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UNIFORMED SERVICES UNIVERSITY OF THE HEALTH SCIENCES

POSTGRADUATE DENTAL COLLEGE
AIR FORCE POSTGRADUATE DENTAL SCHOOL
2133 PEPPERRELL ST
JBSA-LACKLAND, TEXAS 78236



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Name of Candidate: Daniel A. Reid, Capt, USAF, DC
Master of Science
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THESIS/MANUSCRIPT APPROVED:

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Wen Lien, Col, USAF, DC
Director, Dental Materials Evaluation and Testing
USAF Dental Research and Consultation Service, Ft. Sam Houston, TX

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Christopher J. Raimondi, Lt Col, USAF, DC
Co-Director, Dental Materials Evaluation and Testing
USAF Dental Research and Consultation Service, Ft. Sam Houston, TX

VANDEWALLE.KR
AIG.STEPHEN.113
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Digitally signed by
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Date: 2022.05.20 09:44:05 -05'00'

Kraig S. Vandewalle, Col (ret), USAF, DC
Director of Dental Research, Advanced Education in General Dentistry Residency
Air Force Postgraduate Dental School, JBSA-Lackland TX

Flexural Fatigue Behavior of New All-Ceramic CAD/CAM Materials

Capt Daniel Reid, DDS¹; Col Wen Lien, DDS, MS, MS²;
Lt Col Christopher Raimondi, DDS, MS, MS²;
Col (ret) Kraig Vandewalle, DDS, MS¹

¹USAF Postgraduate Dental School, JBSA-Lackland, TX;

²Dental Research and Consultation Service, JBSA-Ft. Sam Houston, TX

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Objective: The purpose of this study was to compare the flexural fatigue behavior of new CAD/CAM materials to established materials on the market. **Methods:** Novel lithium-disilicate/lithium-aluminum silica-based (Tessera, Dentsply/Sirona) and gradient zirconia oxide-based ceramic materials (4Y-PSZ/5Y-PSZ: IPS e.max ZirCAD MT Multi, Ivoclar Vivadent; 3Y-TSP/5Y-PSZ: IPS e.max ZirCAD Prime, Ivoclar Vivadent) were compared to more traditional lithium-disilicate silica-based (IPS e.max CAD, Ivoclar Vivadent) and zirconia oxide-based ceramic materials (4Y-TZPs: IPS e.max ZirCAD MT, Ivoclar Vivadent; Katana STML, Kuraray; YZ ST, VITA). Thirty beams were sectioned from blocks or milled from discs for each group with final dimensions of 4.0×1.3×18.0mm. Beams were created in the transition zone for gradient zirconia oxide-based materials. Three groups of Tessera were tested: not glazed and regular fired, glazed and regular fired (Programat P500), or glazed and speed fired (CEREC SpeedFire, Dentsply/Sirona). After polishing, each specimen underwent 3-point bend testing using a universal testing machine (ElectroPuls E3000, Instron). Flexural strength and flexural modulus were determined with 15 specimens per group. Flexural fatigue strength, Weibull modulus, and characteristic strength were determined with the remaining 15 specimens using the staircase method (100,000 cycles/20 Hz). Data were analyzed with ANOVAs/Tukey tests

($\alpha=0.05$). **Results:** Significant differences were found between the groups ($p<0.05$). Within the silica-based materials, IPS e.max CAD exhibited significantly greater flexural strength, and greater Weibull modulus and characteristic strength compared to Tessera. However, there was no significant difference in their flexural fatigue strength. Within the oxide-based materials, VITA YZ ST had significantly greater flexural strength and flexural fatigue strength, and greater Weibull modulus and characteristic strength. **Conclusions:** The zirconia oxide-based materials exhibited higher values for all properties evaluated compared to the silica-based materials tested. While the flexural fatigue strength of the new Tessera ceramic was comparable to IPS e.max CAD, it had a significantly lower ability to withstand higher static forces as indicated by its lower flexural strength value.

