On: 14 August 2012, At: 13:39
Publisher: Taylor & Francis
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK

Journal of Adhesion Science and Technology

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/tast20

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Version of record first published: 13 Aug 2012

To cite this article: Ammar A. Mustafa, Jukka Pekka Matinlinna, Andy H. Choi & A.A.A. Razak (2012): Fracture strength and fractographic analysis of zirconia copings treated with four experimental silane primers, Journal of Adhesion Science and Technology, DOI:10.1080/01694243.2012.701507

To link to this article: http://dx.doi.org/10.1080/01694243.2012.701507

Journal of Adhesion Science and Technology
2012, 1–13, iFirst article

Fracture strength and fractographic analysis of zirconia copings treated with four experimental silane primers

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(*Received 19 December 2011; final version received 27 March 2012; accepted 30 April 2012*)

This study evaluated and compared the effect of new four experimental silane coupling agents on the fracture strength of zirconia copings. The findings were supported with fractographic and finite element analyses. All together 125 zirconia copings with a wall thickness of 0.6 mm were fabricated on identical nickel-chromium master dies and then divided randomly into five groups ($n = 25$). Four test groups were prepared according the experimental silane primer (labeled: OIWA1, OIWA2, OIWA3 and OIWA4) and one ontrol group without silanization. The silane monomers used were: 3-methacryloxypropyltrimethoxysilane (in OIWA1), 3-acryloxypropyltrimethoxysilane (in OIWA2), 3-isocyanatopropyltriethoxysilane (in OIWA3) and styrylethyltrimethoxysilane (in OIWA4). Tribochemical sandblasting (silica-coating) treatment was performed to the inner surface of the copings in the test groups. All the specimens were silanized at the inner surfaces of the zirconia copings. Self-adhesive universal resin cement was used to cement the copings to the underlying master die. Zirconia copings were vertically loaded on the cusp area until the first crack failure was occurred using Precision Universal Tester at a constant crosshead speed of 1 mm/min. Then, the machine was manually controlled to cause more failure to further determine the texture of fracture. Three dimensional finite element analysis and fractography were performed to support the fracture strength findings. Based on the finite element analysis results, zirconia silanized with 3-acryloyloxypropyltrimethoxysilane showed the highest fracture strength with a mean of 963.75 N (SD 4.5 N), while zirconia copings silanized with 3-methacryloyloxypropyltrimethoxysilane showed a mean fracture strength value of 925.65 N (SD 2.4 N). Styrylethyltrimethoxysilane-silanised zirconia showed mean fracture strength of 895.95 N (SD 3.5 N). Adding silane coupling agents to the resin-zirconia interface increased the fracture strengths significantly (ANOVA, $p < 0.05$). Silanization with four new experimental silane primers *in vitro* produced significantly greater fracture strength than the control group not treated with the test silane.

Keywords: zirconia; silane coupling agents; tribochemical silica-coating; fracture strength; fractography; finite element analysis

1. Introduction

The interest in using zirconia (zirconia dioxide) ZrO_2 as a restorative material has been increased progressively in the last decades. This petition for zirconia has led to increase the

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ISSN 0169-4243 print/ISSN 1568-5616 online

http://dx.doi.org/10.1080/01694243.2012.701507
http://www.tandfonline.com

attempts to improve not only its characteristics but also to improve the pretreatment methods. As an indirect dental restorative material, zirconia is a crystalline dioxide of zirconium and it is a ceramic material. It is a metal free material characterized with excellent biomechanical properties such as has high flexural strength and fracture toughness with limited crack propagation. In addition, zirconia possesses adequately X-ray opacity and the same white colour characteristics of natural teeth; therefore, zirconia is substituting today many dental ceramic systems of crowns and bridges for fixed dental prostheses [1,2].

Strong and stable bonds of zirconia-based ceramics to adhesive resin cements or self-adhesive resin cements are required to achieve a successful clinical restoration [3,4]. Pretreatment methods have a great influence on the durability of the fixed restorations. These methods are used prior to chemical bonding with a silane coupling agent, ceramic primer or self-adhesive cement. The use of a tribochemical silica coating process and silanization on zirconia-based ceramic can improve both fracture strength as well as bond strength to resin cement [5–9]. Sandblasting is an important method used in dental technology and dentistry as part of a tribochemical silica coating process. It aims at cleansing the substrate surfaces, creating a rough, porous surface to enable a durable cementation. Silica-coating can be performed using a sandblasting system and special silica-modified alumina sand. Silica-coating is followed by silane application i.e. silanization, and this methodology may be used at dental laboratories and dentist's office [10,11]. Conditioning the surface of zirconia-based ceramics with chair-side tribochemical silica-coating and silanization is recommended to minimize the possible contamination during delivery of the restoration from the laboratory to chairside [12,13].

