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ORIGINAL ARTICLE

Effect of Thermocycling on Flexural Strength and Weibull Statistics of Machinable Glass–Ceramic and Composite Resin

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Abstract To evaluate the durability of machinable dental restorative materials, this study performed an experiment to evaluate the flexural strength and Weibull statistics of a machinable lithium disilicate glass–ceramic and a machinable composite resin after being thermocycled for certain cycles. A total of 40 bar-shape specimens of were prepared with the dimension of 20 mm × 4 mm × 2 mm, which were divided into four groups of 10 specimens. Ten specimens of machinable lithium disilicate glass–ceramic (IPS e.max CAD, Ivoclar Vivadent, Liechtenstein) and 10 specimens of machinable composite resin (Paradigm MZ 100, 3M ESPE, USA) were subjected to 3-point flexural strength test. Other 10 specimens of each material were thermocycled between water temperature of 5 and 55 °C for 10,000 cycles. After that, they were tested using 3-point flexural strength test. Statistical analysis was performed using two-way analysis of variance and Tukey multiple comparisons. Weibull analysis was performed to evaluate the reliability of the strength. Means of strength and their standard deviation were: thermocycled IPS e.max CAD 389.10 (50.75), non-thermocycled IPS e.max CAD 349.96 (38.34), thermocycled Paradigm MZ 100 157.51 (12.85), non-thermocycled Paradigm MZ 100 153.33 (19.97). Within each material group, there was no significant difference in flexural strength between thermocycled and non-thermocycled specimens. Considering the Weibull analysis, there was no statistical difference of Weibull modulus in all experimental groups. Within the limitation of this study, the results showed that there was no significant effect of themocycling on flexural strength and Weibull modulus of a machinable glass–ceramic and a machinable composite resin.

Keywords Thermocycling · Weibull modulus · Lithium disilicate · Machinable composite resin

Introduction

The demand for esthetic dental restorations has increased in recent years resulting in rapid development of metal-free restorative materials. Since dental technology has been changing to digital era, metal-free dental restorations can be fabricated using a computer-assisted design/computer-assisted manufacture system (CAD/CAM). Materials used for CAD/CAM-fabricated restoration should have good machinability and resistance to machining damage [1]. Machinable restorative materials are manufactured in preformed blocks under well-controlled condition resulting in uniform microstructures and high reliability of the materials [2]. Machinable lithium disilicate glass–ceramic (IPS e.max CAD, Ivoclar Vivadent, Liechtenstein) is a common material for fabricating monolithic restorations. The flexural strength of fully crystalized lithium disilicate could exhibit up to 417 MPa. Because of its moderately high strength, posterior monolithic crown can be successfully fabricated using machinable lithium disilicate glass–ceramic [3]. Even though lithium disilicate restorations have been reported high clinical success rate especially in the single tooth restorations, chipping and fracture are still common complications since the materials have been challenging with various types of stresses such as mechanical fatigue, chemical irritants and thermal changing [4]. Subcritical crack growth is one of the factors that lead to strength degradation over time. The presence of

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water at the tip of a crack in ceramics under stress such as mechanical or thermal stress can accelerate the subcritical crack propagation lithium disilicate has been reported to have high susceptible to the subcritical crack propagation compared to other glass–ceramics leading to reduction of flexural strength over time [5]. Moreover, lithium disilicate glass–ceramics contain of two different phases: lithium disilicate crystals and amorphous glass, which have slightly different thermal expansion coefficient. Theoretically, when lithium disilicate are subjected to thermal change, the mismatch in the coefficient of thermal expansion between crystal contents and glassy matrix causes thermal stress at the crystal–matrix interface resulting in microcrack formation and causing the reduction of strength [6].

Unlike glass–ceramics, highly-filled composite resins exhibit high resistance to subcritical crack growth. In vitro, conventional composite resins with higher filler content and broader granulometric distribution showed stable in strength over time [7]. Machinable composite resins (Paradigm MZ 100, 3M ESPE, USA) contain 85 wt% ultra-fine zirconia-silica ceramic particles that reinforce a highly cross-linked polymetric matrix. These features contribute to the favorable mechanical properties; therefore, they can be used for fabricating single crowns [8]. However, there is lack of information regarding the degradation of strength of machinable composite resin over time. Machinable composite resins are composed of two major components having different thermal expansion coefficients: methacrylate-based matrix and zirconium oxide fillers [9]. Therefore, thermal aging could cause the internal stress leading to reduction of strength. Previous studies demonstrated the controversy in the degradation of conventional composite resin induced by water and thermal aging [10, 11] and recently introduced machinable composite resins have not yet been investigated.

