Original Article

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Fracture resistance of four dental computer-aided design and computer-aided manufacturing glass-ceramics

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Objective: The objective of this study was to compare the flexural strength of four dental computer-aided design and computer-aided manufacturing (CAD/CAM) glass-ceramics, i.e., lithium-disilicate-based (IPS e.max CAD; EMX), leucite-based (IPS Empress CAD; EMP) and two zirconia-reinforced lithium silicate glass-ceramics (CELTRA DUO; CD and VITA SUPRINITY; VS).

Materials and methods: Thirty bar-shaped specimens (1.5mm x 4mm x 18mm) were prepared for each material by cutting from rectangular blocks, and finishing their surfaces by glazing. All specimens were loaded to fracture at a crosshead speed of 0.5 mm/min using a three-point bending fixture mounted on a universal testing machine. The flexural strength values were calculated and statistically analyzed by one-way ANOVA and Dunnett T3 (α=0.05). The strength data of all groups were also analyzed using the Weibull Statistics.

Results: The result from a statistical analysis showed that VS (319±42MPa) had the highest flexural strength following by CD (278±49MPa) and then EMX (236±20MPa). EMP (157±14MPa) showed the lowest flexural strength. The Weibull moduli and the characteristic strength were 9.02 and 336.97 for VS, 6.55 and 298.87 for CD, 13.90 and 244.69 for EMX, 12.77 and 164.02 for EMP.

Conclusions: For CAD/CAM glass-ceramics examined in this study, the zirconia-reinforced lithium silicate glass-ceramics had higher flexural strength and characteristic strength than a lithium-disilicate-based and leucite-based glass-ceramics.

Key word: flexural strength, leucite-based, lithium-disilicate-based, three-point bending, zirconia-reinforced lithium silicate

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Introduction

All-ceramic restorations are widely used due to their acceptable mechanical properties and esthetics [1,4]. Lithium-disilicate-based glass-ceramic is an all-ceramic material currently used in the fabrication of single- and multi-unit dental restorations. It can be customized to match the appearance of natural teeth such as color and translucency. This material is fabricated by the hot-pressing technique. For IPS e.max Press (Ivoclar Vivadent, Schaan, Liechtenstein), its microstructure consists of approximate 70% lithium-disilicate crystals ($Li_2Si_2O_5$), which are embedded in a glassy matrix. IPS e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein) was introduced in 2005 for a computer-aided design and computer-aided manufacturing (CAD/CAM) technique [5].

Leucite-based glass-ceramic consists of a glassy matrix and leucite crystals. The glass matrix is based on an alumino-silicate glass. Leucite crystal is used to reinforce a glass-based material and to improve its mechanical properties. IPS Empress CAD (Ivoclar Vivadent, Schaan, Liechtenstein) is a CAD/CAM machinable ceramic. It was introduced in 2006 with a flexural strength of

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about 160 MPa and designed to be used either with chairside or laboratory-side CAD/CAM systems. This material can be used for veneers, inlays, onlays, and anterior crowns.

For zirconia-reinforced lithium silicate glass-ceramics, CELTRA DUO (Dentsply, Hanau-Wolfgang, Germany) was introduced in 2012 and VITA SUPRINITY (VITA Zahnfabrik H. Rauter GmbH&Co.KG, Bad Säckingen, Germany) was introduced in 2013. These materials contain a large number of very fine-grained lithium crystallites, which high glass content gives the material its excellent optical and mechanical properties. Their compositions contain approximate 10 percent by weight of ZrO_2. It has a comparable flexural strength to lithium-disilicate-based ceramic. They can be used for single-unit anterior and posterior crowns, inlays, onlays and veneers. VITA SUPRINITY's strength is approximate 420 MPa by three-point flexural method. Weibull modulus is approximate 8.9.

The Weibull distribution is the most widely used function in describing strength data of ceramics. Ceramic strength data are generally not normally distributed, but often skewed in the high strength portion [6]. The Weibull characteristic strength is the strength occurring at a probability of failure at 63.2% [7]. The Weibull modulus (*m*) is the parameter describing the shape of the distribution of strength as a function of failure probability [8] Higher values of Weibull modulus relate to greater materials structural reliability, consistent flaw distribution and their strengths would be narrowly distributed.[7] The Weibull moduli of most ceramics are reported in the range of 5-15 [9].

