Effects of thermal cycling and surface roughness on the Weibull distribution of porcelain strength

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The objective of this study was to test the hypothesis that thermal cycling weakens the flexural strength of porcelain. Specimens of Deguceram Gold and Vita Omega 900 were tested in four groups of 30 specimens each: in the original glazed condition *versus* being ground with 1000-grit, 600-grit, and 100-grit silicon carbide abrasives. Corresponding to these four types of surface treatments, four groups of 30 specimens per group received 5,000 times of thermal cycling. Flexural strength was measured using a four-point flexural test, and Weibull modulus was calculated. Within each type of surface treatment, the thermal cycling treatment did not result in any decrease in flexural strength although it caused the Weibull modulus to become smaller — except for the control and thermal-cycled groups of 600-grit surface treatment.

Keywords: Thermal cycling, Weibull modulus, Dental porcelain

INTRODUCTION

Ceramic restorations with a zirconium framework are often used because of their excellent esthetics and strength. They can also be used as a bridge component¹⁾. However, the mechanical strength of the veneering porcelain is not markedly different from that in metal-ceramic crowns. Consequently, fracture of the veneering porcelain with a zirconium framework is frequently reported^{2,3)}.

In a routine clinical procedure, occlusal adjustment typically accompanies the placement of ceramic restorations in the mouth. After adjustment, flaws will inevitably and inadvertently appear on the porcelain surface such that the latter requires new polishing. Nonetheless, it is difficult to completely remove the flaws and restore the original glaze of the porcelain surface. With the flaws still present on the porcelain surface, an undesirable result would be the occurrence of cracks. These cracks then propagate and finally culminate in the fracture of the porcelain surface following repeated occlusal loading.

In the oral cavity, saliva also plays a role in breaking the siloxane bonds and hence reduces the strength of ceramic restorations in a time-dependent manner⁴⁾. Besides, temperature changes in the mouth have a negative effect on promoting crack growth over a prolonged period. By the same token, reports have emerged on the effects of thermal cycling on the flexural strength of metal-ceramic complexes^{5,6)}, marginal adaptation of metal-ceramic restorations⁷⁾, and fracture strength of resin-bonded, all-ceramic fixed partial dentures⁸⁾. However, information is scarce on the effects of thermal cycling on dental porcelain.

Against this backdrop of information scarcity, the purpose of the present study was to test the hypothesis that thermal cycling weakens the flexural strength of porcelain and influences the Weibull distribution of porcelain strength.

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MATERIALS AND METHODS

Porcelain specimens

Two types of feldspathic body porcelain were used in this study: Deguceram Gold (Dentsply International Inc., PA, USA) and Vita Omega 900 (Vita, Bad Säckingen, Germany).

Porcelain powder was mixed with distilled water and poured into a metal mold (48×6×5 mm). Excess moisture was removed from the body porcelain with a tissue paper, and specimens were removed from the metal mold. A porcelain furnace (Austromat 3001, Dekema Dental-Keramiköfen GmbH, Freilassing, Germany) was used to fire the specimens. All specimens were fired twice, polished initially with 1000-grit abrasive by hand lapping under running water, and glazed according to manufacturers' instructions. The final dimensions of the specimens were approximately 40×4×3 mm, according to JIS (Japan Industrial Standard, R1601-1995) requirements.

Surface treatment

Specimens were divided into four groups of 30 specimens each as follows: in the original glazed condition (as-glazed group); ground with 1000-grit silicon carbide abrasive (1000-grit abrasive); ground with 600-grit silicon carbide abrasive (600-grit abrasive); and ground with 100-grit silicon carbide abrasive (100-grit abrasive).

To achieve the surface roughness obtained with 1000-grit silicon carbide abrasive, the porcelain specimens were polished with 28- μ m silicon carbide

combined with silicon (silicon rubber point, Shofu Inc., Kyoto, Japan). Similarly, to achieve the surface roughness obtained with 600-grit silicon carbide abrasive, the specimens were polished with 74- μ m silicon carbide combined with silicon, while that of 100-grit silicon carbide abrasive was achieved by using silicon carbide (Carborundum Point, Shofu Inc., Kyoto Japan). These levels of surface roughness matched the levels of roughness likely to occur following occlusal adjustment in the clinical situation⁹.