There are studies on silane coupling agents (trialkoxysilanes) and their use as adhesion promoting agents on silica-coated zirconia surfaces as they can play a significant and recognized role in promoting adhesion in composite and coating technology [14,15].

Silane coupling agents are hybrid inorganic–organic bi-functional molecules that bond together dissimilar materials, such as inorganic surfaces to polymeric organic surfaces and coatings. They contain an organo-functional part and three hydrolyzable alkoxy groups (Figure 1). Before acting as adhesion promoters, silanes must first be activated and condensed so as to be able to act as coupling agents. Trialkoxysilanes necessitates going through a hydrolyzation reaction (activation) in slightly acidic ethanol–water solvent to form silanols from trialkoxy groups. The organo-functional part, most often a methacrylate group, can then be polymerized with the monomers of a resin composite system. There are in the dental market a plethora of prehydrolyzed, ready-to use dental silanes that, however, differ from their exact constituents, pH, solvent and other properties and they may exhibit varying adhesion promotion [15].

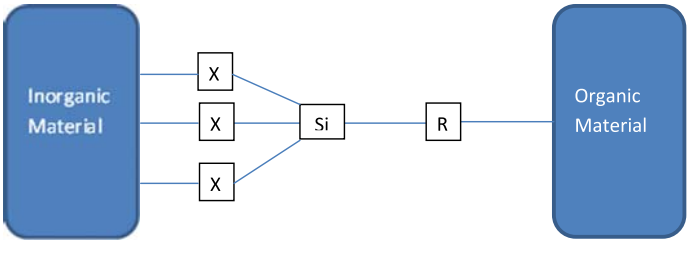


Figure 1. The silane molecule contains one silicon (Si) in the center and an organo-functional group, (R), e.g. vinyl, amino, epoxy, etc. and (X) functional hydrolysable group, e.g. methoxy, ethoxy, etc. The functional group R will bond with an organic resin material while the functional group (X) will attach to an inorganic material. By this, coupling effect can be attained.

Silane coupling agents with varying functionalities have been assessed for resin zirconia and resin titanium bonding, e.g. assessing mercaptosilane, acrylatesilane, polysulphur and aminosilanes alone or blended with a cross-linking silane [16–19]. Various silane functionalities have thus exhibit different reactivities with luting resin composites and experimental resins *in vitro* as reported above.

Four different types of new experimental silane primers (labelled: OIWA1–4) were employed for fracture strength test. The silane primers based on methacrylate, as a functional group, were expected to have the highest impact on fracture strength of zirconia copings due to copolymerization with methacrylate groups at the resin cement. On the other hand, 3-isocyanatopropyltriethoxy silane was expected to have no impact on the fracture strength of zirconia copings. The null hypothesis tested was that the new experimental silane primers have no significance on the fracture strength of zirconia copings and that the fracture line propagation can be initiated at any part of the coping including the inner surface.

2. Materials and methods

2.1. Zirconia copings preparation

A total of 125 master nickel-chromium (Wiron 99, BEGO) dies were identically duplicated from prepared mandibular molar tooth. Then, 125 zirconia copings (Lava™ Zirconia 3M ESPE) with a wall thickness of 0.6 mm were fabricated and divided randomly into five groups ($n = 25$). Four test groups according the experimental silane primer (labeled: OIWA1–4) and one control group without silanization.

