EFFECT OF SURFACE TREATMENT AND ARTIFICIAL AGING ON MICROTENSILE BOND STRENGTH OF ZIRCONIA TO RESIN CEMENT

Ahmed M. Hamdy* *and* Abou Bakr Hossam Hashem**

ABSTRACT

Statement of problem: Reliable bond to zirconia based materials has proven to be difficult which is the major limitation against luting zirconia restorations.

Purpose: This in vitro study compared effect of selective infiltration etching (SIE) and tribo-chemical silica sandblasting (TSS) on microtensile bond strength to zirconia based material before and after different artificial aging intervals.

Materials and methods: Twenty Lava plus high translucency zirconia disks (3 M, ESPE) were randomly divided into two study groups according to their surface treatment: (a) selective infiltration etching (SIE) and (b) tribo-chemical silica sandblasting (TSS). Zirconia disks were bonded to composite resin specimens using Panavia F2.0 resin cement. Zirconia resin bond strength was evaluated using the microtensile bond strength test (MTBS) and the test was repeated before and after the following intervals of accelerated artificial aging; 1 week, 2 weeks, 3 weeks and 4 weeks of water storage (37°C). A repeated measures ANOVA and Bonferroni post-hoc tests were used to analyze the data.

Results: The interaction between surface treatments and storage times were statistically significant. Data analysis revealed significant differences between the two surface treatments (P<0.05).Selective infiltration etched specimens demonstrated relatively higher MTBS values than TSS (48.5 and 24.8 MPa).SEM examination of broken microbars revealed more interfacial failure type for TSS than SIE specimens that showed a predominantly cohesive failure type.

Conclusions: Selective infiltration etching provided superior micro-tensile bond strength results compared to tribo-chemical silica sandblasting. Established bond strength by selective infiltration etching was not affected by artificial aging as tribo-chemical silica sandblasting.

* Associate Professor, Fixed Prosthodontic Department, Faculty of Dentistry, MSA University.
** Lecturer, Research Institute of Ophthalmology, Dental Department.

INTRODUCTION

Zirconia based materials are dynamic on microscopic structural level. These materials can undergo transformational phase changes in response to thermal and mechanical stresses[1]. Structural changes occur on the grain level, which tends to mature and grow in size when given sufficient time and temperature. Grain growth and cubic grain formation where observed when zirconia was heated to 1450°C for 2 hours[2]. When heated for 30 minutes at lower temperatures (700-900°C), the surface of zirconia was reported to undergo a thermal aging process, especially in the presence of water vapor. On a microscopic level, thermal aging resulted in the creation of surface elevations, grain pullout, detachment and increase grain boundary thickness [3,4].

During thermal etching of zirconia at a temperature of 1350°C for 12 minutes, surface elevations, rippled grain surfaces and vertical grooves at grain boundaries were previously reported using electron microscope. These observations were related to the tetragonal monoclinic transformation of zirconia crystals at the surface grains, which can accommodate the accompanying increase in volume, a state determined by grain boundaries and surface energies [4,5]. However, deeper grains are restrained and constrained by the bulk of the materials and thus become strained[5].

Several mechanical and chemical surface pre-treatments have been recommended to increase the bonding effectiveness of composite cement to zirconia. Former pre-treatments such as surface grinding using diamond burs[6], chemical etching using hydrofluoric acid[7] and laser irradiation [8] were applied to roughen the surface of zirconia ceramics, but none of them resulted in durable bonding to zirconia[6].

It appears essential to create both micro-mechanically prepared and a chemically activated surface. For instance, tribo-chemical silica sandblasting with 30 and 110 μm silica-coated aluminum oxide particles roughens and also chemically activates zirconia, thus making it more receptive for chemically bonding via silane coupling agents[8].

Previous in vitro study[10] showed that combination of mechanical pre-treatment using tribo-chemical silica sandblasting (Cojet, 3M ESPE; Seefeld, Germany) and chemical pre-treatment using a ceramic primer provided the highest bonding effectiveness to dental zirconia. More-over, the resultant bond appeared insensitive to mechanical aging[11].The difference between conventional Al2O3 sandblasting and tribo-chemical silica sandblasting in terms of their efficacy in making zirconia receptive to bonding is still unclear.

