



## EFFECT OF DIFFERENT SURFACE TREATMENT ON BOND STRENGTH OF RESIN CEMENT TO ZIRCONIUM AND LEUCITE-REINFORCED GLASS CERAMIC<sup>‡</sup>

### ZİRKONYUM VE LÖSİTLE GÜÇLENDİRİLMİŞ CAM SERAMİKLERİN REZİN SİMANLA BAĞLANMA DAYANIMINA FARKLI YÜZEY İŞLEMLERİNİN ETKİSİ<sup>‡</sup>

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#### ABSTRACT

**Aim:** The aim of this study was to determine shear bond strength of resin cement to zirconium and leucite-reinforced glass ceramic after different surface treatment.

**Materials and Methods:** A total of 50 discs of zirconium and leucite-reinforced glass ceramic were prepared (10 mm in diameter and 1 mm in thickness) according to the manufacturer's instructions. Specimens were embedded in self-cure acrylic resin blocks. Specimens were divided into five groups randomly (n=5), and the following treatments were applied: 1. Air abrasion with aluminium-oxide particles (50 µm), 2. Acid etching with 9,5% hydrofluoric acid, 3. Co-jet, 4. Nd:YAG laser irradiation (1 mm distance, 100 mJ, 20 Hz, 2 W) and 5. Er:YAG laser irradiation (1 mm distance, 400 mJ, 20 Hz, 8 W). After the surface-treatment methods, Panavia F dual-cure resin cement were applied on the specimens. All the specimens were subjected to a shear test to evaluate their bond strengths. The data were analyzed with one-way variance analysis (ANOVA).

**Results:** In the leucite-reinforced glass ceramic specimens, the highest bonding values (12.06±1.4) was obtained in HF acid group. In the zirconium specimens, the highest bonding values (9.7±4.05) was obtained in CoJet group. However, adhesive failures were obtained in these groups. But, cohesive failures were obtained commonly in Co-jet groups.

**Conclusions:** The in vitro findings from this study indicate that surface-treatment procedures applied to leucite-reinforced glass ceramic and zirconium are important to bond strength of resin cement.

**Keywords:** Zirconium, leucite-reinforced glass ceramic, resin cement

#### ÖZ

**Amaç:** Bu çalışmanın amacı, farklı yüzey işlemlerinden sonra rezin simanın zirkonyum ve lösitle güçlendirilmiş cam seramiklerle olan makaslama bağlanma dayanımını değerlendirmektir.

**Gereç ve Yöntem:** Zirkonyum ve lösitle güçlendirilmiş cam seramikten toplamda 50 tane disk şeklinde (10 mm çap ve 1 mm kalınlığında) örnekler üretici talimatlarına uygun olarak hazırlandı. Örnekler self-cure akrilik rezine gömüldü. Örnekler rastgele 5 gruba ayrıldı ve şu yüzey işlemleri uygulandı: 1. Alüminyum oksit tozu ile kumlama (50 µm), 2. % 9.5' lik hidroflorik asit ile pürüzlendirme, 3. Co-Jet, 4. Nd:YAG lazer uygulaması (1mm mesafeden, 100 mJ, 20Hz, 2W), 5. Er:YAG lazer uygulaması (1mm mesafeden, 400 mJ, 20Hz, 8 W). Yüzey işlemlerinden sonra, örneklerin üzerine Panavia F dual-cure rezin siman yerleştirildi. Tüm örneklerle bağlanma dayanımlarını değerlendirmek için makaslama testi uygulandı. Elde edilen sonuçlar tek yönlü Varyans analizi ile değerlendirildi.

**Bulgular:** Lösitle güçlendirilmiş cam seramik örneklerde en yüksek bağlanma dayanımı değerleri (12.06±1.4) hidroflorik asit uygulanan grupta, zirkonyum örneklerde (9.7±4.05) ise Co-Jet uygulanan örneklerde belirlendi. Ayrıca, bu gruplarda adeziv kopmalar gözlemlendi. Fakat koheziv kopmalar yaygın olarak Co-Jet uygulanan gruplarda gözlemlendi.