Background

Driven by demands for efficiency, productivity, and naturalistic esthetics, “direct” digital manufacturing seems to become a new paradigm for delivering high-quality dental restorations. Rapidly prototyping a ceramic restoration has led to the popular success of same-day, single crown delivery. Today, various ceramic prostheses can be designed and milled with computer-aided design and computer-assisted milling (CAD/CAM). One popular material of choice for same-day delivery is the lithium-disilicate reinforced glass ceramic, particularly IPS e.max CAD (Ivoclar Vivadent, Amherst, NY). Introduced in 2006 as a monolithic block with a flexural strength reported to be as high as 500 MPa,⁽¹⁾ IPS e.max CAD has been shown in a systematic review to have a five-year cumulative survival rate of 97.8%.⁽²⁾ However, debates have ensued over the limitations of IPS e.max CAD for single and multi-unit restorations in posterior heavier stress-bearing areas.

In the early 2000's, zirconia debuted in dentistry to replace metal substructures for veneered porcelain. However, chipping of the veneered porcelain became a significant complicating factor in the longevity of zirconia restorations.(3, 4) To overcome this problem, full-contour zirconia restorations were marketed with a flexural strength as high as 1400 MPa.(5) Zirconia is the strongest all-ceramic material available on the market, but its lack of translucency made it difficult to use in esthetics cases as a monolithic material.(6)

In its pure form, zirconia can exhibit three phases; monoclinic, tetragonal, and cubic. While the stability of these phases is dependent on increasing temperature, it is possible to achieve each phase at room temperature by adding stabilizing oxides. Small amounts of oxides, or dopants like yttrium oxide (Y_2O_3) are added to zirconia, stabilizing its tetragonal phase and preventing it from transformation into the monoclinic phase after sintering. Of all the restorative ceramics, 3 molar percent Y_2O_3 stabilized tetragonal zirconia polycrystal (3Y-TZP) is the most clinically durable.(6) However, most 3Y-TZP materials on the market lack the esthetics of competitive glass-ceramics and are therefore somewhat restricted to the posterior region or frameworks.(6) To increase translucency, new formulations of zirconia oxide were developed. Formulating new translucent dental zirconia materials involved increasing the Y_2O_3 content, which introduced the cubic phase along with the tetragonal phase in the resulting zirconia-oxide materials.(5) The presence of specific percentages of tetragonal and cubic phases defines the mechanical and physical properties of the zirconia. Using a higher Y_2O_3 content produces a partially stabilized zirconia with greater cubic content. For example, the amount of Y_2O_3 dopant in molar concentration that is used in zirconia is abbreviated as 3Y-TZP for 3 mol% Y_2O_3 ,

4Y-TZP for 4 mol% Y_2O_3 , or 5Y-TZP for 5 mol% Y_2O_3 .⁽⁵⁾ In these new zirconia materials, the quantity of the cubic phase increases from 15% in 3Y-TZP materials to approximately 25 vol% in 4Y-TZP materials and up to 50 vol% in 5Y-TZP materials. This increased cubic phase improves the translucency of the zirconia materials but diminishes their strength as compared to traditional 3Y-TZP zirconia materials.^(4, 7) 3Y-TZP has a high flexural strength of 900 to 1400 MPa.^(4, 5) 4Y-TZP has a flexural strength of 600 to 900 MPa and 5Y-TZP has a flexural strength of 700 to 800 MPa.^(4, 7)

A distinct property of the inherently strong 3Y-TZP zirconia is the ability to resist fracture. This property is called transformation toughening. The transformation of zirconia's metastable tetragonal phase to the monoclinic phase allows for this toughening, which increases a zirconia restoration's resistance to fracture.^(4, 8) The more translucent materials have smaller amounts of tetragonal phase (75% in 4Y-TZP and ~50% in 5Y-TZP), leading to a reduced possibility of tetragonal to monoclinic transformation and therefore less transformation toughening.^(4, 7)

From a long-term clinical standpoint, the main factor for fracturing a material is the repeated loading placed on the material during use. The preexisting fractures and defects are propagated through the intermittent loading below the conventional fracture strength of the material. This leads to eventual failure despite occlusal forces applied to the restoration that are well below the maximum strength of the material.^(9, 10) A recent study by Holman et al., using the staircase method found that 4Y-TZP and 5Y-TZP cubic-containing zirconia materials demonstrated greater or similar strength degradation compared to the tetragonal 3Y-TZP materials. The authors concluded that as a result of the fatigue testing and increased degradation in some of the cubic-containing products,

care should be taken with the utilization of these cubic-containing materials in posterior multi-unit fixed dental prostheses.(11)