Tribo-chemical silica sandblasting with 30 and 110 μm silica-coated aluminum oxide particles has been shown not only to roughen but also to chemically activate zirconia, thus making it more receptive for chemical bonding via silane coupling agents. To avoid the well-documented sub-surface damage and transformation induced by high pressure and big particle size, one should apply air abrasion at lower pressure(1-2 bars) using particles up to 50μm in size[2,3]. The sole application of traditional ceramic (silane) primers appeared not very effective on zirconia[4,6], while the application of 10-MDP containing primers has been documented to chemically bond to zirconia, especially when applied on previously air –abraded zirconia using 50 to 110 μm alumina particles or 110 μm silica coated alumina sand[14,15].

This study aimed to assess the effect of two surface pre-treatments on micro-tensile bond strength to translucent zirconia after subjecting specimens to artificial aging. The null hypothesis tested were (1) bonding efficacy of resin-composite cement to zirconia was not different for the two pre-treatments tested, and (2) the strength of the tested samples was not affected by artificial aging.

MATERIALS AND METHODS

Twenty Lava plus highly translucent zirconia disks (3M, ESPE), (diameter :19.5 mm; thickness: 3 mm) were prepared as described in previous study[16], cutting procedure was precisely guided and carried out with a horizontally moving digital micrometer (IDC 1508, mitutoyo; Kawasaki, Japan).

Samples were polished using silicon carbide papers starting with a 120 grit and ending with a 800 grit (Microcut, Buehler). Polishing was carried out by using a rotating metallographic polishing device (Ecomet, Buehler) under a 300gm load and water cooling .

10 disks (group 1) underwent selective infiltration etching (SIE) surface treatment[16], the method employs a heat induced maturation process to pre-stress surface grain boundaries on zirconia to allow infiltration of molten glass. Zirconia is heated to 750°C for 2 minutes, cooled to 650°C for 1 minute, reheated to 750°C for an additional 1 minute and then cooled to room temperature .After this heat treatment ,the grain boundaries become pre-stressed and can be easily infiltrated by other materials. After cooling to room temperature, the glass is then etched using 5% hydrofluoric acid for 30 min. this creates rough surface topography with deep grooves at zirconia grain boundaries, allowing nanomechanical interlocking of resin composite cement.

10 disks (group 2) underwent tribo-chemical silica sandblasting (Co Jet, 3M ESPE; Seefeld, Germany).

A total of 20 resin composite disks (Filtek Z 250, 3M, ESPE; St Paul, MN, USA) measuring 18.0 mm in diameter and 3 mm in thickness were prepared by injecting the composite resin into a mold and light polymerized for 20 sec each from the top and bottom (Elipar Free Light 2,3 M ESPE).

All Disks were coated with a freshly mixed resin composite cement (Panavia F 2.0; Kuraray Co. Ltd.,

Tokyo, Japan) and seated on top of the surface of the treated zirconia specimens under a fixed load of 20N. Excess cement was wiped off and an air barrier gel was applied as a coating around the margins of the double-layered specimens.

After storage at room temperature for 24 h, the specimens were cut into 10 microbars per group (9mm length, 2mm×2mm in cross-section) using a precision cutting device (Isomet 1000; Buehler, Lake Bluff, IL) and a diamond-coated saw (Diamond Wafering Blade, No. 11-4276; Buehler) under water cooling[14]. The microbars were glued to the attachment unit (Clearfil SE Bond, Kuraray) taking care to center the resin-to-zirconia interface in the free space of the attachment unit. Zirconia-resin micro-tensile bond strength was measured by applying an axial load on the bonded interface using a universal testing machine (Instron 6022; Instron Corp., High Wycombe, England).

The load cell (100 N) was calibrated using standardized loads and the cross-head speed was calibrated using a digital micrometer (Millitron; Feinpruf Perthen, Gottingen, Germany).

