Effect of Veneering Dental Zirconia on Bond Strength.

A pilot study

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ABSTRACT

The objective of the study was to evaluate the effect of veneering zirconia surfaces with feldspar-based ceramics or glass ceramics on bond strength when using a resin composite cement. Forty-eight ytteria tetragonal zirconia polycrystal (Y-TZP) specimens were randomly divided into four groups. Six pairs were created in each group by bonding two of the specimens to each other after having the bonded surfaces treated differently as the following: I) polished and heat-treated (served as control), II) veneered with Vita VM9, III) veneered with e.max CAD Glaze and, IV) polished and heat-treated. The luting agent used in Group I - III was RelyX Unicem and in Group IV Charisma. Using a universal testing machine, the load at debonding (Newton) was recorded and the bond strength (MPa) was then calculated. Group I exhibited statistical significantly superior bond strength compared to all other groups. A statistical significantly superior bond strength was seen for the specimens in Group II compared to Group III and IV. Group IV presented the lowest bond strength but there was no significant difference to Group III. The present paper show no promotion in bond strength when zirconia was veneered with feldspar-based ceramic or glass ceramic.

INTRODUCTION

For the past three decades, all-ceramic resin-bonded fixed dental prosthesis (RBFDP) has been known as an appropriate solution to fill tooth gaps, mainly for the anterior part of the mouth (Kern, 2005). This could be a timesaving method considering that it is simple with no or minimal tooth preparation (Botelho *et al.*, 2014). Two main types belong to this kind of constructions. One type is Rochette, which is provided with holes and is based on the macro-mechanic bonding between the framework and the abutment teeth (Ziada e*t al.,* 2000). The other type is Maryland in which the surface is treated by, among others, sand blasting or chemical etching which allows a micro-mechanic bonding to take place (Imberg *et al.*, 1992). One study showed no significant difference in failure rates when comparing the two named types of RBFDPs, whereas another study presented a higher failure rate for Rochette (Ziada *et al.*, 2000). Such constructions can have either single- or double retainer abutment teeth with or without statistical significant differences in survival rate depending on the different criteria evaluated in the studies (Kern, 2005; Kern and Sasse, 2011).

Different materials have been used to modify RBFDPs such as metals, ceramics or a combination of them (Wyatt, 2007; Rosentritt *et al.*, 2009; Nakamura and Matsumura, 2013). Ceramic in general is used more often nowadays in dentistry because of its aesthetic aspect and the physical properties that are comparable with those properties that metals and other dental materials can offer (Wang *et al.*, 2014). Moreover, it is biocompatible and has shown its ability to integrate with bone (Wenz *et al.*, 2008; Gahlert *et al.*, 2009; Assal, 2013).

Zirconia is among the most promising types of ceramic at the meanwhile due to its higher flexural strength and fracture toughness compared to other dental ceramics (Guazzato *et al.*, 2004). This material is polymorphic and metastable and therefore is available in three distinct phases across different temperature ranges; monoclinic phase (room temperature) which is stable up to 1170 ^{O}C, tetragonal phase (1170-2370 ^{O}C) and cubic phase (> 2370 ^{O}C) (Piconi and Maccauro, 1999). Restorations made of zirconia should remain in the tetragonal phase even at room temperature to prevent collapsing during transformation to the monoclinic phase. To ensure this, different metal oxides are used such as; Y_2O_3, MgO, CaO (Karakoca and Yilmaz, 2009).

One of the most popular dental zirconia ceramic used in dentistry is stabilized with 5.15 wt% Y_2O_3 and is named ytteria tetragonal zirconia polycrystal (Y-TZP). What makes this material more exceptional is the unique self-healing process which occurs during exposure to any unfavorable force leading to inner cracks (Karakoca and Yilmaz, 2009). This suggests that zirconia can utilize the external energy to transform its crystals from the tetragonal to monoclinic phase, which is about 3-4% larger in volume, thus shuts the newly formed cracks and prevents further crack propagation (Karakoca and Yilmaz, 2009).

Feldspar-based ceramics and glass ceramics contain glass matrix and have better aesthetics compared to other ceramics. In addition, their huge content of silicon dioxide (SiO_2) allows them to be etched with hydrofluoric acid (HF-acid) and adhesively bonded to the tooth with resin composite cement (Sriamporn *et al.,* 2014). These materials can also be burned on oxide ceramics to achieve better aesthetics (Sundh and Sjögren, 2006).