**Sonuç:** Bu in-vitro çalışmanın bulguları zirkonyum ve lösitle güçlendirilmiş cam seramiklere uygulanan yüzey işlemlerinin rezin simanla olan bağlanma dayanımında önemli olduğunu göstermiştir.

**Anahtar Kelimeler:** Zirkonyum, lösitle güçlendirilmiş cam seramik, rezin siman

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## INTRODUCTION

Ceramics are still the most aesthetically pleasing existing materials in restorative dentistry. Although physical properties are good, biological and aesthetic compatibility can not be provided in metal-ceramic systems.<sup>1,2</sup> Metal-ceramic systems has proven the success of crown and bridge restorations. However, together with the increase of interest in aesthetic dentistry, development of alternatives of metal-ceramic restorations rapidly continuing.<sup>3</sup> Compared to metal-ceramic restorations, today full-ceramic restorations have been preferred more because of their aesthetic features and better biological features. Upon increasing aesthetic needs of people, use of porcelain restorations in posterior teeth has been vurrent issue. As an alternative to restorative techniques whose success has been proven for many years, new techniques are being developed every day.<sup>4</sup>

Ceramic is one of widely used materials in dentistry because of their inertness, color stability, high abrasion resistance, low thermal conductivity, biocompatibility and aesthetic properties.<sup>5,6</sup> Thermal conductivity and thermal expansion coefficient are very close to the values of enamel and dentin. High resistance against the pressures (350-550 MPa) but very low resistance to stress (20-60 MPa) ceramics' biggest disadvantage is easy fractures. Though fully successful results as metal bekked porcelain are not taken in clinical trials, studies about all ceramics are being carried on.<sup>5,7-11</sup> Compared with materials that have normal resistance distribution values, ceramic shows asymmetric resistance distribution and seen to be able to be broken under any force of maximum resistance value.<sup>12</sup> Unsupported ceramic materials are susceptible to breakage under tensile forces. Metal casting infrastructure provide ceramics with excellent mechanical support but form aesthetic problems.<sup>8</sup> Today, depending on progress in technology and materials, ceramic restorations are able to be cemented with adhesive systems and so ceramic restoration is reinforced by providing direct force transmission from restoration to tooth.<sup>8</sup> One of the techniques of strenghtening ceramic is to use infrastructures that are in color of tooth and more resistant to stretch instead of casting metal infrastructures. Studies on ceramic core structure have been carried out in order to strengthen ceramic, as well.<sup>12</sup>

Physical and chemical properties of dental ceramics have been tried to be improved by oxide added in their structure.<sup>7</sup> This new glass-ceramics, generally known also as oxide ceramics, are based on crystallized zirconium, magnesium or aluminum.<sup>6</sup> Zirconium has been begun to be used within porcelain material in dentistry due to its low grain diameter and high tensile strenght.<sup>4</sup> Optimum physical properties such as high flexural strength, and fracture toughness make convensional cements possible to use for zirconia-base crowns and FPD. But if zirconiabase systems are going to be used to restorate teeth with retention problems caused by short crown lengths, advantages of adhesive cementation can be useful.<sup>13</sup>

Clinical success of full ceramic restorations pasted with conventional cements reduces, also the microleakage seen in conventional cementation causes staining in crowns.<sup>14</sup> Resin cements have been widely used in recent years due to the fact that they increase the mechanical resistance of restorations and prevent microleakage.<sup>15-17</sup> Mechanical retention provided by surface procedures is the most important factor for sufficient bonding.<sup>18</sup> Recent studies show that there are problems about bonding in full ceramic restorations and in order to eliminate these problems, surface properties of ceramic materials are changed and bonding is being tried to be enhanced.<sup>18,19-26</sup> Though there are trails that show the effects of different surface treatments in various ceramics, there is no consensus about the best surface procedure that provide optimal bonding resistance as bound to cement and ceramic used in literature.<sup>27</sup>

Purpose of this study is to evaluate the bonding of resin cement to leucite-reinforced glass ceramic and zirconium applied different surface procedures.