Tests were conducted to the 10 test groups, immediately, then a week, two weeks, three weeks and four weeks from cementation time ,during which time the microbars were stored in demineralized water at 37°C (T 340; Heraeus Kulzer). Failure load was extracted from computer generated data files. The broken microbars were ultrasonically cleaned, dried, gold sputter coated, and examined under a SEM (XL20; Philips, Eindhoven, the Netherlands) and failure type was classified into interfacial failure across the zirconia–resin interface resulting in exposure of the zirconia surface or cohesive failure in the composite material or the adhesive resin (Fig.2-3).

A repeated measure analysis of variance (ANOVA) was selected to analyze the data with 1 factor (time, 5 levels). Bonferroni post hoc test was selected for pairwise comparisons (α=.05).

The sample size (n = 10 discs/group) was based on a power analysis (power=1) set to detect medium effect size differences (F=0.25) which, in terms of the bonding technique and MTBS values, could be of clinical relevance (Sigma-Stat Version 3.0; SPSS Inc, Chicago, Ill). One-way analysis of variance was used to analyze the data.

RESULTS

Data analysis table 1, graph. 1 revealed significant differences between the two surface treatments (P≤0.05). On the other hand, SIE specimens demonstrated relatively higher MTBS values than TSS (48.5 and 24.8 MPa).

In table 2, graph. 2 there was a significant interaction between water storage time and the bonding technique P<0.001 for TSS groups. MTBS values were stable overall of testing intervals for SIE groups.

SEM examination of broken microbars revealed more interfacial failure type for TSS specimens than SIE specimens that showed a predominantly cohesive failure type in resin cement as shown in fig.2, 4.
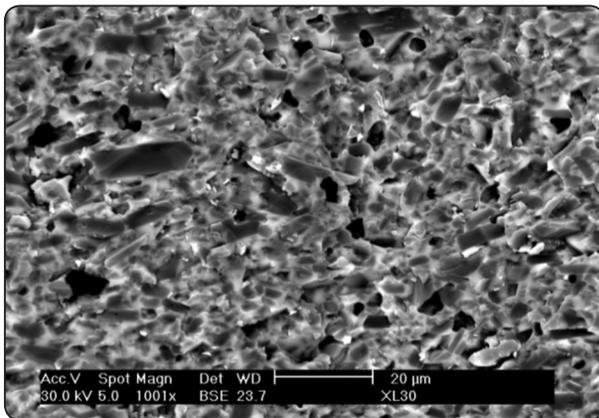
Fig. (1) SEM image after SIE of Zirconia x1000.
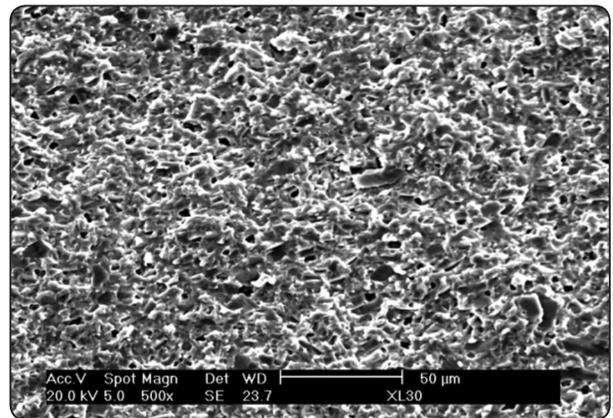
Fig. (2) SEM image showing cohesive failure in resin cement x500.
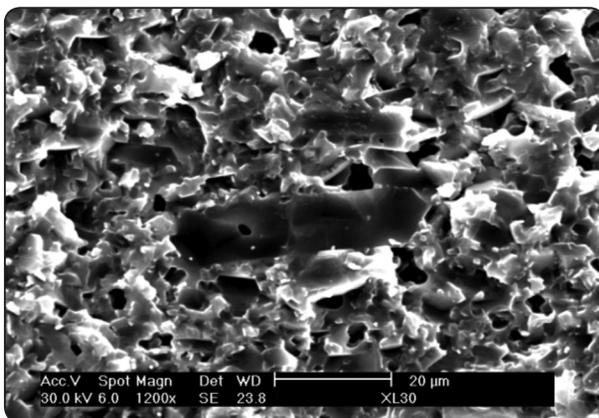
Fig. (3) SEM image showing interfacial failure of TSS specimen x1200.
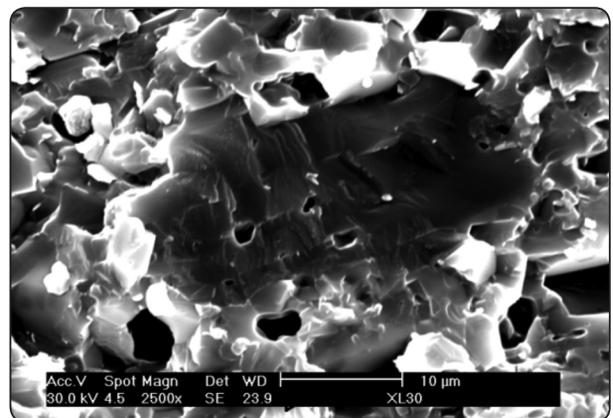
Fig. (4) SEM image showing microgaps due to hydrolysis of TSS specimen x2500.