Debonding remains a problem that is currently challenging the usage of RBFDPs which makes this method still to be considered less favorable than the conventional fixed partial dentures (FPDs) and gives it a lower survival rate (Wyatt, 2007). Different approaches are considered to improve the efficiency of this method such as; carefull abutment selection, optimal tooth preparation and definitely choosing an adequate bonding technique (Wyatt, 2007). Since Y-TZP is an oxide ceramic, etching technique by applying a strong acid such as H_2F_2 is still challenging (Thompson *et al.*, 2011). Based on the facts given above, the chance of reaching an acceptable bonding technique among all-ceramic RBFDPs and teeth is scarce. The purpose of the present study was, therefore, to investigate the effect on the resin composite bond strength when veneering zirconia (Y-TZP) surfaces with feldspar-based ceramic or glass ceramic. The hypothesis was that the bond strength is improved by covering the bonding surface of zirconia with a feldspar-based or glass ceramic.

MATERIALS & METHODS

Preparation of Specimens

Forty-eight square-shaped specimens of hot isostatic pressed (HIPed) Y-TZP (Cad.esthetics AB, Skellefteå, Sweden) were selected out of 150 specimens. The size of 24 specimens was 14 x 14 x 5 mm and for the remaining 24 specimens 14 x14 x 3 mm. To ensure that the surface of the specimens intended to be luted was similar pretreated at the beginning of the study, one of the surfaces of each specimen was wet ground for 2 minutes using a grinder (Struers Tech. A/S, Rødovre, Denmark) and a silicon carbide grinding paper (grit number P 400) (Buehler Ltd, Lake Bluff, IL, USA). The square-shaped specimens were then ultrasonically cleaned (Bransonic B221, Bransonic Ultrasonic Co, Danbury, Conn., USA) for 30 minutes in distilled water. Thereafter, the specimens were wet ground again for 2 minutes using the same grinder (Struers Tech) and a silicon carbide grinding paper (grit number P 1200) (Buehler Ltd), followed by ultrasonic cleaning (Bransonic B221) for 30 minutes in distilled water. The specimens were then cleaned with 95% alcohol and dried in air.

The specimens were then heat-treated in a way similar to when veneering zirconia with a feldspar-based ceramic in a heating oven (Dekema Austromat D4 Dekema Dental-Keramiköfen GmbH, Freilassing, Germany). They were then divided into four different groups with each group containing 12 specimens. Six pairs were created in each group by bonding two of the specimens to each other (Fig. 1).

The first group (Group I) served as control with no other treatment but cemented directly using a resin composite cement (RelyX Unicem Aplicap, 3M ESPE Dental Products, St. Paul MN, USA. Batch number 524549). For the second group (Group II), the wet ground surface on each specimen was veneered manually with a thin layer of a feldspar-based ceramic (Vita VM9, Vita Zahnfabrik, Bad Säckingen, Germany). Two persons carried out the veneering process, six specimens each. The veneered surface was then etched with 9.5% HF-acid for 20 seconds (Ultradent Porcelain Etch, Ultradent Products Inc., South Jordan, Utah, USA. Batch no B3K28), silanated with a silane agent (Ultradent Products Inc., South Jordan, Utah, USA. Batch number B6ZQD) and finally cemented with the same cement used in Group I. In the third group (Group III), the wet ground surface on each specimen was veneered with a glass-ceramic (e.max CAD Glaze, Ivoclar Vivadent, Lichtenstein. Batch number N 74262), then etched, silanated and cemented using similar procedure as in Group II. The last group (Group IV) was just cemented with a light curing hybrid resin composite filling material (Charisma, Hereaus Kulzer GmbH, Hanau, Germany. Batch number 010535) and OptiBond as bonding agent (Kerr, Biogross, Switzerland. Batch number 4789811) following similar procedure as in Group I.

Cementing Procedure

The RelyX Unicem resin composite cement was mixed for 15 seconds using an amalgam vibrator (Duomat, Degussa, Germany) and polymerized by means of a halogen lamp (Norlite, Germany) on each side for 20 seconds. The Charisma resin composite was delivered in capsules and polymerized by means of the halogen lamp (Norlite, Germany). For a better simulation of the oral environment and to standardize the polymerization conditions of the resin composite materials, all specimens were then stored for 24 hours in water at 36 ^{O}C in the dark.

