

A CONTROLLED CYCLIC LOADING ON THE SURFACE TREATED AND BONDED CERAMIC: STAIRCASE METHOD

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INTRODUCTION

Use of the ceramic restoration is increased by specific esthetics and biocompatibility, and new material and techniques are being developed continuously. Single-layer structures of esthetic ceramics are also commonly used as veneers, inlays, onlays and anterior single-unit crowns. However, abrupt catastrophic fracture by its brittleness is still problem in the oral cavity, so there has been attention on the strength of esthetic ceramics for the long-term survival rate.

The factors affecting the strength are such as material characteristics¹⁻⁴, lab processing⁵, surface treatment^{6,7}, cement type⁸ and oral environment. Because the crack propagation of ceramic rather depends on the surface quality than a thickness⁹, the method and quality of surface treatment affect the result so much.¹⁰⁻¹²

Usually ceramic needs surface treatment to increase the bonding strength, and etching or sandblasting is the most common surface treatment method.^{6-8,10,11} Although etching is known for increasing the bonding strength with evenly roughened surface, it depends on the type of ceramic¹³⁻¹⁵ and shows sensitivity to concentration.^{14,15} On the other hand, sandblasting is easy to use but the volume loss can be highly variable according to the blasting time¹⁶ and pressure in some substances and the severe surface change can be evoked because of its brittleness.^{7,10} Till now there are

several studies about the effect of etching and sandblasting on the bonding strength, but very little studies on the fracture resistance of bonded feldspathic porcelain in view of mastication driven crack development following surface treatment. In the bonding of ceramic experiment, contrary to flexure test, effect of flaw healing by resin and effect of dentin elasticity can be also expected and results can be more clinically relevant.¹⁷

The differences between the clinically failed ceramic crown and fractured ceramic crown in the lab test are well known (Fig. 1). *Hertzian cone crack*, characterized by the formation of a ring crack just outside the contact area that eventually grows into a sub-surface cone crack is common characteristics of usual lab test but is not seen in the clinically failed crown.¹⁸ Moreover, because traditional laboratory tests require high loads to failure, compared with low occlusal force measured during mastication, they are not clinically relevant.

The main reason of ceramic restoration failure in the oral cavity is the radial crack from the cement-ceramic interface.¹⁹ The radial crack has to be discriminated from the Hertzian cone crack occurring in the surface from high stress concentration, because the radial crack develops under very low load, remains latent in the interface, and fails to catastrophic fracture with repeated loading.¹⁹ In order to develop the radial crack, well controlled contact study is required and careful analysis is also needed, since

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Received June 3, 2008 Last Revision June 17, 2008 Accepted June 20, 2008.

※ This work was supported by grant no. 2006-0116 from Kangnung National University.



Fig. 1. Clinically failed all ceramic crown.

crack originates in the interface of bonded ceramic from cumulated fatigue. However, even in the cyclic loading experiment in the water environment on the bonded ceramic, induction of radial crack was not concerned and not described clearly before. To my knowledge, cyclic loading experiment to induce radial crack only, instead of cone crack, is not introduced ever.

The ball indenter is popular in dental literature, apparently widely thought to simulate clinical loading when applied to an anatomical occlusal surface. However, this type of indenter was proven to induce surface contact damage.¹⁸

Therefore, following conditions are requested to simulate clinically relevant fracture of ceramic in the lab: specimen bonded to dentin analog base having similar elastic modulus, indenter inducing tensile stress in the bonding interface, cyclic loading in the wet environment. This study was performed to evaluate fracture resistance of surface treated ceramic under above mentioned loading condition. Treatments with sandblasting and etching on Mark II ceramic surface were compared with polished surface in the bonded specimens. To get clinically relevant result, controlled cyclic conditions for radial cracking in the wet environment were designed and 500,000 cycles mean fatigue limit was calculated from the staircase method.

MATERIAL AND METHODS

Cerec Vitablocs mark II (Vita Zahnfabrik, Germany) blocks were cut into 1.1-1.2 mm thickness with a diamond saw (Isomet 1000, Buehler Ltd., IL, USA) and 60

specimens were prepared. Specimens were divided into 3 groups randomly and groups were polished (control), sandblasted, and etched group. Specimens of each group were gradually polished manually to # 600 roughnesses under water circulation, and this status, polished specimens were used as a control group. Specimens of sandblasted group were air blasted with 50 μm aluminum oxide particle at a 2 cm distance, under 2.7 bar pressure for 5 seconds. Sandblasted group had 20 μm more thickness for compensation of the volume loss from the blast procedure. This amount was set from the preliminary test. Specimens of etched group were treated with 9 % buffered hydrofluoric acid (Porcelain Etch, Ultradent Products Inc., Koln, Germany) for a minute. All specimens were cleaned in the ultrasonic cleaner and dried thoroughly before bonding. The final thickness of polished and etched group for bonded test were 1.0 ± 0.0 mm and 1.0 ± 0.02 mm for sandblasted group.