TABLE (1) Microtensile bond strength (Mpa) of groups at different time intervals

Groups	Zero time		Week 1		Week 2		Week 3		Week 4		P value * for time effect
	Mean	(SD)	Mean	(SD)	Mean	(SD)	Mean	(SD)	Mean	(SD)	
SIE	48.50	(1.72)	46.90	(1.66)	47.80	(1.99)	47.60	(1.78)	45.90	(1.72)	0.001
TSS	28.40 **A**	(2.17)	29.40 **A**	(0.84)	27.40 **AB**	(2.07)	28.10**A**	(2.02)	24.90 **B**	(2.17)	

__ P value is significant £ 0.05 , All comparisons are Bonferroni adjusted . Time points sharing same letter are not significantly different__*
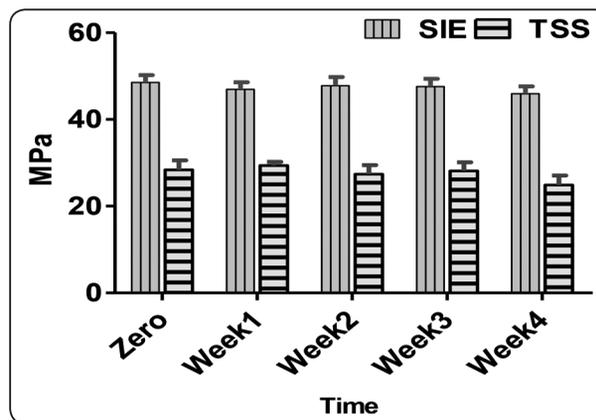
Graph. (1) Mean MTBS at different time intervals.

TABLE (2) Different types and percentage of failures.

		Study group				P value
		SIE		TSS		
		No	%	No	%	
Mode of failure (0)	Cohesive	10	100.0	6	60.0	**0.09**
	Interfacial	0	0.0	4	40.0	
	Total	10	100.0	10	100.0	
Mode of failure (w1)	Cohesive	9	90.0	7	70.0	**0.58**
	Interfacial	1	10.0	3	30.0	
	Total	10	100.0	10	100.0	
Mode of failure (w2)	Cohesive	10	100.0	7	70.0	**0.21**
	Interfacial	0	0.0	3	30.0	
	Total	10	100.0	10	100.0	
Mode of failure (w3)	Cohesive	8	80.0	5	50.0	**0.35**
	Interfacial	2	20.0	5	50.0	
	Total	10	100.0	10	100.0	
Mode of failure (w4)	Cohesive	9	90.0	6	60.0	**0.30**
	Interfacial	1	10.0	4	40.0	
	Total	10	100.0	10	100.0	

DISCUSSION

As SIE influenced significantly the bonding effectiveness to zirconia, null hypothesis (1) was rejected. Null hypothesis (2) was also rejected, as mechanical aging affect bonding effectiveness in TSS groups.

Results of this study emphasized superior MTBS results of SIE than TSS groups and were consistent with previous studies [17-21]. Fracture mode analysis for SIE groups remained primarily cohesive in the resin cement over the testing intervals (fig.2), while TSS groups showed an increase in the percentage of interfacial failure between zirconia and adhesive resin with water storage, indicating that the established bond was liable to hydrolysis and degradation in the presence of water [22] (Fig. 4).