To standardize the area luted and the load applied on the specimens, a mold of a vinyl polysiloxane impression material (Provil Novo Putty Regular Set, Hereaus Kulzer GmbH, Hanau, Germany. Batch nr 390348) was used (Fig. 1). The specimen was placed in the mold and then the luting agent was placed on the surface intended to be the cemented area. Thereafter, the other specimen was placed onto the cemented area and subsequently loaded with 50 Newton for 5 minutes using a plunger (Fig. 1).

Methods of Measuring the Force at Debonding

Using a universal testing machine (Tinius Olsen H10K-T, Horsham, PA, USA), the load at debonding (Newton) was determined for all of the specimens groups. The attached specimens were placed so that one of the free sides was fixed in the machine, while the other free side was loaded with a plunger (Ø 15.8 mm) in a vertical direction (1mm/min) with a gradually increasing force until debonding (Fig. 2). Results of maximum loading force at debonding were recorded automatically by the machine and presented in Newton. A digital caliper (Solar Absolute Digimatic, Mitutoyo Corp, Kanagawa, Japan) was then used to determine the area covered with the cement and the bond strengths were given in MPa for each test. To examine the tested surfaces after fracture, a light microscope (Leitz UWM-Dig-S, Ernst Leitz GmbH, Wetzlar, Germany) was used.

Statistical Analysis

The values of the bond strength for the specimens in the four named groups were statistically analyzed using the Kruskal-Wallis test supplemented with Bonferroni at a significance level of $p < 0.05$.

Searching for Peer-reviewed References

The PubMed database was used to search for peer-reviewed articles in English applicable to serve the present study. Hence, the following MeSH-terms were used; bond strength, fixed partial denture, FPD, resin-bonded fixed dental prosthesis, RBFDP, zirconia.

Ethical Considerations

The current study was an *in vitro* study and no human or animal subjects were involved in the experiments. The Ethics Forum at the Department of Odontology, Umeå University, Sweden found that appropriate ethics considerations have been integrated into this project. In addition, all the materials used in the study are intended for clinical use.

The authors declare no commercial or financial interest related to the study.

RESULTS

The values of the bond strength (MPa) for the specimens studied are presented in Table 1 and Fig. 3. The results of the statistical analysis are presented in Fig. 3.

Among the investigated four groups, the highest mean bond strength (6.1± 0.9 MPa) was observed for the specimens which were only polished, heat-treated and cemented with RelyX Unicem, that is Group I (Table 1). Group I exhibited significantly higher mean bond strength compared to Group II (p=0.004), Group III (p=0.001) and Group IV (p=0.000) (Fig. 3). Group II, which was veneered with VM9 and cemented with RelyX Unicem, was significantly superior to Group III that was veneered with e.max CAD Glaze and cemented with the same resin composite cement (p=0.001) and to Group IV which was cemented with Charisma resin composite (p=0.009) (Fig. 3). Group IV, exhibited the lowest mean bond strength (0.7 ± 0.3 MPa) but there was no statistical significant difference (p=0.199) compared to the values of Group III (1.4 ± 1.1 MPa), (Table 1, Fig. 3).

The mean value of the cemented area of all the 24 pairs of specimens was 62.5 ± 6.7 mm^2. The cemented area of Group I was significantly superior (p<0.05) to that of Group II, whereas there were no statistical significant differences (p>0.05) between the other groups. No fracture in the veneering layers or in the HIPed Y-TZP specimens was observed in any of the performed tests during this study. For all the explored specimens, a mix of adhesive and cohesive fractures of the cement could be noticed when examining the debonded surfaces.

DISCUSSION

Since no improvment in bond strength was seen when veneering zirconia with feldspar-based ceramic or glas ceramic the hypothesis was rejected.