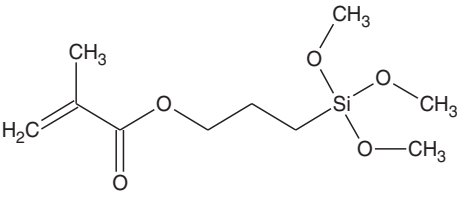
2.2. Activated trialkoxysilane solutions and silanization

The experimental primers were two-bottle system, consisting of two components which were combined just prior to the use, to activate these primers. As ready, the primers contained 99% solvent (5 vol.% water–95 vol.% ethanol, set at pH 5 with acetic acid) and 1.0% reactive silane monomer. The silane monomers used were: 3-methacryloxypropyltrimethoxysilane (OIWA1; purity 98%, Aldrich, Steinheim, Germany, Lot 00901DJ), 3-acryloxypropyltrimethoxysilane (OIWA2; purity 95%, Gelest, Morristown, PA, USA, Lot 5C-6412), 3-isocyanatopropyltriethoxysilane (OIWA3; purity 100%, Gelest, Morristown, PA, USA, Lot 9E-14595) and styrylethyltrimethoxysilane (OIWA4; purity 92%, ABCR, Karlsruhe, Germany, Lot 7D-10467-S) (Figure 2). After mixing, they were allowed to activate for 1 h at room temperature and then used immediately.

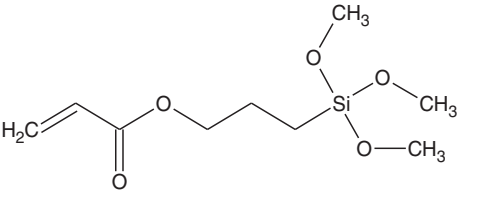
2.3. Bonding of cement to copings

A tribochemical sandblasting treatment was performed to the inner surface of the copings in the test groups to achieve coating with small particles of silica Rocatec® (ESPE, Seefeld, Germany).

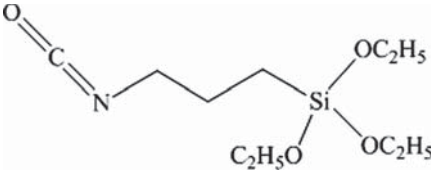
The sandblasted copings were submitted to tribochemical silica-coating for 15 s at an operating pressure of 40 psi and at a standoff distance of 1 cm at a right angle from the sandblaster nozzle tip (2 mm in diameter) [20]. Sandblasting procedure was carried out uniformly. Then all the specimens were silanizated at the inner surfaces of the zirconia copings. Self-adhesive universal resin cement (RelyX™ Unicem, 3M ESPE) was used to cement the copings to the underlying master die. Each silane primer coating was applied with a new, clean brush. The silane was allowed to dry and react for 3 min, and then gently dried with oil-free compressed air. A static load of 5.0 kg for 10 min was applied to the copings.



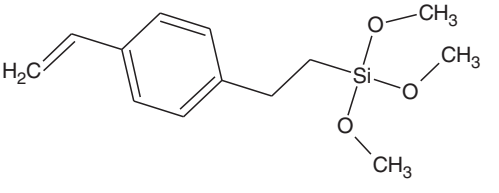
3-Methacryloxypropyltrimethoxysilane (in OIWA1)



3-Acryloxypropyltrimethoxysilane (in OIWA2)



3-Isocyanatopropyltriethoxysilane (in OIWA3),



Styrylethyltrimethoxysilane (in OIWA4)

Figure 2. Silanes used in the study.

2.4. *Fracture strength test*

After 24 h of storage in de-ionized water at 37 °C, the copings were vertically loaded on the cusp area until the first crack failure was occurred using Precision Universal Tester (Shimadzu Autograph AG-X series, Japan) at a constant crosshead speed of 1 mm/min. Then, the machine was manually controlled to cause more failure to further determine the texture of fracture (visually observed).

2.5. *Fractographic analysis*

Fractographic analysis of failed zirconia copings can provide understandings as to the failure origin and related mechanisms. Random copings were selected from each group for qualitative

fractographic analysis. The broken parts of the zirconia copings were examined by scan electron microscopy (EVO 50, ZEISS Germany) to identify the actual patterns of fractography. Different magnifications were utilized to conclude the fractography modes. The direction of crack propagation was plotted and taken back to the origin of failure whether at the interface between the silanized surface and the cement and the zirconia or at the outer surface of the coping.

2.6. *Finite element analysis*

To gain a better understanding into the mechanical behaviours of the zirconia crown and the nickel-chromium die and how the stresses are transferred from the crown to the coping through the cementing material, three-dimensional finite element analysis was used. The model was constructed using physical measurements taken from the crown and coping. During sample preparation for the fracture testing, the thickness of the cementing material used was recorded applied during the construction of the model (Figure 3).

Since the silane was an activating agent to achieve better adhesion between the cement and zirconia coping, and does not play a part in the transfer of mechanical stresses, it was neglected in the finite element model.