New material are introduced by manufacturers every year. One example is the new lithium-disilicate and lithium aluminum silicate (Virgilitite) glass ceramic material called Tessera, by Dentsply Sirona (Charlotte, NC). The manufacturer claims that this new material has a firing cycle of only 4.5 minutes in the CEREC SpeedFire furnace (Dentsply Sirona) and a flexural strength greater than 700 MPa.(12) Novel zirconia materials have recently been introduced by Ivoclar Vivadent using gradient technology. Gradient technology is a unique manufacturing process that uses special powder conditioning to combine 3Y-TZP and 5Y-TZP (IPS e.max ZirCAD Prime) or 4Y-TZP and 5Y-TZP (IPS e.max ZirCAD MT Multi) oxide ceramic powders. Unlike traditional multi-layered zirconia dental materials on the market which may only have layers of color, the unique gradient technology reportedly offers a seamless progression of strength, with 3Y-TZP or 4Y-TZP in the dentin zone, and 5Y-TZP in the incisal or occlusal translucent zone for esthetics.(4, 13) VITA YZ ST (VITA, Bad Sackingen, Germany) is a new 4Y-TZP with a reported flexural strength of 850 MPa and adequate esthetics.(14)

No research has been published evaluating the flexural fatigue behavior of these novel silica- and oxide-based ceramic materials. The purpose of this study was to investigate the flexural strength and the effect of various flexural stresses on the time-to-failure relationship of varying new CAD/CAM materials and to compare those properties to the performances of the more established all-ceramic materials on the market. Furthermore, the life of the CAD-CAM materials at normal-use conditions can be predicted based on the life-stress relationship obtained from conducting accelerated life

tests. The null hypotheses tested were that there would be no difference in flexural strength, flexural modulus, flexural fatigue strength, Weibull modulus or flexural characteristic strength between the different ceramic materials.

Materials and Methods

This study evaluated the flexural strength, flexural fatigue strength, and survival probability for two silica-based and five zirconia oxide-based materials. The two silica-based materials were the lithium disilicate, IPS e.max CAD (Ivoclar Vivadent), and lithium disilicate/lithium-aluminum silicate, Tessera, (Dentsply Sirona). The five zirconia-oxide materials were 4Y-TZPs (IPS e.max ZirCAD MT, Ivoclar Vivadent; Katana STML, Kuraray America, New York, NY; VITA YZ ST), multi-layer gradient combination of 4Y-TZP and 5Y-TZP (IPS e.max ZirCAD MT Multi, Ivoclar Vivadent), and multi-layer gradient combination of 3Y-TZP and 5Y-TZP (IPS e.max ZirCAD Prime, Ivoclar Vivadent).

Specimen Preparation

Thirty specimens were prepared for each ceramic material. A CAM (computer-aided manufacturing) machine (I-Mes iCore 450i, Eiterfeld, Germany) was used to mill the zirconia beams out of the zirconia blanks. The beams were designed using CAD (computer-aided design) software (DS, SolidWorks, Waltham, MA) and the file was imported into milling software (Sum 3D, iCAM V5, I-Mes, iCore). For the two zirconia materials containing a gradient, IPS e.max ZirCAD MT Multi and Prime, the beam specimens were aligned in the center of middle gradient transition zone. The surface of each zirconia specimen was polished with 1000-grit polishing paper before sintering. The

final size of the beam specimens was 4.0 mm in width, 1.3 mm in depth, and 18.0 mm in length after sintering in a furnace (Programat S1 1600, Ivoclar Vivadent) according to the manufacturer's instructions. The IPS e.max CAD beam specimens were cut from blocks using a precision saw (IsoMet 5000, Buehler, Lake Bluff, IL) and crystallized in a furnace (Programat P500, Ivoclar Vivadent) according to the manufacturer's instructions. Ninety Tessera beam specimens were cut from blocks using the IsoMet 5000, and separated into three groups: not glazed or glazed and fired in the Programat 5000 furnace, or glazed specimens fired in a high-speed furnace (CEREC SpeedFire, Dentsply/Sirona), according to the manufacturer's instructions. The surface of each IPS e.max CAD and Tessera specimens were polished with 600-grit polishing paper.