This study was aimed to examine the bond strength to RBFDPs made of zirconia. More specifically, studying the bonding part between zirconia constructions and resin composite cement and how to improve it. An accepted clinical bonding between teeth and resin composite cement can be acheived by etching the enamel, which creates a rough surface and hence, a micro-mechanical bonding (Miyazaki *et al.,* 2014). On the other hand, Y-TZP is acid resistant due to its polycrystalline structure and non-silica composition, thus conventional adhesive techniques used with silica-based ceramics should not work effectively with it (Thompson *et al.,* 2011). Veneering zirconia with feldspar-based ceramic or glass ceramic, that have a large proportion of silica, could be a promising solution to attain an adhesive bonding to zirconia constructions. This could be possible through etching such silica-based ceramics with HF-acid and adhesively cement them (Sriamporn *et al.,* 2014). According to the facts given above, it was decided to design the study as the following; two zirconia specimens with different surface treatments or resin composites (group I - IV) were bonded to each other instead of bonding one zirconia specimen to a tooth. This was supposed to give the study a higher validity as the aim was to examine just the zirconia-cement side of the bonding and not the one to the tooth. On the other hand, this might maintain a higher reliability of the study which made it more reproducible. It is easier to reexamine a number of standardized prefabricated zirconia specimens instead of reexamining teeth specimens with a large number of varieties in quality and quantity of teeth substance.

A universal testing machine was used in the present experiment, the attached pair of specimens were placed in it so that one side was fixed and the other one free for the load to be performed until debonding. The load was carried out by a plunger in a vertical direction with a gradually increasing force at the free specimen until debonding (Fig. 2). This type of load seems to be extremely violent and probably more similar to the one we have in the mouth. In a previously published study where different "glaze-on" techniques were used, a higher bond strength values were shown compared to the values obtained in the present study (Everson *et al.*, 2012). Since similar veneering materials were used in both studies, a possible explanation for the differences in results could be the different types of loading tests used. Unlike our study, Everson *et al.* (2012) used a so called "shear bond strenght test" with a shear flat blade which slided along the surface of the substrate (Everson *et al.*, 2012). The shear blade would debond the cemented specimens by direct contact whereby, the maximum load at debonding was automatically recorded. This kind of load seems to be less violant than the one used in the present study and therefore would probably give higher bond strengths.

Liu *et al.* (2014), tested the effect of a similar glazing technique as in the present study on the bond strength measured by a shear bond strength test (Liu *et al.*, 2014). In contrast with the results obtained in the present paper, it showed that this method promoted a stronger bonding with a significant difference when compared to no surface treatment (Liu *et al.*, 2014). Apart from the different kind of test used, they had the examined specimens stored at room temperature for 24 hours after the cementation process, unlike the present study where the cemented specimens were kept in water at 36 ^{O}C in the dark and for 24 hours. Furthermore, Liu *et al.* (2014), discussed the negative effect of water on siloxane bonds at the connected surfaces. This could motivate the impaired bond strength observed when specimens were stored in water after cementation as in the present study.

Since the present study was a pilot study, a few numbers of specimens was used. Therefore, the Kruskal-Wallis test supplemented with Bonferroni was selected for the statistical analysis after consulting an experienced statistician. Another finding in the current study concerned the distribution of the values of the load at debonding that increased when the zirconia surface was covered with feldspar-based ceramic (Fig. 3). One reason could be that there were more manual steps involved, which rendered more variations in the cementing process. In contrast, it was aimed to get the veneering processes done manually and by two different persons to check whether or not this could affect its quality and thus the bond strength. This resembles real life practice in which this process could be done in different dental laboratories and by different technicians with varied experiences. The results showed no fracture in veneering layers in all tests, but the debonded surfaces presented a mix of adhesive and cohesive fractures of the cement. It is difficult to draw a definite conclusion out of this; however, the manual veneering work could be the underlying cause of a different performance of the adhesive bonding. An even and thin veneering layer is almost impossible to achieve at all cases when it is performed manually. Consequently, the distribution of a sufficient thickness along the veneering layer could vary. A good adhesion could be obtained at some sites and thus a cohesive fracture when debonding, but not at the others where an adhesive fracture was seen.

To study whether RelyX Unicem, as a resin composite material, has any special effect to the zirconia surface, another resin composite filling material (Charisma) was used as a luting agent in the present study. Charisma is mainly recommended as dental filling material and exhibited significantly lower bond strength compared to Relyx Unicem. However, there was no significant difference between Charisma bonded to zirconia (Group IV) and RelyX Unicem bonded to zirconia specimens covered with glass ceramic (Group III). This indicates that RelyX Unicem seems to affect the zirconia surface and improves its bond strength but not the glass ceramic surface.