For this analysis, a maximum force of 963.75 N was evenly distributed and applied to the top of the zirconia crown in a downward motion to simulate a compressive load. All the materials in this finite element study were considered to be homogeneous and isotropic (Table 1). Perfect bondings were also assumed between the zirconia crown to the cementing material and to the nickel-chromium die. Finite element simulations were carried out using a commercially available package, STRAND7.

For statistical analysis for the zirconia copings, a two way ANOVA (IBM SPSS version 16, USA) was used with fracture strength as the dependent variable, p-values less than 0.05 were considered to be statistically significant in the tests. The fracture strength analysis curve was assessed by using Trapezium-X software (Shimadzu, Japan).

For the finite element analysis, the statistical analysis was performed using SPSS 16.0 software for Windows (SPSS 10.0, SPSS, USA).

3. Results

The results of the fracture strength are shown in Table 2. All four experimental silanes groups showed greater fracture strength than the control group. ANOVA revealed significant difference

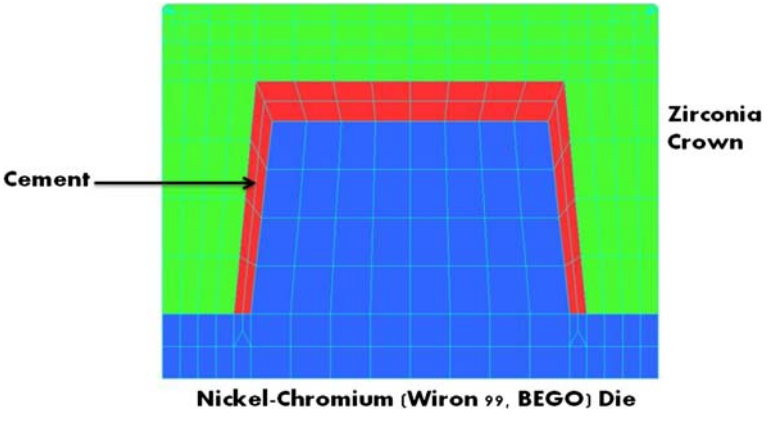


Figure 3. Cross-section of the finite element model.

Table 1. The mechanical properties of the materials used in this study and analysis.

Materials	Elastic modulus (GPa)	Density (g/cm^3)
Wiron 99[a]	~205	8.2
Lava™ Zirconia[b]	210	6.08
RelyX™ Unicem[c]	6.6	

[a]http://begousa.com/Results.wss/search_display/individual_product/item_guid/c00a33f5-c1bc-aaa2-2ae8-e0edb4b63285.
[b]http://www.gandhdental.com/pdfs/3M_LAVA.pdf.
[c]http://multimedia.3m.com/mws/mediawebserver?mwsId=66666UuZjcFSLXTtnxf2l8TXEVuQEcuZgVs6EVs6E666666-&fn=rx_u2_auto_tds.pdf.

Table 2. The mean fracture strength for the silane groups with standard deviation.

Silane code in testing	Silane name	*n*/group	Mean fracture strength (N)	Standard deviation (N)
OIWA1	3-Methacryloxypropyltrimethoxysilane	25	925.65	2.4
OIWA 2	3-Acryoxypropyltrimethoxysilane	25	963.75	4.5
OIWA3	3-Isocyanatopropyltriethoxysilane	25	689.80	5.2
OIWA4	Styrylethyltrimethoxysilane	25	895.95	3.5
Control	No silanation	25	598.80	2.2

in the fracture strength between the test groups and the control group ($p < 0.05$). There was no significant difference among OIWA 1, 2 and 4. The highest mean fracture strength was obtained for 3-methacryloyloxypropyltrimethoxysilane (963.75 N) while the lowest mean fracture strength was obtained by 3-isocyanatopropyltriethoxysilane (689.8 N). Fracture strength values of 925.65 and 895.95 N were obtained for 3-methacryloyloxypropyltrimethoxysilane and styrylethyltrimethoxysilane respectively.

Fractographic results are shown in Figures 4–6. The texture of fracture was visually observed and it was found that the fracture cracks for most of the specimens of the test groups were intact with no piece separation observed while 10 out of 25 specimens in the control group have lost parts of porcelain after the test. Fractographic analysis showed that the propagation of the fracture line was at the outer surface of the coping for all the specimens of the test groups except one specimen from OIWA 3 which exhibited irregular cracks and some distant fracture lines. Some of tested control specimens showed distant fracture line from the area of load application.