Therefore, the objective of this study was to evaluate the effect of thermal aging on flexural strength of the machinable lithium disilicate glass–ceramics and composite resin. Also the reliability in strength after thermal aging of those materials was compared. Furthermore, a Weibull analysis was carried out to obtain two parameters, Weibull modulus (m), expressing the variation in the distribution of strength values, and the characteristic strength (σ_0), representing the stress that causes 63.2 % of the samples to fail [12].

Materials and Methods

Twenty specimens of machinable lithium disilicate glass–ceramic (IPS e.max CAD, Ivoclar Vivadent, Liechtenstein) and 20 specimens of machinable composite resin (Paradigm MZ 100, 3M ESPE, USA) were prepared for in in vitro study (Table 1). Bar-shape specimens were prepared with the dimension of 20 mm × 4 mm × 2 mm complying with the International Standard Organization (ISO) 6872:2008 for dental ceramics [13]. Specimens were cut using a precision diamond saw (Isomet 2000, Buehler Ltd., Lake Bluff, IL, USA) and polished using Ecomet Grinder Polisher (Buehler Ltd., Lake Bluff, IL, USA) with the 45 and 15 µm diamond disc and smoothen with the 1 µm diamond polishing paste (MasterMet, Buehler Ltd., Lake Bluff, IL, USA). Final dimensions were recorded with digital caliper (Mitutoyo, Mitutoyo America Corp., USA).

Ten specimens of each material were assigned as without thermocycling group (w/o TC) and subjected to a 3-point flexural strength test using a universal testing machine (Lloyd LRX-Plus, Lloyd Instruments Ltd., Fareham Hants, UK) equipped with a 1 KN load cell and a crosshead speed of 0.5 mm/min. Other 10 specimens of each material were assigned as with thermocycling group (TC) and thermocycled between water temperature of 5 and 55 °C. The soaking time in each water chamber was 30 s and the travelling time between two chambers was 15 s. After being thermocycled for 10,000 cycles, a 3-point flexural strength test was performed. Table 2 shows the treatment conditions and code used in this study.

The flexural strength was calculated using the following formula:

$$\sigma = 3PL/2wb^2$$

where P is the maximum load exerted on the specimen (N), L is the distance between the supports (mm), w is the width

Table 1 Lists of machinable restorative materials used in this study

Product	Material	Manufacturer	Code	Lot number
IPS e.max CAD	Lithium disilicae glass–ceramic	Ivoclar Vivadent, Liechtenstein	EMAX	K11234
Paradigm MZ 100	Resin composite with 85 wt% ultrafine zirconia–silica ceramic particles	3 M ESPE (USA)	MZ	2714A2-S

Table 2 Mean values, standard deviation and 95 % confidence interval (CI) for mean of the flexural strength

Material	Treatment	n	Mean (95 % CI for mean)	Standard deviation
EMAX	w/o TC	10	349.96 (322.54–377.39)	38.34
	TC	10	389.10 (352.79–425.41)	50.75
MZ	w/o TC	10	153.00 (138.72–167.29)	19.97
	TC	10	157.51 (148.32–166.71)	12.85

Table 3 Two-way analysis of variances for investigated parameters

Source of variation	Sum of squares	df	Mean square	F value	p value
Thermocycling treatment	4,761.342	1	4,761.342	4.131	0.050
Materials	459,131.328	1	459,131.328	398.356	0.000*
Thermocycling*Materials	2,997.573	1	2,997.573	2.601	0.116
Error	41,492.376	36	1,152.566		
Total	3,262,475.291	40			

* p value is significant at $p < 0.05$

(mm) and b is the height of the specimen (mm). Data were recorded and statistically analyzed using SPSS 16.0 for Windows (SPSS Inc., Chicago, IL, USA). Differences in flexural strength between the group with (TC) and without thermocycling treatment (w/o TC) were evaluated using two-way analysis of variances (ANOVA) and the Tukey multiple comparisons with a significance level of 5 %.