Despite the fact that conventional cements, such as zinc phosphate or resin-modified glass ionomer cements, could be used when luting zirconia restorations giving an adequate initial clinical stability (Qeblawi *et al.,* 2010; Menani *et al.,* 2014), adhesive cementation is considered to be more favourable in most cases. It has shown some advantages such as increased stability, better marginal adaption and good aesthetic outcome. Resin cements are considered the first choice when it comes to luting zirconia constructions because of their flowability, wettability and composition (Menani *et al.,* 2014). To obtain a good bonding to zirconia, different surface treatments are being used today, but only few available that are effective and with clinical acceptance (Menani *et al.,* 2014). Qeblawi *et al*. (2010) refered to the lack of consensus for the best surface treatment method of zirconia prior to cementation, given that different results had been shown on short- and long-term studies. Air Particle Abrasion (APA) is a method used to roughen the zirconia surface before cementation to enhance a better bonding (Qeblawi *et al.,* 2010). While some studies supported APA, others discussed the long-term effect on zirconia’s flexural strength that could be reduced up to 20-30% because of the creation of superficial microcracks and/or phase transformation (Qeblawi *et al.,* 2010; Menani *et al.*, 2014). Application of primer, as a bonding promoter, to high-crystalline content ceramics is another method used for the same purpose (Menani *et al.,* 2014). One type of primers showed a significant decrease in shear bond strength after 6 months of aging in water (Lindgren *et al.,* 2008). Another study concluded the good effect of primer application but highlighted the need for a micro-retentive and high-energetic surface (da Silva *et al.,* 2014).

A possible and expected complication with the method used in the present study is debonding of the veneering layer (chipping). One study stated that chipping failures after an observation period of 2 - 5 years of veneered zirconia FDPs were reported to be 15% (Elsaka, 2013). Interestingly, the results showed no fracture in veneering layers in all tests in the present study regardless type of luting agent and pretreatments used. One possible explanation to that could be the weakness of the luting agents used.

In addition, a recently published study showed that zirconia could be etched with hydrofluoric acid (Sriamporn *et al.,* 2014). The study evalutated the effect of applying HF-acid with different concentrations in different immersion times and solution temperatures (Sriamporn *et al.*, 2014). In the study, it was concluded that HF-acid could cause changes in surface topography of zirconia specimens in form of micro-morphological changes and phase transformation (tetragonal to monoclinic) (Sriamporn *et al.*, 2014). This might be promising and would get developed in the near future giving an acceptable bond strength of such constructions.

The values for the bond strength reported in previous studies of zirconia bonded to resin cements with/without surface treatments ranged between 3.62 – 26.01 MPa (Blatz *et al.*, 2007, Peutzfeldt *et al.,* 2011, Liu *et al.*, 2014). Those studies were all determined using shear bond strength test. The sufficient bond strength for RBFDPs is still unknown. In other words, we do not know the desirable bond strength for this type of restorations and it should be kept in mind that it is often more favourable that the RBFDP gets loose before the tooth will be damaged. The most reliable test to find out the advantageous designs and luting systems for this type of restorations is, therefore, an increased number of retrospective and prospective studies of RBFDPs placed in various patients.

Conclusion

Within the limitations of this *in vitro* study, the following conclusions could be drawn; the bond strength was not improved by covering the surface of zirconia with a feldspar-based or glass ceramic. The null hypothesis was therefore rejected.
RelyX Unicem seemed to affect the zirconia surface and improved its bond strength but not the glass ceramic surface.

Clinical relevance

Today, layering glass-ceramic on Y-TZP RBFDPs is used in some dental laboratories to improve the bonding to resin cements. However, based on the findings in the present study, more studies are necessary before general recommendations can be made.

Strength and limitations

The direction of the force applied on the specimens in the present study seems to be more similar to clinical situations than "shear bond strenght test".
Only one type of resin composite cement was used to examine the effect of veneering.
Only six pairs of specimens was tested in each group. Different types of resin composite cements and a larger number of specimens could have influeced the outcome.

Recommendation

The effect of various types of ceramic and metal primers and long-time storing should be evaluted.

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Table 1. Values of the bond strength (MPa) for each pair of the studied specimens (Group I - IV).