The objective of this study was to compare the flexural strength of leucite-based (IPS Empress CAD), lithium-disilicate-based glass-ceramic (IPS e.max CAD) and zirconia-reinforced lithium silicate glass-ceramics (CELTRA DUO and VITA SUPRINITY) using three-point bending method. The characteristic strength (σ_0) and Weibull modulus (*m*) of the materials were determined using the Weibull statistics.

Materials and methods

Specimen preparation

Four dental ceramics (IPS e.max CAD, IPS Empress CAD, CELTRA DUO and VITA SUPRINITY) were examined in this study. Thirty ceramic bar-shaped specimens were fabricated following the manufacturer's instructions for each ceramic. Bar specimens were cut into the dimension of 1.5 mm × 4 mm × 18 mm using a low-speed diamond-coated disk with water cooling. The four sharp edges of the specimens were chamfered to the length of between 0.09 to 0.15 mm and at 45° angle with a 600-grit SiC paper. IPS e.max CAD and VITA SUPRINITY were further crystallized according to the firing program provided by the manufacturer. All bars were coated by their glazing materials and fired according to their firing schedules to obtain glazed surfaces which were used as a control surface condition for all experimental groups.

The three-point bending test

For the flexural strength (σ_f) measurement, all specimens were loaded in three-point bending at a cross-head speed of 0.5 mm/min using a universal testing machine (LF Plus Lloyd instruments, Ametek. Inc., USA). Failure loads were recorded and the flexural strength values were calculated using the following equation [1].

$$\sigma_f = \frac{3Pl}{2wb^2} \quad [1]$$

Where σ_f is the flexural strength (MPa), *P* is the load at fracture (N), *l* is the center-to-center distance between the support rollers (mm), *W* is the width of the specimen (mm), *b* and is the thickness of the specimen (mm). The flexural strength values of all groups were statistically

analyzed using one-way ANOVA and Dunnett T3 at the significance level of .05.

The Weibull modulus *(m)* and the characteristic strength (σ_0) were calculated relative to the cumulative probability of failure (P_f) as shown in Equation [2]. [6]

$$P_f = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right] \qquad [2]$$

The fractographic analysis

The fractured specimens were ultrasonically cleansed in 35% ethanol solution for 5 minutes and mounted on a metal stub. They were coated with a 5-nanometer-thin gold-palladium using a sputter-coating machine (SPI Module®, SPI Supplies, Structure Probe Inc., Canada). Then, the crack initiation flaw of each specimen was located and the patterns of the fractured markings were observed using a light microscope (Nikon SMZ1000, Japan).

Results

The mean flexural strengths of all groups are summarized in Table 1. The flexural strength values of the all groups were significantly different from each other. VITA SUPRINITY had the highest flexural strength while IPS Empress CAD possessed the lowest strength. The two estimated parameters of the Weibull statistics, the modulus (*m*) and the scale (σ_0) of all groups are described in Table 2. The Weibull probability plots of four dental ceramics are demonstrated in Fig1. Even the characteristic strength of VITA SUPRINITY was also the highest but its modulus (*m*) was lower than that of IPS e.max CAD. CELTRA DUO had the lowest modulus (*m*) value.

Discussion

The CAD/CAM system is a popular processing technique that is used for fabricating

Table 1. The flexural strength of the four experimental groups.

Material	Flexural Strength (MPa)
VITA SUPRINITY	319.0 ± 42.8[a]
CELTRA DUO	278.9 ± 49.7[b]
IPS e.max CAD	245.3 ± 23.5[c]
IPS Empress CAD	157.1 ± 14.9[d]

Values with different superscript letter are statistically different ($P < 0.05$).

Table 2. Weibull modulus and characteristic strength of the four experimental groups.

Material	Weibull modulus (m)	Characteristic strength (σ_0)
VITA SUPRINITY	7.2	336.9
CELTRA DUO	5.3	298.8
IPS e.max CAD	9.7	254.7
IPS Empress CAD	10.4	162.4