The variability of the flexural strength values was analyzed using the Weibull distribution function [14]:

$Pf = 1 - \exp(-\sigma/\sigma_0)^m$;

where Pf is the fracture probability, σ the fracture strength, σ_0 is the characteristic strength or scale parameter at the fracture probability of 63.2 % and m is the Weibull modulus or shape parameter of the distribution of strength data as a function of failure probability, which is the slope of linear fittings to the strength data when plotted in a lnln $(1/(1-Pf))$ versus ln (σ) graph [15].

Results

The means and standard deviations of flexural strengths of all experimental groups are listed in Table 2. Table 3 show the result of the two-way analysis of variance, there is a significant influence of the material factor ($p < 0.05$) on the flexural strength. However, the interactions between both parameters were not significant ($p > 0.05$). In EMAX group, there is no significant difference in flexural strength between TC and w/o TC treatment. Similar to EMAX, MZ shows no significant difference in flexural strength between TC and w/o TC treatment. However, both EMAX with TC and w/o TC treatment show significant higher flexural strength than MZ groups.

Table 4 shows the Weibull modulus (m) and characteristic strength (σ_0). The Weibull modulus are statistically different when the confidence bound values fail to overlap. Considering the Weibull modulus, there was no statistical difference in all experimental groups. For each material, the position and slope of the curve in the Weibull plot (Fig. 1) are determined by σ_0 and m values, respectively. The curve of a material that has a high m value is steeper than the curve of a material with low m value. In addition, the curve of a material with higher O_0 value, such as EMAX, is located more to the right of the curve of a material with lower O_0, such as MZ.

Table 4 Weibull modulus (m), 95 % confidence interval (CI) for m, characteristic strength (σ_0) and coefficient of correlation (r^2)

Material	Treatment	m (95 % CI for m)	σ_0	r^2	n
EMAX	TC	10.78 (8.86–12.70)	366.29	0.95	10
	w/o TC	8.55 (6.31–10.17)	411.52	0.95	10
MZ	TC	9.03 (7.38–10.68)	161.36	0.95	10
	w/o TC	14.21 (9.95–18.47)	163.25	0.88	10

m is statistically different when the confidence bound values fail to overlap

Discussion

In previous studies, flexural strength of machinable lithium disilicate ranged from 360 MPa [16] up to 440 MPa [6]. Mean flexural strength value of EMAX in this study fell into that range. EMAX contains 70 vol% of lithium disilicate crystals, which is considerable higher volume of crystal than other glass–ceramics. The final microstructure consists of highly interlocked lithium disilicate crystals leading to multiple crack deflection [17, 18]. MZ is a composite material containing 85 wt% ultra-fine zirconia-silica ceramic particles that reinforce a highly cross-linked polymetric matrix consisting of Bisphenol A-diglycidyl ether dimethacrylate and triethylene glycol dimethacrylate, and uses a patented tertiary initiator system. The particles have a spherical shape, and an average particle size of 0.6 µm. These features contribute to outstanding wear and favorable mechanical properties [9]. The fracture strength of Paradigm MZ 100 was significantly higher than feldspathic porcelain because of the higher resistance to tensile stress of polymer matrix in MZ itself [19, 20]. The standard deviations of this study were in the same range as other previous study on the strength of ceramic and composite resins [21–24]. Since these two materials, especially ceramics are brittle material presenting of the internal