Flexural Strength

Flexural strength testing was completed in accordance with the international standard for ceramic materials ISO 6872:2015 (International Organization for Standardization).⁽¹⁵⁾ Fifteen beam specimens per group were fractured in a universal testing machine (ElectroPuls E3000, Instron, Norwood, MA). Each specimen was placed on a 3-point bending-test device, which was constructed with a 15.0 mm span length between the supporting rods. See Figure 1. A central load was applied with a head diameter of 2.0 mm at a crosshead speed of 1.0 mm/minute. The flexural strength was obtained using the equation $FS = 3Fl/2bd^2$, where F was the loading force at the fracture point, l was the length of the support span (15 mm), and b was the width and d the depth of the beam specimen. Measurements were made using electronic digital calipers (GA182, Grobet Vigor, Carlstadt, NJ). The flexural modulus was determined from the

slope of the linear region of the load-deflection curve using analytical software (Excel, Microsoft Corp, Redmond, WA).

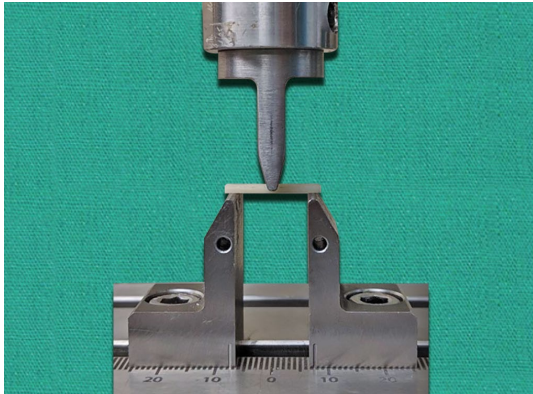


Fig. 1: Specimens were placed on a 3-point bending device for flexural strength and flexural fatigue strength testing

Flexural Fatigue Strength

The flexural fatigue strength (σ_{fr}) of fifteen beams from each group was determined for 100,000 cycles at 20 Hz per group. Each specimen was placed on the 3-point bending test device as was done with flexural strength testing. See Figure 1. The staircase method was used for the evaluation.(11, 16) For each group, the starting force value was determined by using 1/2 of the maximum flexural strength. The amplitude (stress alternating) was determined by using 1/2 of the standard deviation of the maximum flexural strength. Tests were be conducted sequentially with force applied values increasing or decreasing by 20% of initial starting force value whether the previous beam resulted in failure or survival. The flexural fatigue strength and standard deviation was determined using the following equations:

$$\sigma_{ff} = X_0 + d \left(\frac{\sum in_i}{\sum n_i} \pm 0.5 \right)$$

$$SD = 1.62d \left(\frac{\sum n_i \sum i^2 n_i - (\sum in_i)^2}{(\sum n_i)^2} + 0.029 \right)$$

X_0 was the lowest stress level considered in the analysis and d was the fixed stress increment. To determine σ_{ff} , the analysis of the data was based on the least frequent event (failures vs survivals). The negative sign was used when the analysis was based on failures; otherwise the positive sign was used. In the second equation, the lowest stress level considered was designated $i = 0$, the next $i = 1$, and so on, and n_i was the number of failures or survivals at the given stress level.(11, 16)

Weibull Statistical Analysis

The Weibull statistical analysis predicts the time-to-failure/probability of failure of a component. It is a way to obtain the Weibull modulus (m) and the characteristic strength (σ_0). (17, 18) The characteristic strength is the strength at a failure probability of 63.2%. (17, 18) The Weibull modulus is used to determine the level distribution of strength and weakness properties of a material. (17, 18) The Weibull's 2-parameter (σ_0 and m) distribution function is:

$$P_f = 1 - e^{-\left(\frac{\sigma}{\sigma_0}\right)^m} \quad (1)$$

where P_f is the cumulative probability of failure, σ [MPa] is the flexural strength, σ_0 [MPa] is the Weibull characteristic strength, and m is the Weibull modulus.

The two parameters, σ_0 and m , are calculated by using rank order statistics. For example, after the flexural strength for all 15 specimens were measured, the flexural

strength (σ) values were ranked in ascending order and were assigned a probability of failure per each specimen based on its ranking using the following equation:

$$P_f(\sigma_i) = \frac{i-0.5}{N} \quad (2)$$

where i represents 1, 2, 3, ..., N of the i^{th} datum in ascending order of the flexural strength (σ_i), and N is the total number of specimens tested. Next, equation (1) is rearranged into the following equation:

$$\ln \left[\ln \left(\frac{1}{1-P_f} \right) \right] = m \ln \sigma_i - m \ln \sigma_0 \quad (3)$$