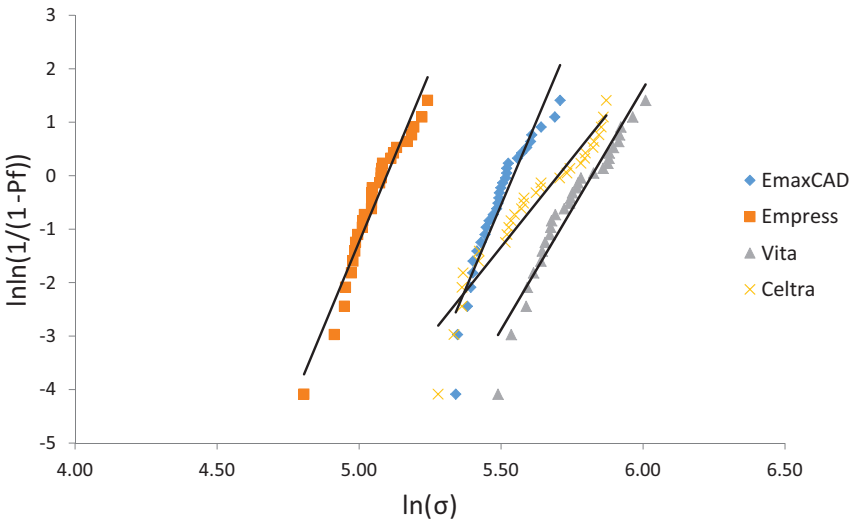

Figure 1. The Weibull probability plots of four dental glass-ceramics.

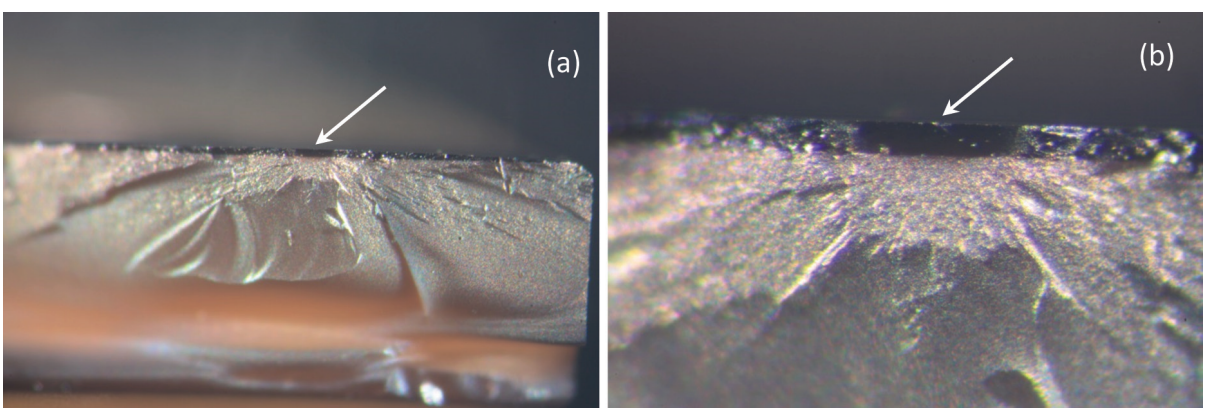

Figure 2. The representative fracture surfaces of VITA SUPRINITY dental ceramic showing the failure origin on the surface at 30x (a) and 120x (b)

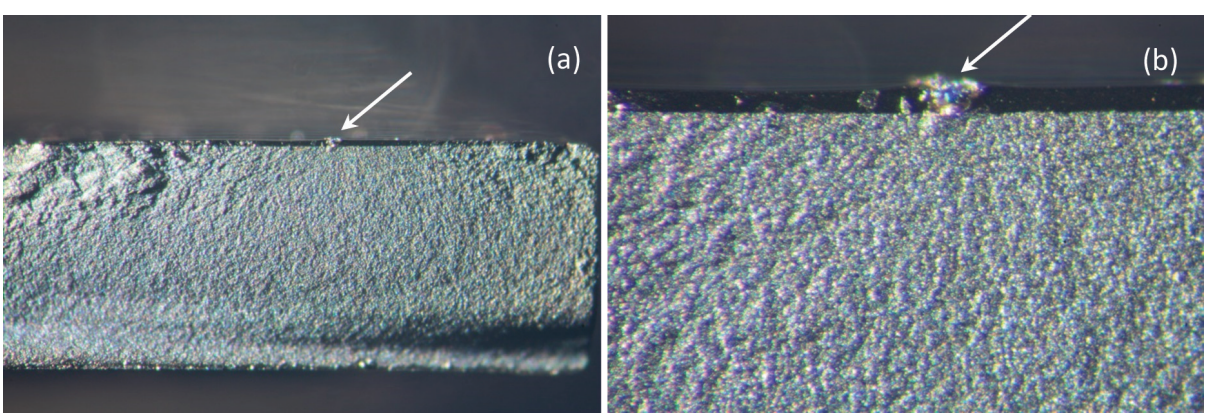

Figure 3. The representative fracture surfaces of IPS e.max CAD dental ceramic showing the failure origin on the surface at 30x (a) and 120x (b)

numerous of all-ceramic restorations in dentistry. One of the main advantages of using CAD/CAM is its fast processing speed and many types of restoration can be delivered in one-visit appointment. The prefabricated blocks that used to fabricate dental restorations are made from their manufacturers. Therefore, flaws and defects formation would be less compared with the laboratory-made restorations [10].