Support bases were made from a dentin-analog material having contact stress-strain behavior similar to hydrated dentin (woven glass fiber-filled epoxy; NEMA G10, International Paper, Hampton, SC, USA). Bases were 20 mm in diameter and 5 mm thick and modified by creating small channels and a well to allow water to access the cementation surface, simulating dentinal tubules (Fig. 3). The micro-channels were filled with accessory gutta-percha points so that they remained open following cementation.

Discs were cemented to bases between the measuring platens of a micrometer adjusting to achieve 50 μm cement thickness (Panavia 21 EX; Kuraray Medical Inc., Okayama, Japan). Discs were stored for 7 days in the water after 72 hour-setting in the ambient air. In order to eliminate air bubbles from the micro-channels of the base, a #30 endodontic K-file was used to remove trapped air.

Specimens were set in the water chamber and pre-loaded 10N compressive stress by Chewing simulator (Elf 3300, EnduraTEC Systems Co., MN, USA). Cyclic loadings were then delivered at a frequency of 15Hz using a haversine wave form (Fig. 4). Indenter was also made of G10 to avoid surface cracking with flat end, 3 mm diameter. The indenter but end of 1 mm, was reinforced by metal ring to prevent spreading out during cyclic loading. To avoid cone cracking by piston edge-loading, a 125 μm thick plastic tape was placed between the indenter and the ceramic surface. After



Fig. 2. Specimens were sectioned from Mark II blocks.



Fig. 3. Micro-channels, lateral channels and central well were prepared for water pumping effect.

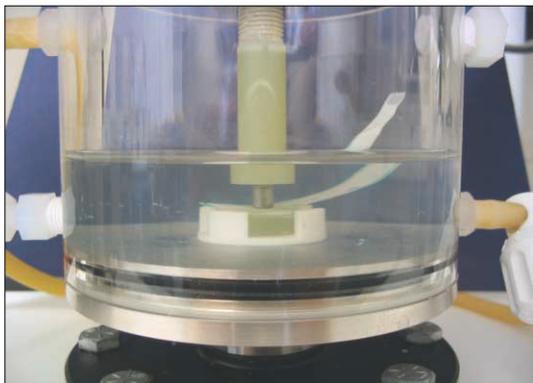


Fig. 4. Specimens were cyclically loaded in the wet environment.

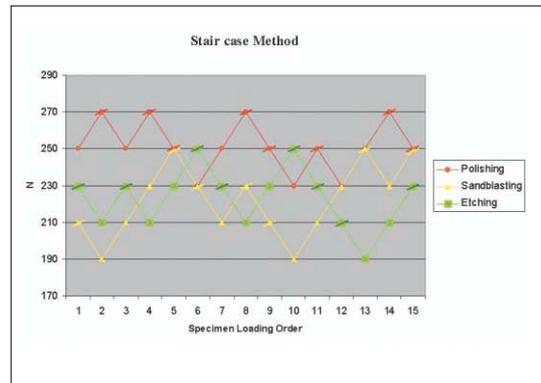


Fig. 5. Diagram illustrating the results of three groups. Fractured specimens are indicated by an oblique line. Indications without line are run-out ones.

completion of 500,000 cycles, specimen was examined under a microscope (Leica MZ95, Leica Microsystems Inc., IL, USA) using trans-illumination to confirm the radial crack and to screen for unwanted Hertzian cone cracking.

Staircase method²⁰⁻²² was employed to determine mean fatigue limit with a 20 N increment and 15 specimens for each test. If the specimen was not cracked after 500,000 cycles, the next specimen was tested at a load one increment higher. If the specimen was cracked, the next specimen was tested at a load one increment lower (Fig. 5). The initial load for the first specimen in each group was set from the preliminary experiment where it shows reversal point. To get initial load, 2-5 specimens were used separately before the test. Mean fatigue limit was calculated by the following

formula.²³

$$S_m = S_0 + d[A/N \pm 1/2]$$

Where, S_m = statistical estimate of mean fatigue load at prescribed life, S_0 = lowest load level at which the less frequent event occurred, d = step size, N = total number of less frequent events, A = sum of iN_i (Table I). The lowest load level at which fracture or non fracture occurred was designed as $i = 0$, the next as $i = 1$, and etc. The plus sign was used if the less frequent event was run-out, and the minus sign if the less frequent event was failure.