Then, $\ln \left[\ln \left(\frac{1}{1-P_f} \right) \right]$ is plotted on the ordinate against $\ln \sigma_i$ on the abscissa, from which a linear regression model can be estimated. One can use either a least squares method or a maximum likelihood estimation procedure for the linear regression model. The slope and intercept of equation (3) yield m and $m \ln \sigma_0$ respectively. Then, σ_0 is solved from the intercept. When the characteristic strength is the strength value, the cumulative probability of failure, P_f , is at 63.2%. (17, 18)

Statistical Analysis

The mean and standard deviation was determined for flexural strength, flexural modulus, and flexural fatigue strength for each of the groups. Also, a mean was determined for Weibull modulus and characteristic strength for each of the groups. Data were analyzed using a Kaplan-Meier survival test and one-way ANOVAs followed by Tukey *post hoc* tests ($\alpha = 0.05$) for each of the tested properties.

Results

The one-way ANOVA found significant differences between groups ($p < 0.05$). Within the silica-based materials, IPS e.max CAD exhibited significantly greater flexural strength (384.14 ± 32.30 MPa) compared to Tessera (not glazed, regular fired: 206.47 ± 46.54 MPa; glazed, speed fired: 231.93 ± 35.97 MPa; or glazed, regular fired: 243.61 ± 35.10 MPa). The flexural modulus of IPS e.max CAD (103.15 ± 9.51 GPa) was significantly greater than the Tessera glazed, speed fired (85.77 ± 3.36 GPa) and glazed, regular fired (88.13 ± 5.31 GPa), but not significantly different from Tessera not glazed, regular fired (99.20 ± 5.19 GPa). There was no significant difference in the flexural fatigue strength between IPS e.max CAD (185.42 ± 36.05 MPa) and Tessera (not glazed, regular fired 140.40 ± 12.18 MPa; glazed, speed fired 148.27 ± 23.55 MPa; and glazed, regular fired 131.48 ± 37.34 MPa). IPS e.max CAD had the greatest Weibull modulus (14.05), which was greater than all of the silica-based groups (range: 5.13 - 7.92) and oxide-based groups (range: 5.95 – 10.37). VITA YZ ST had the greatest characteristic strength (926.53) which was greater than all of the tested materials (range: 224.60 – 789.75). All of the tested properties of the silica-based materials were significantly less than the oxide-based materials ($p < 0.05$).

Within the oxide-based based ceramic materials, VITA YZ ST had the greatest flexural strength (883.40 ± 101.23 MPa) and flexural fatigue strength (647.83 ± 73.33 MPa), which were significantly greater than all of the other materials. IPS e.max ZirCAD Prime had the lowest flexural strength (584.47 ± 102.03 MPa), but it was not significantly different from IPS e.max ZirCAD MT Multi (621.19 ± 127.45 MPa). IPS e.max ZirCAD MT had the greatest flexural modulus (203.34 ± 12.71 GPa), but it was not significantly

different from any of the other oxide-ceramic materials, except for Katana STML (191.64 ± 10.97 GPa). See Table 1.

Group	n	Flexural			Flexural			Flexural Fatigue Strength			Weibull	Characteristic	
		Strength (MPa)	SD		Modulus (GPa)	SD		(MPa)	SD		Modulus	Strength (MPa)	
Tessera	NG RF	15	206.47	46.54	e	99.20	5.19	CD	140.40	12.18	h	5.13	224.60
	G SF	15	231.93	35.97	e	85.77	3.36	E	148.27	23.55	h	7.56	246.85
	G RF	15	243.61	35.10	e	88.13	5.31	DE	131.48	37.34	h	7.92	258.65
IPS e.max	CAD	15	384.14	32.30	d	103.15	9.51	C	185.42	36.05	h	14.05	398.41
IPS e.max ZirCAD	Prime	15	584.47	102.03	c	196.22	12.63	AB	411.22	34.22	g	6.61	626.39
	MT Multi	15	621.19	127.45	c	197.69	14.89	AB	454.91	116.18	g	5.59	672.32
	MT	15	732.40	146.48	b	203.34	12.71	A	457.75	64.68	g	5.95	789.75
Katana	STML	15	732.92	87.13	b	191.64	10.97	B	448.67	61.07	g	10.11	769.59
Vita	YZ ST	15	883.40	101.23	a	194.98	7.06	AB	647.83	73.33	f	10.37	926.53

Groups with the same case letter per column are not significantly different (p > 0.05).
G = glazed; NG = not glazed; SF = speed fired; RF = regular fired

Table 1: Properties of the various ceramic materials

Discussion

The null hypotheses for this study was rejected because there were significant differences in the tested properties based on the type of ceramic material.