To evaluate surface roughness, arithmetic mean roughness (Ra) was measured using a surface roughness measuring machine with a contact-type stylus (Surftest SV-400, Mitutoyo Corporation, Kanagawa, Japan), according to JIS (Japan Industrial Standard, 0601-1994) requirements.

Thermal cycling

For each type of surface treatment, a corresponding group of 30 specimens were prepared to undergo 5000 times of thermal cycling using a thermal testing machine (Neocool BE200, Yamato Scientific Co. Ltd., Tokyo Japan; Thermominder SM-05R, Taitec Co. Ltd., Saitama, Japan). Specimens were immersed in water baths at temperatures of 5°C and 55°C for one minute each as one cycle.

Flexural strength test

A four-point loading test was used to compare the flexural strength of the thermal-cycled group against that of the control group which did not undergo thermal cycling (Fig. 1). The test was conducted using a universal testing machine (Autograph AG-500A, Shimadzu Corp., Kyoto, Japan) according to JIS recommendation. The flexural strength for each specimen group was then calculated using the following equation:

$$\sigma = 3P(L-l)/2bd^2 \quad (MPa)$$

where P is the breaking load, L is the lower test span, l is the upper test span, b is the width of the specimen, and d is the thickness of the specimen.

Flexural strength distributions were estimated based on Weibull distributions, which were calculated using the following equation:

$$Pf = 1 - \exp[-(\sigma/\sigma_{n0})^m]$$

where Pf is the fracture probability defined by the relation Pf = i/(N+1), *i* is the rank of strength, *N* denotes the total number of specimens, *m* is the shape parameter, also called the Weibull modulus, σ is the strength at a given Pf, and σ_{n0} is the characteristic strength.

The Weibull moduli were obtained using a computer program designed from the fracture data. For statistical analysis, one-way ANOVA and Tukey's multiple range test were employed at a significance level of 0.05.



Fig. 1 Schematic illustration of four-point flexural strength test.

RESULTS

Flexural strengths and Weibull distributions of Deguceram Gold specimens

For Deguceram Gold specimens, Table 1 lists their mean flexural strength values, the mean values of the specimens' final dimensions, and the mean surface roughness values. For both the control and thermalcycled groups, their flexural strengths decreased as the surface roughness of the porcelain increased. With the as-glazed group, the mean flexural strengths of both the control and thermal-cycled groups were significantly greater (89.9 MPa, 89.6 MPa) than all the other surface treatment groups (1000-grit abrasive, 600-grit abrasive, and 100-grit abrasive). With the 100-grit abrasive group, the flexural strengths of the control and thermal-cycled groups were significantly lower than the 600-grit abrasive groups (a decrease of 26.6 MPa for the control group and 11.6 MPa for the thermal-cycled group).

Within each surface treatment type, the only significant difference that occurred between the control and thermal-cycled groups was found in the 600-grit abrasive group.

Figure 2 shows the Weibull distributions of the control groups of Deguceram Gold for each type of surface treatment, with the probability of fracture plotted against the flexural strength for each specimen in the group. The Weibull modulus of the 1000-grit abrasive group (m=9.9) was slightly lower compared to the other surface treatment groups. On the contrary, the 100-grit abrasive group recorded a slightly higher Weibull modulus (m=10.8), although its flexural strength (56.2 MPa) was significantly lower than all the other surface treatment groups.

Figure 3 shows the Weibull distributions of the thermal-cycled groups of Deguceram Gold specimens. Results showed that the flexural strength distributions of the 1000-grit and 600-grit groups became similar to

Table 1 Flexural strengths of Deguceram Gold specimens

	As-glazed		1000-grit abrasive		600-grit abrasive		100-grit abrasive	
	Control	Thermal	Control	Thermal	Control	Thermal	Control	Thermal
Specimen width (mm)	4.02 (0.02)	4.00 (0.01)	4.02 (0.01)	4.01 (0.02)	4.08 (0.01)	4.01 (0.02)	4.01 (0.01)	4.02 (0.02)
Specimen thickness (mm)	3.03 (0.03)	3.01 (0.03)	3.00 (0.02)	2.98 (0.02)	3.05 (0.02)	2.98 (0.02)	2.99 (0.01)	2.97 (0.02)
Surface roughness (μ mRa)	0.04 (0.01)	0.07 (0.07)	0.08 (0.02)	0.08 (0.02)	0.16 (0.05)	0.14 (0.03)	0.82 (0.08)	1.07 (0.16)
Flexural strength (MPa)	89.9 (10.2) ^a	89.6 (10.0) ^a	84.1 (11.7) ^b	85.4 (9.82) ^b	82.8 (8.00) ^c	70.8 (7.67) ^d	56.2 (5.49) ^e	59.2 (6.88) ^e