The results of the finite element analysis are shown in Figures 7 and 8. The distribution of the tensile and compressive stresses under loading is shown in the form of contoured colour plots where each colour represents a different value.

Maximum compressive stresses were observed at the top surface of the zirconia coping where the load was applied (Figure 7). Compressive stresses were also observed on the interior of the coping. It is noteworthy to mention that most of the compressive stresses were dissipated by the zirconia coping; only a small magnitude of the compressive stress was transferred from the coping to the nickel-chromium die.

While the compressive stresses acted mainly at the top of the zirconia coping, tensile stresses were observed on the inside of the zirconia coping at the interface of the zirconia coping and cement layer. This can be explained by the bending of the zirconia coping where the top surface will experience compression and the bottom will experience tension. Cracking of the zirconia coping may occur due to this high magnitude of tensile stress. Failure of the adhesion, i.e. delamination, between the zirconia coping and cement layer may also occur when the load is applied.

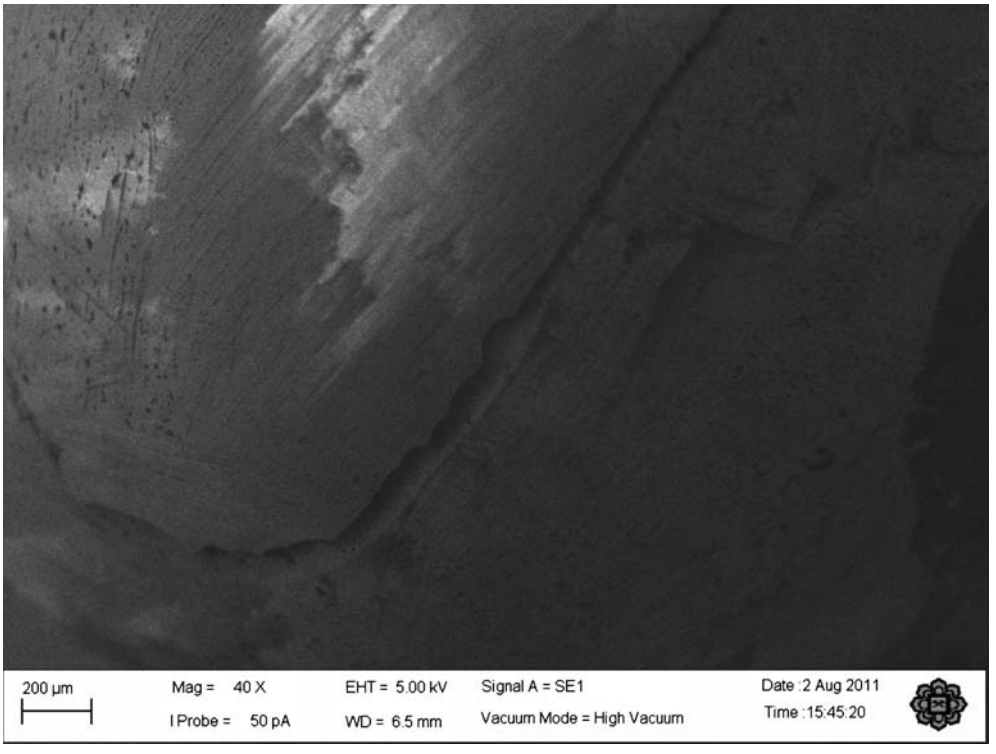


Figure 4. Initial crack formation at the outer surface of the zirconia coping (magnification = 40×) (test group).

