.56 | 1 | 32689.56 | 9071.01 | 0.01 |
| Type of ceramic | 1.18 | 1 | 1.18 | 0.33 | 0.57 |
| Type of bracket | 1015.85 | 3 | 338.62 | 93.96 | 0.01 |
| Interaction between type of ceramic and type of bracket | 3.14 | 3 | 1.05 | 0.29 | 0.83 |
| Error | 259.48 | 72 | 3.61 | | |
| Total | 33969.21 | 80 | | | |
| Corrected total | 1279.66 | 79 | | | |

Discussion

The present study showed that the SBS of ceramic brackets bonded to either aluminous (Vitadur Alpha) or fluorapatite (IPS e.max Ceram) ceramic was greatly affected by base design but not by the type of ceramic. For Vitadur Alpha and IPS e.max Ceram, Inspire Ice resulted in the highest SBS, followed by Crystalline IV and Clarity. Optimesh XRT showed the lowest SBS in agreement with previous findings (Odegard and Segner, 1988; Swartz, 1988; Flores *et al.*, 1990; Viazis *et al.*, 1990).

The characteristics of various base designs were the reason for the results for both ceramics. Base design with irregular shapes incorporate small glass particles fused to the polycrystalline alumina to increase the surface area for adequate bonding. However, these glass particles might not have adequately adhered to the alumina base or there might be inadequate mechanical retention of the adhesive resin to penetrate to the rough base surface (Solderquist *et al.*, 2006). Similarly, large round pit base designs, having about 12 pits of 1 mm diameter in one bracket surrounded by a flat surface (Figure 1C,D), did not have any undercut for mechanical interlocking of adhesive resin. These results were confirmed by the type 1 bond failure (adhesive–bracket failure). Thus, the SBS of irregular and large round pit base designs showed no significant difference. Conversely, the bead base surface had as many as 50 μ m round monocrystalline beads completely distributed on the base surface (Figure 1A,B). These beads have undercuts for mechanical interlocking of adhesive resin resulting in the statistically highest SBS among all groups of both ceramics.

However, resin thickness and inherent flaws or defects in brackets or ceramics would influence bond strength. In this study, an attempt was made to control these factors. The ceramic brackets were bonded under pressure for the best fit on the ceramic surfaces and to minimize the thickness of the adhesive layers which may result in more imperfections, greater variability in the amount of polymerization obtained, and fracture (Whitlock *et al.*, 1994). No bracket or ceramic fractures were found in the present study; therefore, the inherent flaws did not affect SBS.

The maximum bond strength of ceramic brackets bonded to ceramics which may be achieved is usually not required for orthodontic purposes. The ideal bond strength should be sufficiently strong to endure a course of orthodontic treatment, yet sufficiently weak to permit adhesive removal from the ceramic surface following bracket removal. Reynolds (1975) recommended a tensile force of 60–80 kg/cm² and Whitlock *et al.* (1994), based on the work of Reynolds (1975), also suggested that 6–8 MPa was adequate for orthodontic attachments. In the present study, the SBS of all groups of both ceramics exhibited higher values than the minimum orthodontic bracket bond strength and therefore could be considered sufficient for clinical application.

Glazed ceramic surfaces are not amenable to resin penetration for orthodontic bonding (Lu *et al.*, 1992). Glazed surface removal has been advocated to create mechanical retention for adhesive resin by surface roughening (Hulterström and Bergman, 1993). However, the aesthetic and structural qualities of the ceramic may be irretrievably lost with surface roughening. The glaze is effective in strengthening the ceramic and reducing crack propagation. When the ceramic restoration is heated, the

self-glaze layer fills in surface flaws, reducing their depth and blunting the flaw tips. This should increase their strength because, for given ceramics, strength increases with decreasing sharpness and flaw depth (Griggs *et al.*, 1996). If the glaze is removed by grinding, the flexural strength of the ceramic unit may be reduced. For safety reasons several studies have recommended not removing the glaze by grinding (Kao *et al.*, 1988; Lu *et al.*, 1992; Zelos *et al.*, 1994). That recommendation is confirmed by the results of the present investigation. Even though this study used ceramic brackets bonded with the glazed ceramic surfaces, high SBSs occurred.