Same superscript letters indicate no statistically significant difference by Tukey's multiple range test (p>0.05). Standard deviations are shown in parentheses.



Fig. 2 Cumulative Weibull distributions for the control groups of Deguceram Gold, where *m*: Weibull modulus.

that of the as-glazed group as a result of thermal cycling. On the overall, the Weibull moduli of the thermal-cycled groups were lower than their control groups for each surface treatment type — except for the 600-grit abrasive group which also recorded the highest Weibull modulus.

Flexural strengths and Weibull distributions of Vita Omega 900 specimens

For Vita Omega 900 specimens, Table 2 lists their mean flexural strength values, the mean values of the specimens' final dimensions, and the mean surface roughness values. For each surface treatment type, the flexural strengths of Vita Omega 900 specimens were higher than those of Deguceram Gold specimens. Nonetheless, a common trend was observed for both Vita Omega 900 and Deguceram Gold, in that the flexural strengths of both the control and thermalcycled groups tended to decrease with increase in surface roughness. Another similarity between Vita Omega 900 and Deguceram Gold was that the flexural strengths of both the control and thermal-cycled groups of 100-grit abrasive treatment were significantly lower than the 600-grit abrasive groups (a decrease of 38.0 MPa for the control group and 27.9 MPa for the thermal-cycled group).

Within each surface treatment type, no significant



Fig. 3 Cumulative Weibull distributions for the thermalcycled groups of Deguceram Gold, where m: Weibull modulus.

differences in flexural strength were observed between the control and thermal-cycled groups.

Figure 4 shows the Weibull distributions of the control groups of Vita Omega 900 for each type of surface treatment. The 600-grit abrasive group recorded the highest Weibull modulus (m=13.7), whereas the 100-grit abrasive group recorded the lowest (m=9.3).

Figure 5 shows the Weibull distributions of the thermal-cycled groups of Vita Omega 900 specimens. The flexural strength distributions of the as-glazed, 1000-grit abrasive, and 600-grit abrasive groups were similar to those of the corresponding thermal-cycled groups for Deguceram Gold. Moreover, the Weibull modulus of the 600-grit abrasive, thermal-cycled group (m=11.4) was almost the same as the corresponding Deguceram Gold group (m=11.3). On the overall, the Weibull moduli of the thermal-cycled groups were lower than their control groups for each surface treatment type — a trend also observed for the Deguceram Gold specimens.

Effect of thermal cycling on flexural strength

For both Deguceram Gold and Vita Omega 900, 5000 times of thermal cycling did not adversely affect the flexural strength for each surface treatment type although a significant difference was observed between

Table 2	Flexural	strengths of	Vita	Omega	900	specimens
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	As-glazed		1000-grit abrasive		600-grit abrasive		100-grit abrasive	
	Control	Thermal	Control	Thermal	Control	Thermal	Control	Thermal
Specimen width (mm)	4.05 (0.03)	4.26 (0.04)	4.06 (0.04)	4.03 (0.03)	4.03 (0.04)	4.04 (0.03)	4.06 (0.03)	4.06 (0.03)
Specimen thickness (mm)	3.02 (0.02)	3.19 (0.02)	2.95 (0.03)	3.00 (0.04)	2.95 (0.05)	2.93 (0.05)	2.94 (0.02)	2.95 (0.06)
Surface roughness (μ mRa)	0.03 (0.01)	0.04 (0.01)	0.09 (0.03)	0.11 (0.02)	0.21 (0.06)	0.13 (0.02)	1.10 (0.08)	1.08 (0.06)
Flexural strength (MPa)	112.9 (8.75) ^a	104.3 (12.4) ^a	105.4 (9.26) ^b	107.3 (13.6) ^b	101.0 (9.15) ^c	93.0 (8.72)c	63.0 (7.31) ^d	65.1 (7.15) ^d

Same superscript letters indicate no statistically significant difference by Tukey's multiple range test (p>0.05). Standard deviations are shown in parentheses.