Procedure of selective infiltration etching create a retentive architecture (fig. 1) that differs from airborne particle abrasion as it is self-introduced material without applied external mechanical stresses, second, it occurs on ultrastructural grain level without creation of surface defects on material loss, and finally it creates 3-dimensional retentive features where the adhesive resin can infiltrate [23].

Tribo-chemicalsilica sandblasting was opted in this study since this form of treatment –against conventional sandblasting using alumina powder –appeared as the most effective mechanical pre-treatment of zirconia regarding bonding receptiveness. Some authors [24,25] reported that sandblasting may negatively influence the properties of zirconia ceramics, it can produce micro-cracks and thus decrease the strength and longevity of zirconia.

In vitro dental researches conventional methods for aging the specimens are storage in deionized water, thermocycling, fatigue strength testing and chemical degradation in NaOH solution [26,27]. In the current study, 4 weeks aging period was selected. Oczan et al.[28], in a recent systematic review found that number of storage days varied enormously between 0.16 and 730 days, in different studies on bonding to ceramic oxide.

As the size of bonding area in the current study was relatively small; thus a faster aging effect could be expected. Data from 4 weeks immersion in this study showed a significant decrease in bond strength in TSS group. Degradation of resin –zirconia bonding might be attributed to water sorption, leading to hydrolysis degradation [29]. Water may decrease bond strength, especially to polycrystalline ceramics such as alumina and zirconia, as they can establish only weak chemical bonds to organo-phosphate based monomers as 10-MDP [30].

In this study, short term water storage might be considered as one of the limitations, because long term water storage will deteriorate the bond and affect the data to a greater extent [31].

TSS was not effective in creating adequate retentive features (fig. 4), which explains the lower MTBS values observed. On the contrary, TSS abrasion resulted in the creation of non-retentive sharp cracks which may act as stress concentration sites that could further weaken zirconia-resin bond strength [23]. Modified nanomechanical retention of SIE group was confirmed by the significantly higher bond strength of 45.9 MPa (4 weeks), while only 24.9 MPa (4 week) was found for TSS specimens bonded with same adhesive resin. Fracture mode for SIE group remained primarily cohesive in resin cement over testing intervals, meanwhile TSS group showed interfacial failure.

Composite resin cement including 10-MDP (10 methacryloyloxydecyl dihydrogen phosphate) are the recommended cement for zirconia restorations, because the chemical interaction between the hydroxyl groups of oxide ceramic and organophosphate ester monomer of MDP in cement [28].

Selective infiltration etching (SIE) change the low surface energy surface of zirconia to a highly active and retentive surface. A systematic review searched for highest bond strength values of adhesion to zirconia and found surface conditioning and MDP monomer based cement showed the

highest bond strength results (ranging from 17.1 to 42.3 MPa, depending on testing and aging method)[32].

Microtensile bond strength testing has many advantages over other bond strength test methods as applied load is vertical to the bonded area and small specimen size reduces the percentage of incorporation of structural defects, resulting in less scattering of data[33]. Microtensile bond strength test is a meticulous method, which requires investing time and effort, especially during preparing and cutting the specimens, to avoid damage of microbars. When combined with water storage as a type of artificial aging process, not only accurate determination of the initial bond strength value is obtained, but also the long –term performance of this bond may be predicted[34].

As the present data were obtained under controlled laboratory conditions, additional in vivo studies are needed to observe the long-term performance of the achieved bond strength. More investigations are required to optimize selective infiltration etching technique in terms of the resultant surface changes and technique simplicity and to optimize the properties of the used adhesive resin.

CONCLUSIONS

1- Selective infiltration etching provided superior micro-tensile bond strength results compared to tribo-chemical silica sandblasting.

2- Established bond strength by selective infiltration etching was not affected by artificial aging unlike tribo-chemical silica sandblasting that showed decreasing in bond strength values after 4 weeks water storage.

CLINICAL IMPLICATIONS

Heat induced maturation and selective infiltration etching produced a reliable and profound zirconia – resin bond strength resistant to artificial aging.

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