## MATERIALS AND METHODS

The materials used in this study are shown in Table 1. 10 mm in diameter and 2 mm thick in accordance with the manufacturer's instructions 25 each samples were prepared from zirconium and leucite-reinforced glass ceramic for this study. Samples were embedded in self-cure acrylic resin to be 12 mm in diameter and 6 mm in length. Respectively 300 -, 600 -, 800 -, 1000 - and 1200 -of grit SiC abrasives were applied on sample surfaces. Samples were allowed to stand in ultrasonic cleaner filled with



distilled water for 5 min.

Table 1. Materials used in this study

Material	Manufacturer	Lot number
Zirconium	Kuraray Noritake Dental Inc.	054256
Leucite-reinforced glass ceramic	IPS Empress, Ivoclar Vivadent	S07664
Resin cement (Dual-cure)	Panavia F 20, Kuraray, Japan	041332
Acid (% 9.5 HF gel)	Bisco, BIDENT, USA	1100008120

Samples were divided into 5 groups randomly according to surface procedures to be applied. Surface procedures applied samples;

1. Acid etching (A group): 9.5 % HF acid was applied on sample surfaces for 20 sec. They were washed with pressured water for 20 seconds in order to remove acid and then dried.
2. Sandblasting (S group): they were sandblasted with fine-tipped sandblasting device (Macro Cab, Great Lakes Dental Products, USA) and 50 µm particle size of Al<sub>2</sub>O<sub>3</sub> sand under atmospheric air pressure from a distance of approximately 1 cm for 20 sec.
3. Co-jet (C group): 30 µm particle size of salinized Al<sub>2</sub>O<sub>3</sub> sand was applied with intraoral air-abrasion device and pressure of 30 psi from a distance of 1 cm for 15 sec.
4. Er-Yag laser (E group): Laser was applied from a distance of 1 mm for 20 seconds with water-cooling on Er-Yag laser unit (Smart, DEKA, Italy). Laser parameters: 400 mJ, 20 Hz, 8 W.
5. Nd-Yag laser (N group): Laser was applied from a distance of 1 mm for 20 seconds with water-cooling on Nd-Yag laser unit (Smartlife, DEKA, Italy). Laser parameters: 100 mJ, 20 Hz, 2 W.

After applying surface procedures, samples were cleaned with distilled water in ultrasonic cleaner for 5 minute. SEM analysis was made a samples from each group to see changes that occurred on sample surfaces as a result of surface procedures and SEM images were obtained. Dual-cure resin cement (Panavia-F) was applied on samples' surfaces (4 mm in diameter and 2 mm height) and polymerized with light.

Samples were respectively placed in a universal testing device to evaluate the resin cement bonding to zirconium and leucite-reinforced glass ceramic samples and the shear force was applied until the break and separation occurred.

Data were analyzed by Analysis of Variance

using SPSS package program (IBM SPSS for Windows, version 20.0) (Table 4)

## RESULTS

Means and standard deviations of the samples are shown in Table. 2. C group showed the highest fracture value while the N group showed the lowest fracture value among leucite-reinforced glass ceramic and zirconium samples.

Table 2. Means and standard deviations of groups

	Surface treatment	Mean	Std. Deviation
<b>Zirconium</b>	<b>Acid etching</b>	8,30	2,62
	<b>Co-jet</b>	9,70	4,05
	<b>Sandblasting</b>	9,22	4,81
	<b>Er:YAG laser</b>	7,56	4,50
	<b>Nd:YAG laser</b>	7,24	1,74
<b>Leucite-reinforced glass ceramic</b>	<b>Acid etching</b>	9,98	6,37
	<b>Co-jet</b>	12,06	1,40
	<b>Sandblasting</b>	9,46	3,27
	<b>Er:YAG laser</b>	6,22	2,12
	<b>Nd:YAG laser</b>	6,02	0,46

Whether there are differences between groups were evaluated with variance analysis. (Table. 3) In the analysis result, while no significant differences were observed depending on the material, the difference between groups were observed significantly ( $p < 0.05$ ) as a result of surface procedures. In addition, interaction between surface procedure and material was not significant.