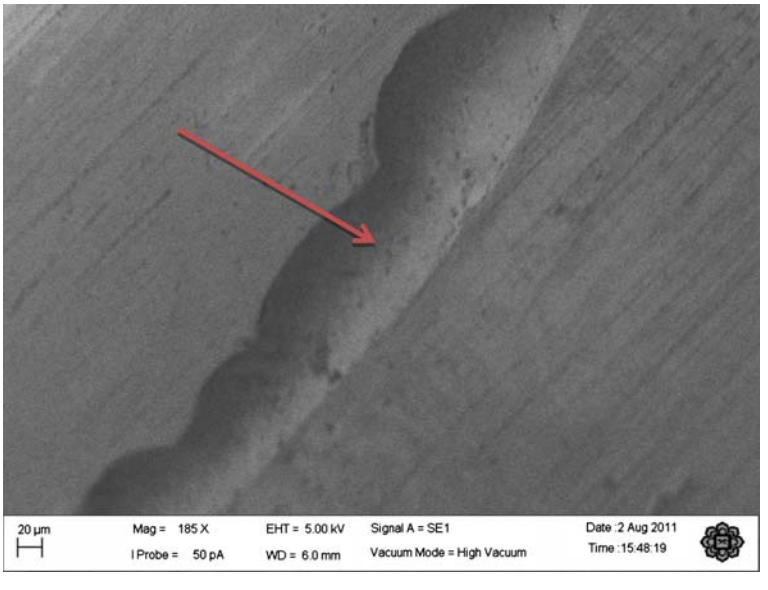


Figure 5. The initial crack was originated on the outer surface of the zirconia copings (magnification = 185×.

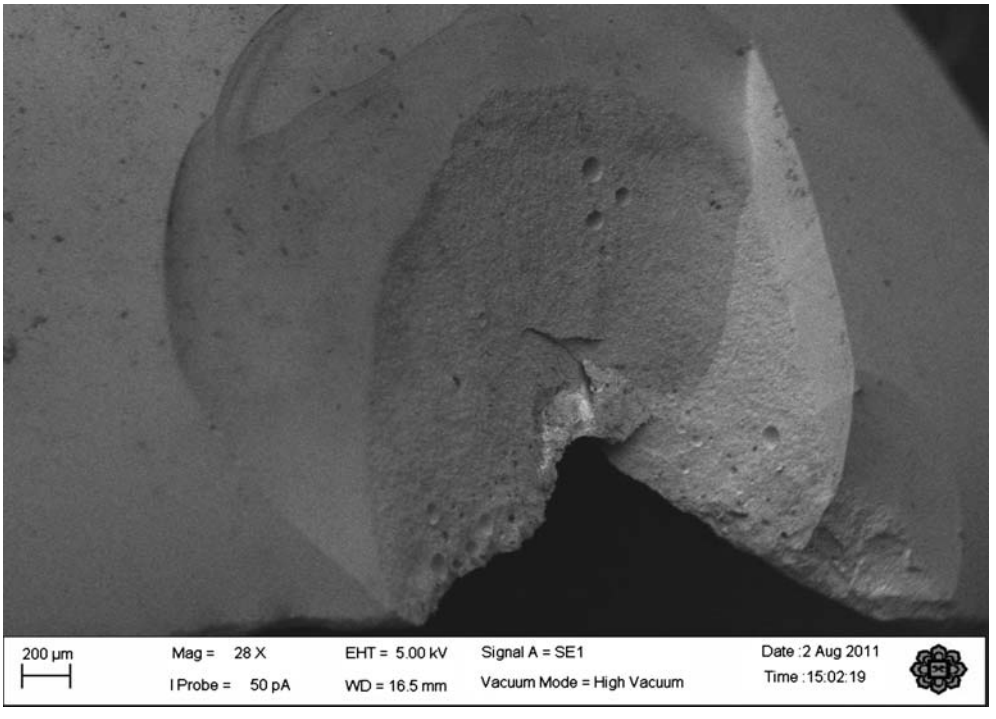


Figure 6. Fractography after complete separation of the fractures part. The propagation of the fracture starts at the outer surface of the zirconia coping (test group).