The high SBS of ceramic orthodontic brackets bonded to glazed ceramic in this research may also be a result of phosphoric acids and silanes. Phosphoric acid, at 37 per cent concentration, does not etch ceramic and does not produce physical or topographical changes in the ceramic surface. Instead, the effect of phosphoric acid is to neutralize the alkalinity of the adsorbed water layer, which is present on all-ceramic restorations in the oral cavity and thereby enhance the chemical activity of subsequently applied silanes (Wolf *et al.*, 1993). Silane-coupling agents act as a chemical link between the inorganic ceramic surface and the organic resin adhesive agent (Lu *et al.*, 1992; Major *et al.*, 1995). The findings of the present research confirm the necessity of using silanes, which correspond with the results of other studies (Newman *et al.*, 1984; Kao *et al.*, 1988; Lu *et al.*, 1992; Whitlock *et al.*, 1994; Major *et al.*, 1995; Kocadereli *et al.*, 2001; Harari *et al.*, 2003; Türk kahraman and Küçükşümen, 2006). Clinically, for bonding ceramic brackets to aluminous and fluorapatite ceramics, data from the present study indicate preserving the glaze, treating the porcelain with 37 per cent phosphoric acid, applying a porcelain primer, and using either type of ceramic bracket with adhesive resin.

The results of the stereomicroscope examination showed no damage to the ceramic surfaces in any group. It has been reported that if the bond between ceramic and adhesive resin is higher than 13 MPa, the ceramic will fracture

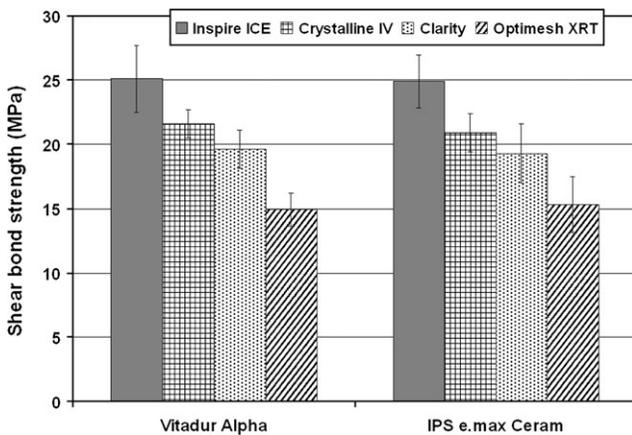


Figure 2 Mean shear bond strength (MPa) and standard deviation among various base designs of ceramic brackets.

Table 5 Mode of failure of both ceramics after shear bond strength testing.

| Ceramic | Ceramic bracket (manufacturer) | Type of failure | | | | | Total |
|-----------------|--------------------------------|-----------------|----|----|---|---|-------|
| | | 1 | 2 | 3 | 4 | 5 | |
| Vitadur Alpha | Inspire Ice (Ormco/Sybron) | 0 | 0 | 10 | 0 | 0 | 10 |
| | Crystalline IV (Tomy) | 10 | 0 | 0 | 0 | 0 | 10 |
| | Clarity (3M Unitek) | 0 | 6 | 4 | 0 | 0 | 10 |
| | Optimesh XRT (Ormco/Sybron) | 0 | 10 | 0 | 0 | 0 | 10 |
| IPS e.max Ceram | Inspire Ice (Ormco/Sybron) | 0 | 0 | 10 | 0 | 0 | 10 |
| | Crystalline IV (Tomy) | 10 | 0 | 0 | 0 | 0 | 10 |
| | Clarity (3M Unitek) | 0 | 5 | 5 | 0 | 0 | 10 |
| | Optimesh XRT (Ormco/Sybron) | 0 | 10 | 0 | 0 | 0 | 10 |