Limited research has been published evaluating the strength properties of Tessera, the novel lithium-silicate/lithium-aluminum silicate-based ceramic from Dentsply Sirona. In this study, the flexural strength values for the Tessera groups were 36.5% - 45.3% less than the IPS e.max CAD group and was statistically significant. Although not

statistically significant, the flexural fatigue strength values of the Tessera groups were 20.0 – 29.1% less than the IPS e.max CAD group. The Weibull modulus was used to evaluate the distribution of strengths and weaknesses of the materials.(17, 18) The Weibull modulus of the IPS e.max CAD group was 43.6 – 63.5% greater than the Tessera groups. This would suggest a more homogenous distribution of irregularities in the IPS e.max CAD ceramic, resulting in a more consistent material. Additionally, the characteristic strength of IPS e.max CAD (i.e., the strength at a failure probability of 63.2%), was 35.1 – 43.6% greater than the Tessera groups. The results of this study would not support the manufacturer's claim that Tessera is the "strongest glass-ceramic block currently on the market – up to 32% stronger".(19) A recent study by Lubauer et al. found that the fracture toughness of Tessera after firing was 28.9% less than the crystallized IPS e.max CAD.(20)

The instructions for use by the manufacturer for Tessera require the material to be glazed during a firing cycle. The manufacturer also reports that Tessera can be speed-fired in just 4.5 minutes in the CEREC SpeedFire furnace versus regular-fired with up to 12.5 minutes in a conventional furnace.(19) In this study, there was no statistically significant difference in flexural strength or flexural fatigue strength between the Tessera specimens whether they were glazed or unglazed or regular- or speed-fired. However, the glazed, regular-fired Tessera specimens had the greatest characteristic strength.

The 4Y-TZP zirconia materials (IPS e.max ZirCAD MT, Katana STML, VITA YZ ST) showed a statistically significant higher flexural strength than the gradient 3Y-TZP/5Y-TZP and 4Y-TZP/5Y-TZP. However, a limitation to this study is that only the middle transition zone of the gradient zirconia materials was evaluated in this study. The

gradient oxide ceramics have a transition in the amount of cubic content and therefore a transition in flexural strength. A recent study by Winter et al., evaluated the biaxial flexural strength of IPS e.max ZirCAD Prime and found a significant range of strength values from 848.6 ± 108.8 to 494.4 ± 109.6 MPa of the 3Y-TSP/5Y-TSP gradient zirconia. They concluded that the mechanical properties of the gradient zirconia was affected by the positioning within the blank and that nesting of the restoration in the cervical position could have a positive impact by combining the high mechanical strength in the dentin and body layers with improved optical properties in the occlusal and incisal areas.(21)

YITA YZ ST had the best overall performance to include the highest flexural strength, flexural fatigue strength, Weibull modulus, and characteristic strength of all of the tested oxide-based zirconia materials. With a higher Weibull modulus, it can be expected that the material ensures reliable use within its respective recommended indication range.(22) Interestingly, the Weibull modulus of IPS e.max CAD (14.05) was greater than YITA YZ ST (10.37) and all the other tested ceramic materials.

The material thickness of a restoration has a significant impact on translucent and mechanical properties of ceramics. In this study, the specimen thickness was limited to 1.3mm according to ISO standards and the specimens were not bonded to a substrate. Bonding specimens to a substrate, such as tooth structure, has been shown to increase the amount of stress required to fracture the material.(23, 24) Thinner/thicker specimen thickness would alter the performance of the all lithium-based and oxide-ceramic based ceramic materials.

Conclusion

Based on this study, oxide-based zirconia exhibited higher values for all properties evaluated when compared to silica-based ceramics. While the flexural fatigue strength of the new Tessera ceramic was comparable to IPS e.max CAD, it had a significantly lower ability to withstand higher static forces as indicated by its lower flexural strength value.

Disclaimer: The views expressed are those of the authors and do not reflect the official views or policy of the Uniformed Services University, Department of Defense, or its Components. The authors do not have any financial interest in the companies whose materials are discussed in this abstract.

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