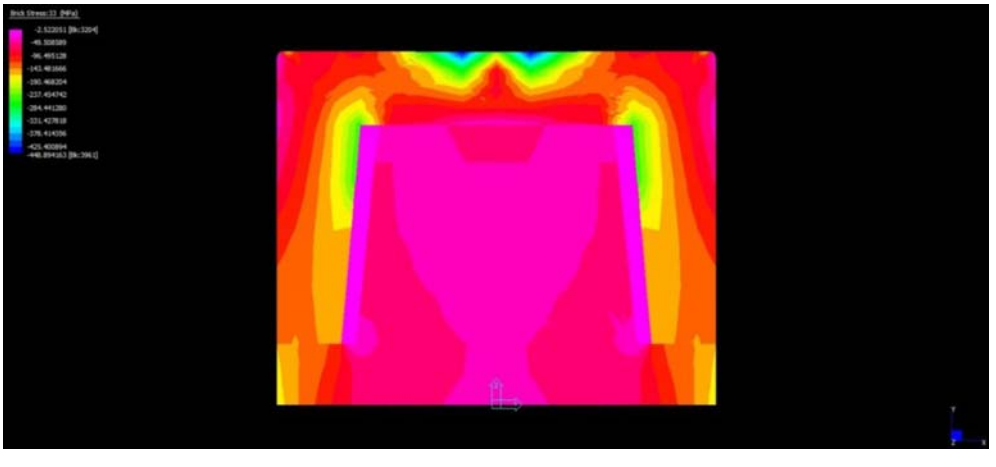


Figure 7. Compressive stresses observed on the finite element model under loading (cross-sectional front view).

4. Discussion

In recent years, there were growing studies on the use of zirconia-based dental ceramics because of their high strength and esthetic properties. Metal master dies were used in this study to secure fractures between the cement and the zirconia [21].

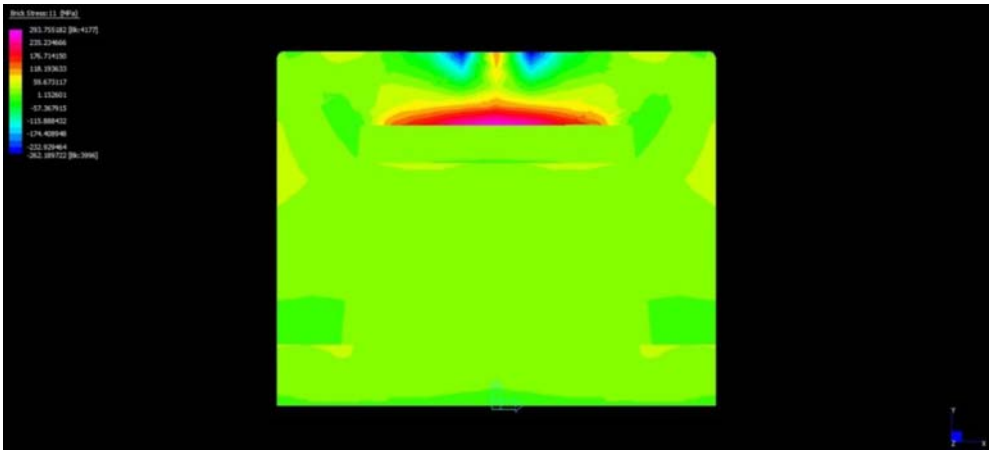


Figure 8. Tensile stress observed on the finite element model under loading (cross-sectional front view).

A strong chemical bond with zirconia has proven very challenging to obtain which may be attributed to the non-reactive nature of its surface. Multiple treatment techniques have been investigated like the application of reactive monomers to promote chemical attachment to the polymer adhesive [22].

Selection of the best combination between the resin adhesive cement and the surface treatment is more important than selecting the adhesive itself or the type of the surface treatment [20]. There is no significant difference in bond strength between self-adhesive resin cement and multistep adhesive resin cement [23]. Adhesive cements are used to prevent the dislodgement of the zirconia crowns and to obtain strong and durable bond between the restoration and the tooth [20].

Resin-ceramic bond requires silica-coating and tribochemical pre-treatment to produce irregularities or micro-porosities to achieve mechanical interlocking between the cement and the zirconia. In this study, applying silica coating and tribochemical treatment improved the bond strength of self-adhesive resin cement to zirconia [24,25]. It is very important to realize that sandblasting is expected to induce crack initiations in the inner surface of the zirconia copings. Phase transformation of zirconia from tetragonal phase to the monoclinic phase resulting in degradation of zirconia. On the other hand, this compression stress associated with transformation may increase mechanical properties of zirconia ceramics. This can clearly explain the relative high fracture toughness and strength for zirconia along with the ability to resist crack propagation [20,26].

Roughening the inner surface of zirconia by tribochemical method followed by silanization obviously increased the bond between the resin cement and the zirconia surface [27,28]. The silica layer left by silica coating on the ceramic surface affords a foundation for silane to react. In the ceramic-resin bond, silane acts as a coupling agent which is needed as a ceramic primer for silica-based ceramics. It adsorbs onto the surface of the ceramic and at the same time it alters the texture of the ceramic surface, in that way silane is facilitating the chemical interaction [29,30]. Silane coupling agent can react with the hydroxyl group on embedded silica at the surface through the formation of siloxane networks [31]. The use of proper silane has more important effect to achieve resin-ceramic bond even if no pre-treatment method is used [32,33]. Organo-functional silanes are usually hydrolyzed sufficiently in one hour or less and it was concluded to be adequate in this study [34,35].