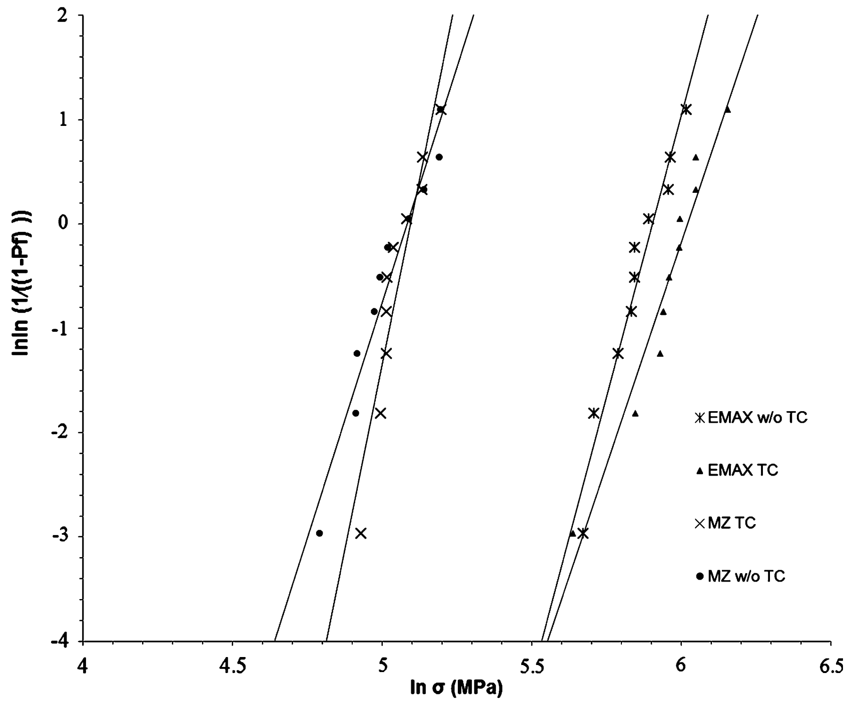

Fig. 1 Two-parameter Weibull plot for experimental groups

flaws. Therefore, the standard deviation usually presents in high value. However, this study included the Weibull analysis which is the suitable statistical analysis for imperfect materials.

Aging specimens using thermocycling treatment is a common protocol to evaluate the degradation of dental materials overtime. Also, immersing restorative materials in water either with or without thermocycling leads to slow crack growth that can weaken the flexural strength. Lithium disilicate has been reported to have high susceptible to slow crack growth after being aged by cyclic fatigue [5]. However, there is lack of data on the effect of thermocycling on the strength degradation of lithium disilicate. Thermocycling protocol used in this study was performed complying with the protocol used in previous literature, in which the exposure temperature ranged from 5 to 55 °C [25]. The 10,000 cycles of exposure can be referred as the thermal changing cycles that happened in the mouth for approximately one year [26]. Even though the thermocycling protocol does not simulate the real condition as in oral environment; at least, it can be used to evaluate the behavior of such materials when they are subjected to thermal stress [26]. Theoretically, the thermal expansion mismatch between ceramic crystals and glassy matrix resulted in tangential compressive stresses around the crystals, potentially responsible for crack deflection and strength increase [6]. Previous study on the microcracking in ceramic induced by thermal expansion has been reported to be an effective method on strengthening the ceramics [27]. For the composite resins, the results in this study showed no reduction of strength of MZ after being thermocycled. There is lack of data on the effect of thermocycling on the strength degradation of the complete polymerized machinable composite resins. However, previous study has reported that thermocycling treatment influenced the reduction of srength of the conventional indirect composite resins [28]. In this study, the null hypothesis has been accepted since the effect of thermocycling on the flexural strength of EMAX and MZ was not significant. Microcrack formation caused by thermal stress did not exhibit the strengthening effect in this study. The reason for that could be explained as EMAX and MZ contain large amount of reinforcing fillers so the predominant strengthening effect came from crack bridging and crack deflection [9, 18].

Weibull distribution function is considered to be an acceptable approach in mechanical design procedure of ceramic components to evaluate the reliability of the materials. It should be noted in almost every experimental study on fracture statistics of ceramics [14, 15, 29]. The distribution of strength values for dental ceramic and composite materials is usually fitted to Weibull distribution [30]. Weibull modulus (m) or shape parameter has important practical implications in Weibull distribution. A high value of m indicates a close grouping of fracture stress values whilst a low value indicates a wide distribution with a long tail at low stress levels [14, 15, 31]. A higher m, meaning higher reliability of materials, is usually preferable to a lower m associated with a higher mean flexural strength. In this study, m values of all groups were in normal range of restorative materials [31]. There is no significant difference in m values between w/o TC and TC groups for EMAX and MZ. Therefore, thermocycling treatment did not alter the reliability of materials in this study.

Conclusions

This study demonstrated that the thermocycling treatment had no significant effect on the degradation of flexural strength of both machinable lithium disilicate glass–ceramic and composite resins. Machinable lithium disilicate showed the highest flexural strength regardless of the themocycling treatment. The reliability of materials was evaluated using Weibull analysis and the result showed that there is no change of the Weibull parameters of both materials after being thermocycled. Therefore, machinable lithium disilicate glass–ceramic and composite resin used in this study exhibited high reliability even they were aged by themocycling treatment.

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