Specimen No	**Group I**	**Group II**	**Group III**	**Group IV**
1	5.34	3.66	0.91	0.61
2	5.14	1.45	0.76	0.74
3	6.12	1.65	0.43	0.48
4	7.77	1.83	1.73	1.32
5	6.21	4.17	3.55	0.48
6	5.96	2.61	1.27	0.80
Mean ± SD	6.1 ± 0.9	2.6 ± 1.1	1.44 ± 1.13	0.74 ± 0.31

Figure 1

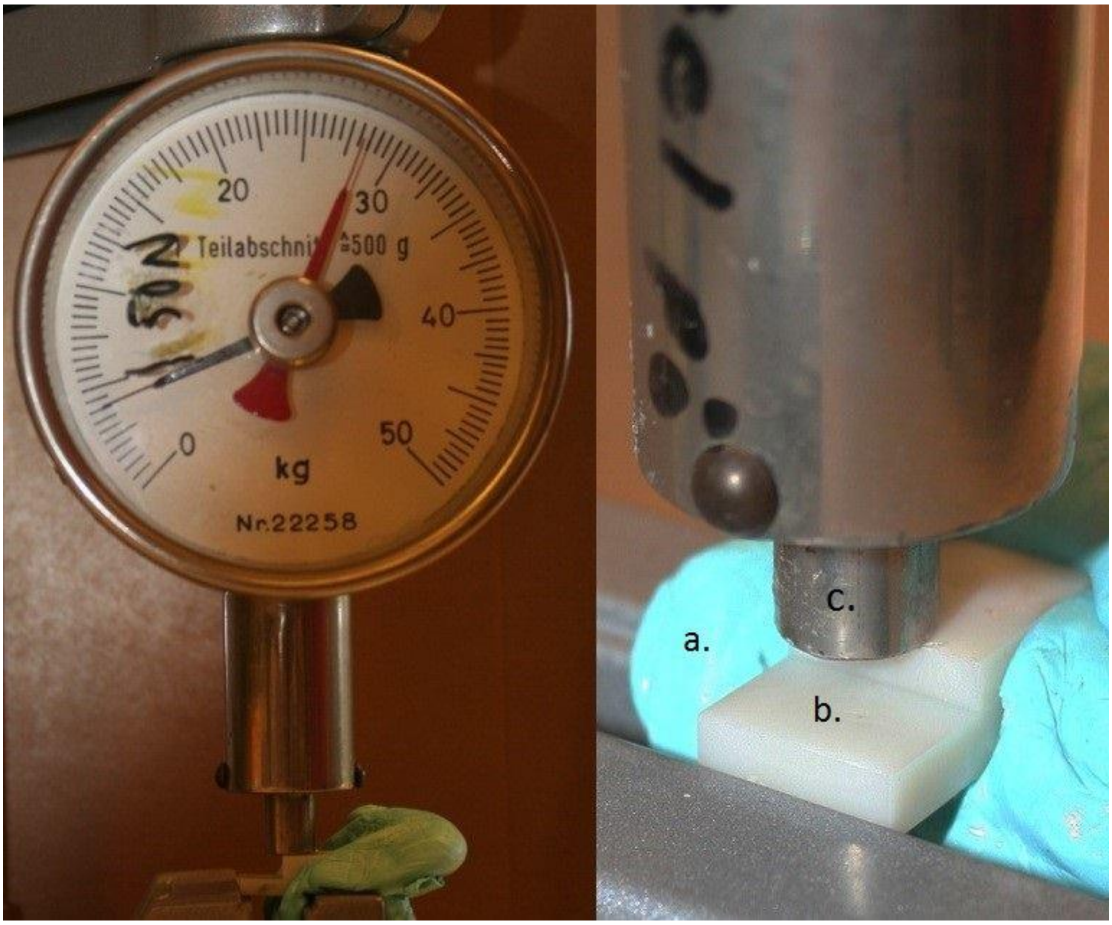

Fig. 1. The specimens placed in a mold of a vinyl polysiloxane impression material (a.) during the cementation procedure. The cemented pair of zirconia specimens (b.) being loaded by a plunger (c.).