Table 3. Results of ANOVA

	df	Mean Square	F	Sig.
<b>Material</b>	1	0,650	0,051	0,823
<b>Surface treatment</b>	4	30,596	2,397	0,04
<b>Inter- action</b>	4	7,837	0,614	0,655

Fracture types that occurred after the shear force had been applied were shown in Table. 4. According to this, the most observed fracture in all samples is adhesive fractures. In addition, cohesive fractures were observed more in both zirconium and

leucite-reinforced glass ceramic samples applied Co-jet. Adhesive fractures occurred completely in all samples applied laser.

Table 4. Number of the failure types after the shear test

	Surface treatment	Adhesive	Cohesive (Cement)
Leucite-reinforced glass ceramic	Acid etching	3	2
	Co-jet	-	5
	Sandblasting	3	2
	Er:YAG laser	5	-
	Nd:YAG laser	5	-
Zirconium	Acid etching	4	1
	Co-jet	1	4
	Sandblasting	4	1
	Er:YAG laser	5	-
	Nd:YAG laser	5	-

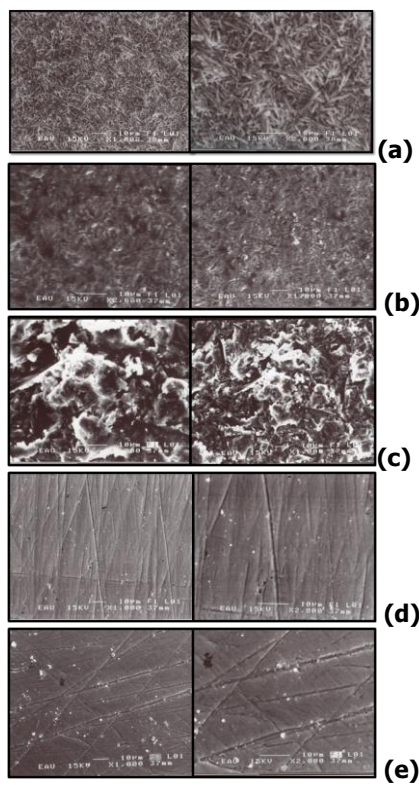


Figure 1. SEM observation of the IPS Empress ceramic after surface treatments at x1000 and x2000 magnification and bar marker indicate 10 µm. (a), hydrofluoric acid (9,5%) etching; (b), Co-jet (silanized Al<sub>2</sub>O<sub>3</sub> with 30 µm particle size); (c), sandblasting (Al<sub>2</sub>O<sub>3</sub> with 50 µm particle size); (d), Er-Yag laser application; (e), Nd-Yag laser application.

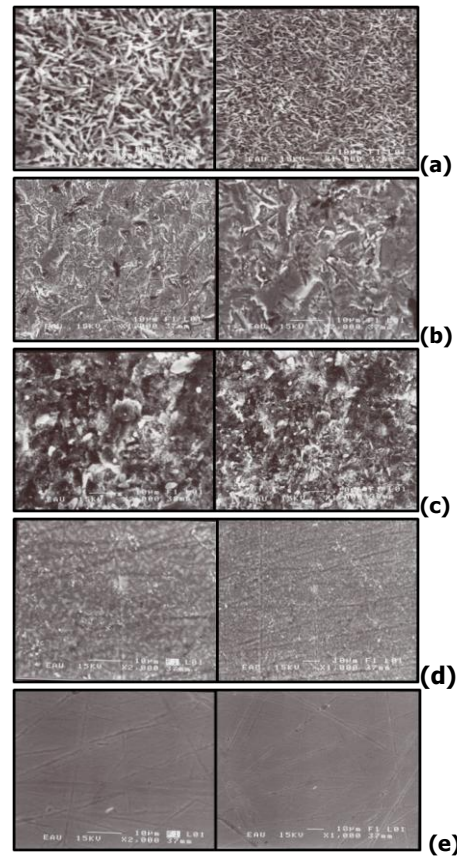


Figure 2. SEM observation of the Zirconium after surface treatments at x1000 and x2000 magnification and bar marker indicate 10 µm. (a), hydrofluoric acid (9,5%) etching; (b), Co-jet (silanized Al<sub>2</sub>O<sub>3</sub> with 30 µm particle size); (c), sandblasting (Al<sub>2</sub>O<sub>3</sub> with 50 µm particle size); (d), Er-Yag laser application; (e), Nd-Yag laser application.