A new dental ceramic material is developed and aimed to match the appearance of natural teeth such as color, translucency. It should have excellent mechanical strength and toughness to obtain structural reliability during dental functions [11]. New innovation and technique such as addition of small and tough crystal particles is frequency used. Zirconia-reinforced lithium silicate glass-ceramics (CELTRA DUO and VITA SUPRINITY) are examples of these new dental ceramic materials. According to the manufacturers, these materials offer mechanical strengths ranging from 370 to 420 MPa which are comparable to lithium-disilicate-based glass-ceramics (IPS e.max Press and IPS e.max CAD) [12].

Flexural strength is a significant mechanical property that is frequently used to evaluate the fracture resistance of brittle materials. [9] In this study, the flexural strength of leucite-based glass-ceramic, lithium-disilicate-based glass-ceramic and zirconia-reinforced lithium silicate glass-ceramics were determined. The results showed that the flexural strength values of these materials are significantly different from each other. VITA SUPRINITY had the highest strength (319 MPa) which was lower than those obtained from a manufacturer and a previous study [13]. Its

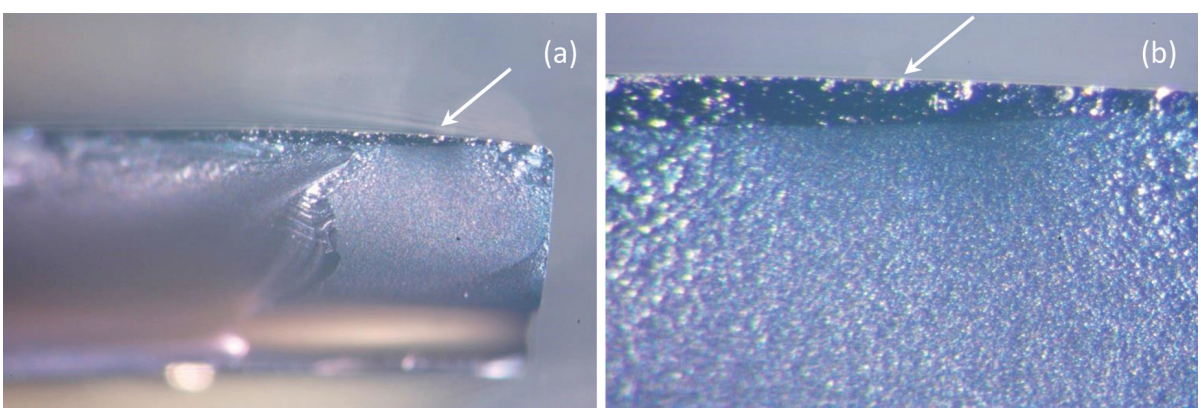

Figure 4. The representative fracture surfaces of CELTRA DUO dental ceramic showing the failure origin on the surface at 30x (a) and 120x (b)

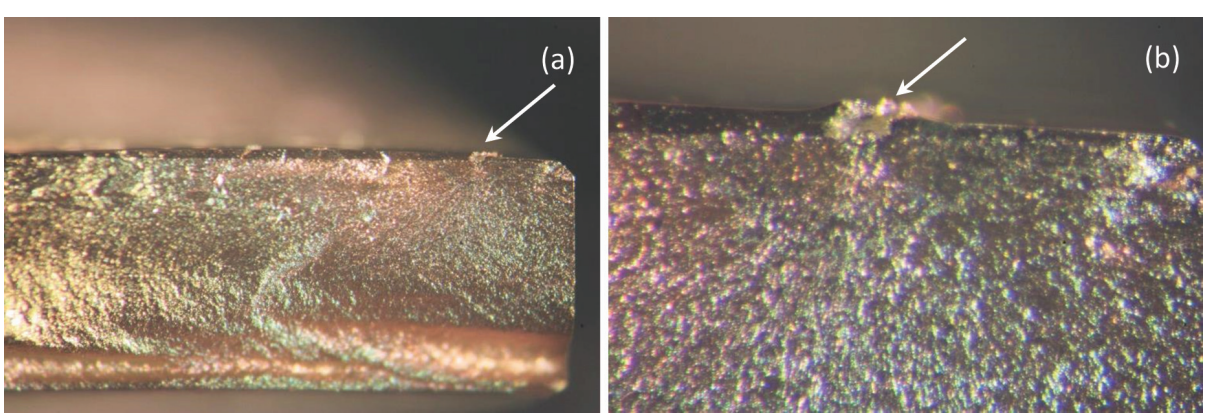

Figure 5. The representative fracture surfaces of IPS Empress CAD dental ceramic showing the failure origin on the surface at 30x (a) and 120x (b)