Standard deviation was calculated according to the following relationship.²³

Table I. Method for analyzing staircase test procedure data on the etched ceramic

Load (N)	i	n _i (less frequent event)	i n _i	i ² n _i
190	0	1	0	0
210	1	4	4	4
230	2	2	4	8
		N = ∑ n _i = 7	A = ∑ i n _i = 8	B = ∑ i ² n _i = 12

Table II. Failure load, coefficient of variation and significance

Group	N	Mean ± SD (N)	Median (N)	Coefficient of Variation	Grouping (α=0.05)*
Polished	15	251.43 ± 10.6	250.00	4.22%	A
Sandblasted	15	222.86 ± 23.42	223.76	10.51%	B
Etched	15	222.86 ± 14.16	221.58	6.35%	B

* Groups with different letters are significantly different.

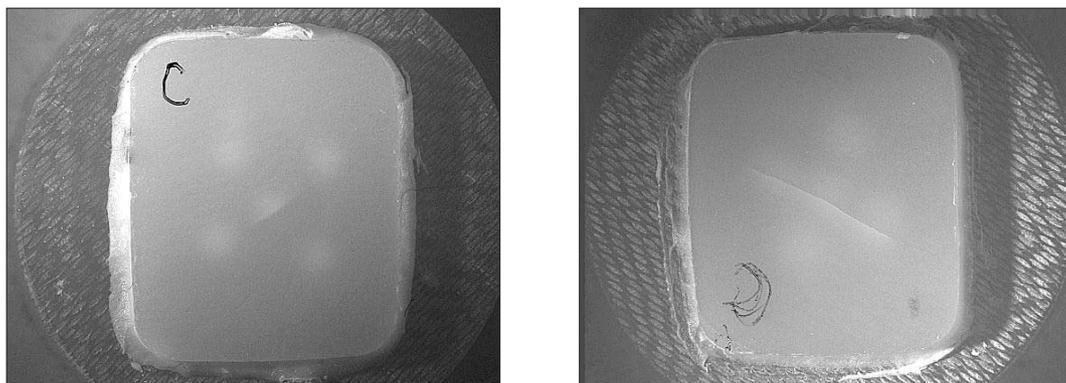


Fig. 6. Difference of crack length was demonstrated.

$$\sigma = 1.62 d[(NB-A^2)/N^2 + 0.029] \quad \text{if } (NB-A^2)/N^2 \geq 0.3$$

$$\sigma = 0.53 d \quad \text{if } (NB-A^2)/N^2 < 0.3$$

Where, σ = statistical estimate of standard deviation, d = step size, N = total number of less frequent event, A = sum of iN_i , B = sum of i^2n_i (Table I).

ANOVA was used for statistic analysis at the 95% confidence level for comparison of thickness of each group. A probit analysis of the data (PROC PROBIT in SAS) was used to estimate the median tolerance and Wald-type test statistics were used for pair-wise comparisons of the median tolerances, yielding *P*-values. Post hoc analysis was performed using Bonferroni test.

For comparison of flaw distribution and crack stability, coefficient of variation (CV) and latent crack propagation were also investigated.

RESULTS

Sandblasted group showed no significant thickness differences with other two groups (*P*<0.05).

Subsequent failure or run-out of specimens of three groups were shown in Fig. 5. Cracks were all radial crack at the bonded interface and there was no Hertzian cone crack.

Calculated mean fatigue limits and standard deviations of polished (control), sandblasted and etched group were 251.43 ± 10.6, 222.86 ± 23.42, 222.86 ± 14.16 N respectively (Table II). There was significant difference between polished group and sandblasted group (*P*=0.0055), polished group and etched group (*P*<0.0001). No significance was shown between sandblasted group and etched group (*P*=0.82).

CV was 10.51% for sandblasted group, and it was higher

than polished group (4.22%) and etched group (6.35%). That shows difference of flaw distribution between surface treated two groups but it was within normal range.

Of cracked specimens followed by cyclic loading, there were differences in the crack propagation after 24 hours. In contrast 7 of 8 for etched cracked specimens and 5 of 7 for sandblasted showed long crack, only 1 of 8 cracked specimens in the polished group showed long crack and rest of them was confined to the diameter of central micro-channel (Fig. 6).