The current study reveals an interesting finding that the use of silane primers together with the resin cements keeps the zirconia coping intact and not simply cracked or fractured under load. Copings silanized with 3-acryloxypropyltrimethoxysilane showed the highest fracture strength 963.75 N and followed by copings silanized with 3-methacryloxypropyltrimethoxysilane 925.65N, which were statistically significant from the control group. 3-Methacryloxypropyltrimethoxysilane did not significantly differ from 3-acryloxypropyltrimethoxysilane which was in agreement of other studies [15]. Silane primers based on methacrylate as a functional group are the most often favored coupling agents because of the excellent copolymerization properties of the methacrylate [36].

Styrylethyltrimethoxysilane, a highly reactive aromatic silane and could be used successfully for filler silanization [37]. In this study, zirconia copings silanized with styrylethyltrimethoxysilane showed fracture strength of 895.95 N which was significantly different from the control group. The lowest fracture strength was obtained with copings silanized by the 3-isocyanatopropyltriethoxysilane 689.80 N. There was no significant difference between 3-isocyanatopropyltriethoxysilane and the control with no silanization 598.80 N. Some studies proved that 3-isocyanatopropyltriethoxysilane did not promote adhesion when bonding an experimental *bis*-GMA resin or the RelyX™ ARC luting cement to silica-coated zirconia [16]. 3-Acryloxypropyltrimethoxysilane, and styrylethyltrimethoxysilane may *in vitro* significantly enhance the bonding of resin to silica-coated zirconia, due to their specific chemical reactivity [38].

In fractography, it was observed that crack propagation was formed in radial shapes around the region of the direct single load by the precision universal tester [39]. Scan electron microscopy (SEM) results showed that all the cracks propagations were originated at the outer surface of the zirconia copings. Fractographic analysis of the zirconia copings cemented with self-adhesive resin cement and silanized with different types of silane primers, showed interesting results with regards to the texture of fracture and the side of fracture propagation. The precision universal tester was controlled manually to cause more fracture. This was observed visually to determine the intact of the zirconia.

All the zirconia copings with silanization showed an integral pattern of fracture with no chipping of the broken pieces from the zirconia; full zirconia crown showed not chipping but bulk fracture [40,41]. The silane primers enhance the bond strength of the resin-ceramic interface hence; the closely adapted interface is a definite sequel of the dual-function of the coupling silane monomers which in turn can promote adhesion between dissimilar matrices, such as resin-ceramic interfaces, because their organo-functional group polymerizes with hydrophobic resin-composite monomers while three hydrolysable alkoxy groups bond with hydrophilic silica and silica-coated surfaces [37]. This is in agreement with recent studies which roved that experimental silane monomer primers can significantly increase the micro-tensile bond strength between resin and zirconia [3,4].

Finite element analysis is used to precisely calculate local stress-strain distributions in geometrically complex structures. The predictive accuracy of the finite element model is influenced by the geometric detail of the object to be modeled, the material properties and the applied boundary conditions. Similar to the fractography tests, high magnitudes of compressive stress were observed at the outer surfaces of the zirconia coping. The finite element method has been used to demonstrate the fracture behaviour of the zirconia coping under compressive loads.

5. Conclusions

This zirconia and silane study, has led us to the following conclusions:

(1) Silane primers as coupling agents with the self-adhesive resin cement improve the integral pattern of zirconia copings without chipping of the parts.
(2) It was obvious from the fracture strength results that 3-acryloxypropyltrimethoxysilane and 3-methacryloxypropyltrimethoxysilane have a great influence on the resin-ceramic interface through their dual function of the coupling silane monomers which in turn can promote adhesion between dissimilar matrices and at the same time can impart fracture resistance to zirconia.
(3) It was clear from the fracture strength results that a non-methacrylate based silane primer (styrylethyltrimethoxysilane) can promote adhesion between cement and zirconia interphase.

Acknowledgements

This study was financially supported by grants from Research Management Center (RMC), International Islamic University Malaysia (IIUM). The Center is highly acknowledged for this generous sponsorship.

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