strength was higher than that of CELTRA DUO which had comparable composition and microstructure. A lithium-disilicate-based glass-ceramic (IPS e.max CAD) had lower strength than those of zirconia-reinforced lithium silicate glass-ceramics but its strength was higher than that of leucite-based glass-ceramic (IPS Empress CAD).

From the fracture surface analysis, the failure origins of most specimens were at the glazed surfaces (Fig.2-5). For zirconia-reinforced lithium silicate glass-ceramics, the glaze layer of these materials was more homogeneous and resulted in a very small failure origin when compared with a lithium-disilicate-based glass-ceramic. A large defect in the glaze layer of a lithium-disilicate-based glass-ceramic could result from the heterogeneity of material compositions or porosity created during glazing material application. This large defect in the glaze layer was also observed in leucite-based glass-ceramic (IPS Empress CAD). This large defect in the glaze layer might be the reason for the lower strength for dental glass-ceramic materials. Glazing is a procedure supposed to increase the mechanical strength of all-ceramic restorations due to the reduction in porosity and surface flaws [14]. However, the major fault occurred in this step is the formation of porosities during condensation procedure or from an inappropriate powder/liquid mixing ratio [15]. These porosities could be the failure origin sites [16] and this idea was confirmed in this study especially when the glaze layer was thick. In this study, the thicknesses of the glaze layer ranged between 40-60 μm.

After crystallization, zirconia-reinforced lithium silicate glass-ceramics consist of a glassy matrix and very fine lithium metasilicate and lithium disilicate crystals with submicron-size ranges and high glass content [12,17]. Because of this high glass content, the fractured surfaces of zirconia-reinforced lithium silicate glass-ceramics were clearly observed with evident fracture markings. Unlike a lithium-disilicate-based glass-ceramic which had larger crystals and lower glass content, its fractured surface was rough and it was more difficult to observe the fracture origin and markings.

The flextural strength of IPS e.max CAD obtained in this study was closely to that reported by Albero A. et al [18]. but it was lower than that reported from Coldea A et al [19]. This difference could be attributed from the specimen preparation with or without glazing for these studies. The flexural strength of IPS Empress CAD was comparable to the results obtained from previous works [18, 20, 21].

The Weibull modulus and the Weibull characteristic strength are generally used to describe the cumulative probability of failure corresponded to the failure stresses of dental ceramics. The characteristic strength (σ_0) is the strength at a probability of failure of 63.2 %. The Weibull modulus (*m*) is a parameter demonstrating the shape and width of strength distribution as a function of failure probability. Materials with high Weibull moduli are more predictable and less likely to break at a stress much lower than a mean value. A typical Weibull modulus for ceramics is reported to be 5-15 [9,13].

In this study, the two estimated parameters, the Weibull modulus (*m*) and the characteristic strength (σ_0) were determined. Ideally, a clinically reliable dental ceramic should tolerate high chewing forces during a long service period. This means that it can be used for a posterior restoration (high loading area) and survived for many years of service (excellent reliability). In order to accomplish these goals, materials with high characteristic strength (σ_0) and Weibull modulus (*m*) should be utilized. For four CAD/CAM dental glass-ceramics investigated in this study, the Weibull modulus was the highest for IPS e.max CAD, while VITA SUPRINITY possessed the highest characteristic strength. The values obtained for these two parameters were not consistent for any material. Therefore, there should be long-term clinical

researches to assure the benefit of use of these materials corresponding to their clinical applications claimed by the manufacturers.

Conclusion

Within the limit of this study, these conclusions could be drawn;

For CAD/CAM glass-ceramic blocks, the zirconia-reinforced lithium silicate glass-ceramics had higher flexural strength and the characteristic strength than a lithium-disilicate-based and leucite-based glass-ceramics.

The characteristics of a glaze layer had an effect on the flexural strength of all dental glass-ceramics used in this study.

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