DISCUSSION

In this study, influence of surface treatment on the fracture resistance, instead of on the bonding strength, was examined with controlled cyclic loading condition.

At present, some kinds of lack of realities in the analysis of mechanical ceramic strength are shown in the dental literature. Patterns of ceramic failure have been believed as a surface crushing of material. Although a bonded crown to die is used popularly to evaluate failure load, that method is meaningful only when the failure mechanism is coincided with that of clinically failed ceramic crown.

Kelly *et al.*^{24,25} and Thompson *et al.*²⁶ analyzed clinically failed ceramic restorations, concluding that failure originated from the inner surface of cemented crowns by cracks starting from the cement-ceramic interface. In most cases it appears that the stress state at initial crack pop-in results from compressive loads on the occlusal surface causing bending of the ceramic at its interface with the less-stiff cement and dentin.¹⁸ Latent radial crack that was developed in the interface at certain time of mastication is growing with repeated cyclic loading and fails catastrophically. Thus, test method evoking crack from the external surface of crown seems inadequate to evaluate the ceramic strength and prognosis.

In order to induce crack in the bonding interface, optimal volume of specimen, bonding, support with similar modulus of elasticity of dentin and indenter to avoid surface stress concentration are necessary. There has been attention in both clinical and laboratory literature regarding the effects of cements type and support material elastic moduli of on failure strength.^{8,27} In contrast the potential role of loading area on sub-surface stress distribution and failure

behavior has not been fully discussed.

Indenter contours along with any ceramic surface curvature determine the contact area under load. Contact area and load together determine surface contact stresses (i.e. pressure) as well as the stress distribution within the material; with both potentially influencing the failure patterns.¹⁸ The ball indenter is popular in dental literature, apparently widely thought to simulate clinical loading when applied to an anatomical occlusal surface. However, steel and tungsten balls typically used in dental literature (2 mm - 8 mm diameter) create point contacts having sub-millimeter radii against even a flat porcelain surface (less against a curved surface) and the first crack formed is typically Hertzian especially at ceramic thicknesses over 1 mm.²⁸ Unless balls approach diameters of around 400 mm to 1 m ball-on-flat contact areas are too small to simulate occlusal contacts, contact pressures quickly rise well above those measured clinically and failures originate from surface contact damage.¹⁹ In the study on the effect of sphere indenter, cone crack was developed under all the 0.8, 3.18, 8, and 20 mm diameter sphere indenter cases.²⁹ In that study, only one case of radial cracking before cone cracking was seen under the 20 mm diameter sphere. Authors stated that the indenter sizes must be sufficiently large to induce radial crack during loading. Tsai *et al.*²⁷ showed radial crack initiated within the bonded surface prior to Hertzian cone crack fracture with a flat indenter. Zhang *et al.*³⁰ used real-time monitoring method that is to use video record set under transparent specimen during cyclic loading but they reported surface cone crack only with sphere indenter.

In this study, Hertzian cone crack was not shown on the top surface with modulus of elasticity of base and indenter adjusted and with flat end indenter used. Under cyclic condition, radial crack shown in the interface of bonding surface proves the fatigue limit of this study is reliable.

Although ceramic is also affected by the internal flaw,⁶ the ceramic strength mainly depends on the surface flaws. Of course, glazed or polished surfaces show better strength than roughened surfaces,^{11,12} however ceramic requires surface roughening to increase the bonding strength through the surface treatment procedure.¹³ Moreover, since procedure of investment removal in the lab and/or fitting procedure in the clinic is usually done, ceramic strength decrease very much.^{3,6}

There are several studies about the effect of etched and sandblasted surfaces on the bonding strength, and also about the change of surface characteristics after surface treatment.^{13,16,31} Sandblasting was known to make many cracks on the surface^{6,7,10,16} and make poor fit of crown with irregular loss of material.¹⁶ Etching was known to remove surface microcrack of glass industry but that was not proven in the clinical study.³¹ Some authors rather reported the weakening effect of etching by the surface hydrolysis, or reported no strengthening effect even though it showed no weakening effect.^{14,15,31}

However, very little is known yet how the internal surface change influences the masticational strength in the bonded feldspathic porcelain. Even though Malament and Socransky^{19,32} showed the better success rate of etched Dicor crown than that of as formed crown, and emphasized the effect of etching, this result has to be believed from the viewpoint that flaws developed from the mild sandblasting in the devesting procedure were blunted with etching instead of the effect of etching itself.