## DISCUSSION

In the present study, C group showed the highest bond strength values in both zirconium and leucite-reinforced glass ceramic samples. N group showed the lowest bond strength values in both sample group.

Bond strength between ceramic surface and resin are based on micromechanical and chemical coupling that provide surface cleanliness and roughness. Several pre-processing procedure has been recommended and being used clinically in order to create micromechanical retentive ceramic surface.<sup>28-32</sup> Etching with HF acid solution can provide suitable surface properties and roughness.<sup>29-32</sup> HF acid provides roughness by dissolving glass phase and crystals on ceramic surface.<sup>33-39</sup> Estafen et al.<sup>40</sup> reported a good bond strenght in leucite-reinforced

glass ceramic samples applied 9.6 % HF acid for 60 seconds if microhybrid composite resin was used. In the present study as well, the highest bonding values were observed in H group after C group in leucite-reinforced glass ceramic samples. According to the Neis et al.<sup>41</sup> surface treatments (tribochemical, sandblasting, acid etching and burning) effect of micro tensile bond strength of feldspathic, lithium disilicate-reinforced and leucite-reinforced ceramics.

Lacy et al.<sup>42</sup> showed that sandblasting provided sufficient bonding strength. Kara et al.<sup>43</sup> reported that the samples applied sandblasting showed maximum surface roughness in their study. In the present study, the highest bonding values were observed in S group after C group among zirconium samples. Also, the highest bonding values among leucite-reinforced glass ceramic samples were observed in S group after H and C group.

Er: YAG laser affects tooth tissues by means of thermomechanical melting.<sup>44</sup> Changes on ceramic surface are also based on the ceramic that is used together with energy density in laser application.<sup>45</sup> Nd: YAG laser application is recommended to modify ceramic surfaces.<sup>46</sup> Shiu et al.<sup>45</sup> observed that 1 W power of Er: YAG laser applied feldspathic ceramic did not provide sufficient roughness connected with ceramic content. In the present study, N group showed the lowest bonding values after E group.

Akyıl et al.<sup>39</sup> found that the Er:YAG laser irradiation increased the resin cement bond strength to zirconium-oxide ceramic. In contrast with Akyıl et al.<sup>39</sup> findings, Stübinger et al.<sup>47</sup> observed that the Er:YAG laser at power output 10 W was not effective on Y-TZP surfaces. They concluded that the Er:YAG laser should not be recommended for the Y-TZP material because it could be emitted from the opposite surface. Likewise, Cavalcanti et al.<sup>48</sup> found that Er:YAG laser irradiation at 200mJ/pulse, 10 Hz, for 5 sec on Y-TZP surfaces provided a mild surface alteration effect between air abrasion with aluminum oxide and higher laser energies (400 and 600mJ/pulse, 10Hz). They concluded that Er:YAG laser irradiation at this power setting was a potential method of surface treatment for Y-TZP material. However, another study by Cavalcanti et al.<sup>48</sup> stated that an Er:YAG laser irradiation power setting of 200 mJ/pulse, 10 Hz for 5 sec did not improve the bond strength as well as air abrasion and it decreased the bond strength

compared to that of untreated surface. Akyıl et al.<sup>39</sup> it was found that Nd:YAG laser irradiation decreased the bond strength compared to that of untreated material. In the present study, laser irradiation decreased the bond strength compared with the CoJet system.

Determining fracture types and analysing them are very important in bonding tests.<sup>38</sup> In our study in 90% of the samples applied Co-jet, cohesive fractures were observed within resin cement while adhesive fractures were mainly observed in other sample groups. Also, 100% of adhesive fractures were identified in all samples applied the Nd: YAG and Er: YAG laser.

## CONCLUSION

Different surface treatment presented different changes on surface of substructure materials. While application of Co-Jet showed high bond strength between leucite-reinforced glass ceramic and resin cement, sandblasting showed high bond strength between zirconium and resin cement. Laser application had insignificant effect on bond strength of resin cement to both materials.

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