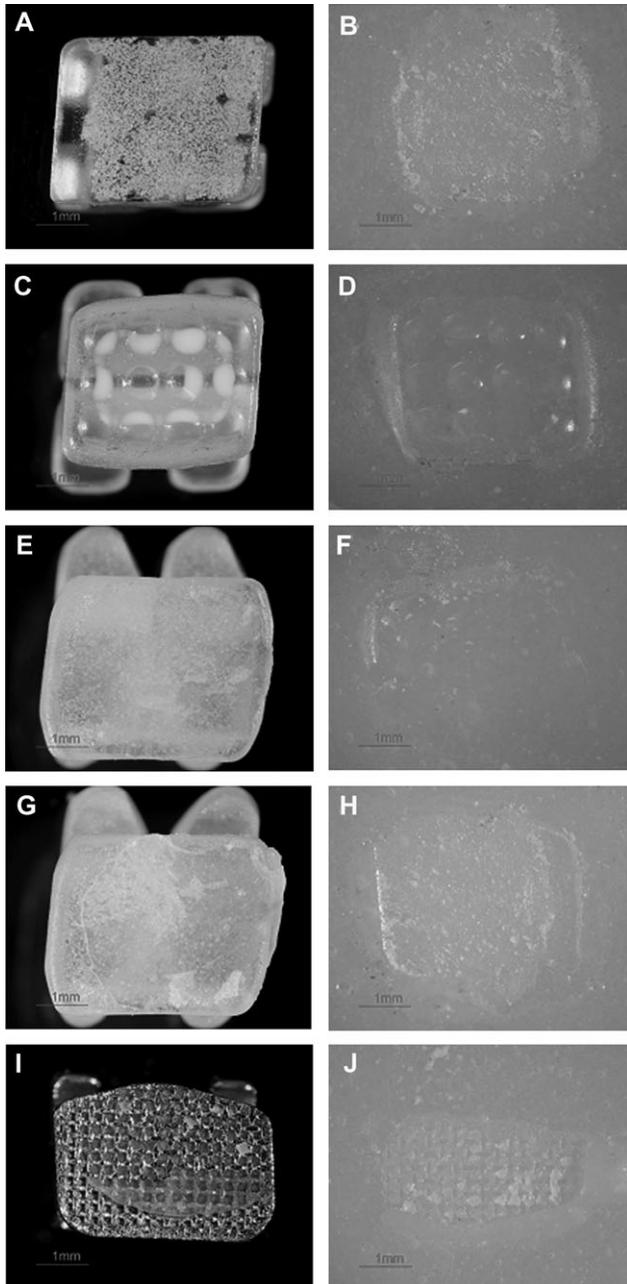


Figure 3 Stereophotomicrographs of failure characteristics (at the ceramic bracket base and ceramic, respectively). (A) and (B), type 3 failure of bead base; (C) and (D), type 1 failure of large round pit base; (E) and (F), type 3 failure of irregular base; (G) and (H), type 2 failure of irregular base; (I) and (J), type 2 failure of mesh base ($\times 20$ magnification, bar = 1 mm).

(Thurmond *et al.*, 1994). In this study, all groups of both ceramics achieved values higher than 13 MPa, which resulted in adhesive failures (type 1–3). No ceramic fractures were observed. This observation is important because bonding and debonding should not cause damage to the ceramic surfaces, which will affect the aesthetics and strength of the restoration.

The most significant finding in this study was that bonding of various ceramic bracket base designs to aluminous and fluorapatite ceramics resulted in high SBS in all groups. However, an *in vitro* study cannot replicate the same environment as the oral cavity. The presence of water, proteins, minerals, differences in pH levels, and temperature changes can affect the bond strength of ceramic brackets to ceramics. In addition, the present study demonstrated the results on a variety of ceramics and one type of adhesive bonding (Vitadur Alpha, IPS e.max Ceram, and System 1+). Therefore, it should not be presumed that other types of ceramic or adhesive will demonstrate the same pattern of bond strength. Further studies are required.

Conclusions

Within the limitations of this *in vitro* study, the following conclusions were drawn.

1. Bead base ceramic brackets and the glazed aluminous and fluorapatite ceramics yielded the statistically highest SBS among all groups.
2. The SBS of all groups exhibited higher values than the minimum orthodontic bracket bond strength range of 6–8 MPa.
3. Debonding characteristics showed no damage to either ceramic surface.

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