Surface roughness is important on fatigue behavior too. Nakazato *et al.*¹² stated both the flexural and fatigue flexural strength increased with improvement of surface profile. Zhang *et al.*¹⁰ reported strengths of sandblasted specimens showed significant reductions in both dynamic and cyclic tests. Therefore masticatory strength could increase higher in the more polished, less etched and sandblasted surface. This weakening effect of surface treatment is obvious in the crack propagation difference of this study. Twenty four hours following cyclic loading, surface treated groups showed more crack propagation. That reveals sandblasting and etching makes more flaws in the surface.

In this study, fatigue limit of sandblasted group was equal to that of etched group but that was due to specimen sizes and has not to be considered they have equal strength after surface treatment. According to Kern and Thompson,¹⁶ sandblasting showed several mm loss of thickness in the various materials and when compared to 1-2 μm or 5-7 μm loss of thickness after etching, sandblasting would make different level of fracture resistance in the ceramic. Therefore identical fatigue limit of two groups was from coincidence and sandblasting might cause more deleterious stress decrease than etching. This explanation coincides with higher CV of sandblasted group than that of etched

group. Considering big different result under various sandblasting condition, more specimens are also needed to get more precise result in the staircase method.

Slow crack growth occurs in ceramics only when stress is applied in a wet environment, *in vitro* condition.^{33,34} Kelly¹⁹ reported that in the dry environment, result of cyclic test coincided with that of monotonic test. Long term cyclic loading and aqueous conditions cause fatigue of the ceramic up to 50 %.³⁵

Within the limit of this study, influence of surface treatment on the fracture resistance rather than on the bonding strength was examined with well controlled cyclic test method.

CONCLUSIONS

Despite homogeneous industrially manufactured blocks, fatigue limit of Mark II ceramic was low applicable to anterior restorations only. Fracture resistance of radial crack-inducing cyclic loading on the Mark II ceramic was relatively low compared to those of monotonic loading test, dynamic fatigue test or combined method, and that was comparable to the masticatory load in the oral cavity. This result explains why materials with high failure load in the lab monotonic test are fractured easily under the repeated low mastication force in the oral cavity. This study also showed flat end indenter was more ideal tool in the cyclic loading test to induce radial crack than sphere indenter, which concentrates stress on the surface. Because surface treatment such as sandblasting and etching either showed weakening effect from flaws and tensile cracks of bonding interface, inner surface treatment of ceramic crown under heavy occlusal force has to be done very cautiously.

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A CONTROLLED CYCLIC LOADING ON THE SURFACE TREATED AND BONDED CERAMIC: STAIRCASE METHOD

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STATEMENT OF PROBLEM: Effect of surface treatment of ceramic under loading does not appear to have been investigated. **PURPOSE:** The aim of this study was to investigate the effect of surface treatment of esthetic ceramic, which is performed to increase the bonding strength, on the fracture stress under controlled cyclic loading condition. **MATERIAL AND METHODS:** Sixty 1.0 mm-thick specimens were made from Mark II Vitablocs (Vita Zahnfabrik, Germany) and divided into 3 groups: polished (control), sandblasted, and etched. Specimens of each group were bonded to a dentin analog material base including micro-channels to facilitate the flow of water to the bonding interface. Bonded ceramics were cyclically loaded with a flat-end piston in the water (500,000 cycles, 15Hz). Following completion of cyclic loading, specimens were examined for subsurface crack formation and subsequent stress was determined and loaded to next specimen by the staircase method according to the crack existence. **RESULTS:** There were significant differences of mean fatigue limit in the sandblasted (222.86 ± 23.42 N) and etched group (222.86 ± 14.16 N) when compared to polished group (251.43 ± 10.6 N) ($P < .05$; Wald-type pair-wise comparison and post hoc Bonferroni test). Of cracked specimens, surface treated group showed longer crack propagation after 24 hours. All failures originated from the radial cracking without cone crack. Fracture resistance of this study was very low and comparable to failure load in the oral cavity. **CONCLUSION:** Well controlled cyclic loading could induce clinically relevant cracks and fracture resistance of Mark II ceramic was relatively low applicable only to anterior restorations. Surface treatment of inner surface of feldspathic porcelain in the masticatory area could influence lifetime of restorations.

KEY WORDS: Surface treatment, Ceramic, Sandblasting, Etching, Cyclic loading, Fracture

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Received June 3, 2008 Last Revision June 17, 2008 Accepted June 20, 2008.