Fig. 4 Cumulative Weibull distribution for the control groups of Vita Omega 900, where *m*: Weibull modulus.

the Deguceram Gold control and thermal-cycled groups for the 600-grit abrasive treatment.

DISCUSSION

For a comprehensive investigation on the flexural strength of feldspathic body porcelains, the specimens in this study received four kinds of surface roughening treatment comparable to those that were obtained following clinical occlusal adjustment. Besides, the effect of thermal cycling on porcelain was also examined in this study.

Results showed that the flexural strength of the as-glazed group was higher than the other surface treatment groups. It was thought that concomitant with high surface roughness, deep flaws were present in the porcelain surface and that these flaws became the origin points for fractures. On the other hand, some studies have found that glazing had no significant effect on the flexural strength or the Weibull modulus^{10,11}.

For the 100-grit abrasive specimens of both Deguceram Gold and Vita Omega 900, the flexural strengths of their control and thermal-cycled groups were significantly lower than those of the 600-grit abrasive groups. In the case of Deguceram Gold, the difference in surface roughness between the 600-grit



Fig. 5 Cumulative Weibull distribution for the thermalcycled groups of Vita Omega 900, where m: Weibull modulus.

abrasive and 100-grit abrasive control groups was merely 0.66 μ mRa — but the change in flexural strength was 14.6 MPa. Similarly, the difference in surface roughness between the 600-grit abrasive and 100-grit abrasive thermal-cycled groups was merely 0.93 μ mRa — but the change in flexural strength was 23.6 MPa. In the case of Vita Omega 900, the difference in surface roughness between the 600-grit abrasive and 100-grit abrasive control groups was 0.89 μ mRa — but the change in flexural strength was 30.0 MPa. As for the thermal-cycled group, a difference of 0.95 μ mRa in surface roughness was accompanied with a change of 39.5 MPa in flexural strength.

As seen from the results above, the greatest influence of surface roughness on flexural strength was found in the 100-grit abrasive group. This means that if a small scratch is present on the porcelain surface after clinical occlusal adjustment, the ceramic restoration will readily fracture. Porcelain has a high compressive strength and low tensile strength, and scratches on the tensile side will result in a higher incidence of failures.

Where a flaw exists on the porcelain surface, water will penetrate under the surface and break down the Si-O bonds, thereby causing the flaw to grow. It should be highlighted that moisture-assisted subcritical crack growth has a more deleterious effect¹²). A similar phenomenon occurs in the mouth because of the constant presence of saliva and the frequent changes in temperature, both of which contribute to crack growth. For this reason, a porcelain surface that has been roughened by occlusal adjustment should be polished and finished to a surface smoothness comparable to that of a glazed surface¹³⁾. Hence, upon excluding the 600-grit abrasive groups of Deguceram Gold, no significant differences in flexural strength were observed between the control and thermal-cycled groups. As for the 600-grit abrasive groups of Deguceram Gold, the statistically significant difference in flexural strength could be attributed to the lower surface roughness after thermal cycling.

However, studies have found that upon exposure to aqueous solutions, a hydroxyl layer was formed on the porcelain surface, which was built through alkali and hydroxyl exchange — thereby allowing the surface flaws to heal^{14,15}. If surface flaws were to heal, then the flexural strength would increase — but such an increase was not observed in the present study.

CONCLUSIONS

Tukey's multiple range test that followed ANOVA revealed significant differences among the four surface treatment groups. When subjected to 5000 times of thermal cycling, the specimens of all surface treatment types (namely, as-glazed, 1000-grit abrasive, 600-grit abrasive, and 100-grit abrasive groups) recorded lower Weibull moduli — except for the 600-grit abrasive groups of Deguceram Gold. On the effect of thermal cycling on flexural strength, the latter did not markedly decrease as a result — except for the 600-grit abrasive, thermal-cycled group of Deguceram Gold.

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