Figure 2

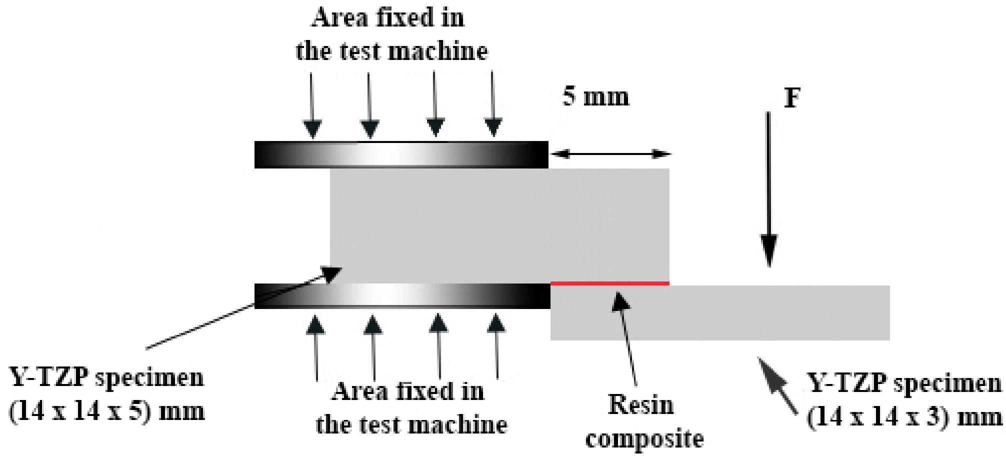

Fig. 2. Schematic drawing of the specimens placed in the testing machine.

Figure 3

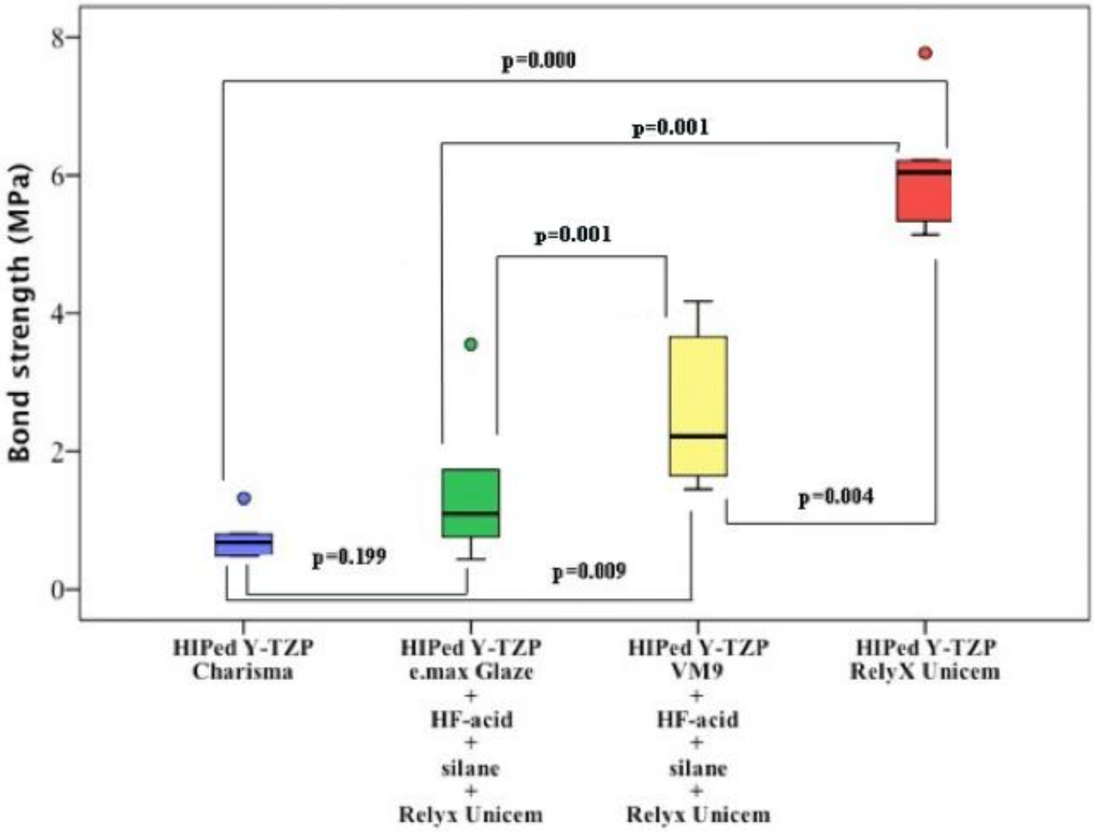

Fig. 3. Box-plot diagram comprising the bond strength (MPa) of the four studied groups. Six specimens in each group. A horizontal line within the box presents the median. Data are presented as medians, 1st and 3rd quartiles. The maximum and minimum values are illustrated via the upper and lower strokes. (•) Marks outliers.