

# Comparison between Biaxial Flexural Strength and Microstructure of Polished and Glaze-Fired Specimens of Zirconia Lithium Silicate Glass Ceramic

Alaa M. Attaallah<sup>1</sup>, Eman M. Zayed<sup>1</sup>, Salma M. Dabees<sup>1</sup>, Youssef Y. Ashour<sup>2</sup>,  
Amal Ezz-Eldin Fahmy<sup>3</sup>

<sup>1</sup>Dental Student, Faculty of Dentistry, Pharos University, Alexandria, Egypt

<sup>2</sup>Assistant Lecturer of Fixed Prosthodontics, Faculty of Dentistry, Pharos University, Alexandria, Egypt

<sup>3</sup>Professor of Dental Biomaterials, Faculty of Dentistry, Pharos University, Alexandria, Egypt

*Received 14 February 2019 and Accepted 13 June 2019*

## Abstract

**Introduction:** There has been a growing interest in glass ceramic systems with good esthetics, high fracture resistance, bonding durability, and simplified fabrication techniques using CAD/CAM. The aim of this study was to compare flexural strength in "polished" and "glazed and fired" specimens of zirconium lithium silicate (ZLS) CAD/CAM blocks Celtra Duo. **Methods:** A total of 14 specimens of Celtra Duo (Dentsply Sirona, Germany) were designated and equally divided into two groups, including group I (n=7; as polished) and group II (n=7; as glazed and fired) according to the manufacturers' instructions. Then biaxial flexural strength was tested according to ISO 6872 using a universal testing machine and piston-on-three-ball method after polishing, glazing, and firing. The fractured surfaces and microstructure were observed by scanning electron microscope (Jeol/GSM5300). Data were then statistically analyzed using SPSS software (version 20.0). **Results:** There were statistically significant differences in biaxial flexural strength between a polished and glaze-fired specimen of ZLS glass ceramic discs as biaxial flexural strength was lower in polished specimens (mean±standard deviation: 158.7±21.67 MPa), while the glazed specimens showed higher biaxial flexural strength (mean±standard deviation: 261.5±31.89 MPa). **Conclusion:** Glazing and firing of Celtra Duo specimens increased biaxial flexural strength significantly in comparison to polishing with no significant change in the microstructure.

**Keywords:** Celtra Duo, Piston-on-three-ball, CAD/CAM, ZLS.

-----  
Attaallah AM, Zayed EM, Dabees SM, Ashour YY, Fahmy AE. Comparison between Biaxial Flexural Strength and Microstructure of Polished and Glaze-Fired Specimens of Zirconia Lithium Silicate Glass Ceramic. J Dent Mater Tech 2019; 8(3): 114-20.

## Introduction

Despite the advantages of zirconia-based restorations, one of the major drawbacks of zirconia is its opaqueness. That is why a highly strong but not esthetic zirconia core should be veneered with less strong esthetic ceramic material. Lithium silicate glass-ceramic e.max is ideally suitable for the fabrication of monolithic single-tooth restorations. This innovative ceramic provides highly esthetic results; however, its strength is still under question (1).

The aesthetic appearance of restoration and a natural tooth is dependent on the optical properties of the hard tissues, restorative material, and interaction between them. Therefore, material translucency is an important factor for the clinical selection of restorative materials. From the esthetic aspect, it is crucial to select a material that closely matches the natural translucency and grey-scale of the tooth. Knowledge of the translucency of aesthetic restorative materials enables clinicians to better match the optical adjustment of the restoration to the individual clinical situation (2).

A new material zirconia-reinforced lithium silicate (ZLS) was recently introduced for the fabrication of monolithic anterior and posterior crowns. It is a new class of high-strength glass ceramic material (1). According to the manufacturer, it comprises fine lithium metasilicate and lithium disilicate crystals in a glassy matrix containing zirconium oxide in solution. It has been reported that its aesthetics is appealing because of high glass content. The increased glass content improves translucency and facilitates in-depth opalescence and fluorescence (1).

Also, they claimed that 10% zirconia is finely distributed in the glass phase, and the crystallization of zirconia particles is eliminated. As a result, the zirconia does not have an opaque effect; therefore, dissolved zirconia reinforces the glass matrix without clouding it and provides high biaxial flexural strength properties with low wearing of tooth structure.

Celtra Duo (Dentsply Sirona, Germany) can be finished by both surface polishing and glaze-firing. It is possible to etch ZLS ceramics then bond with resin cement. Glazed ZLS can be luted with conventional cementation with glass-ionomer cement or resin-

modified self-adhesive luting materials (3). The present study was an attempt to evaluate the biaxial flexural strength of polished and glaze-fired specimens of CAD/CAM ZLS and evaluate their microstructure under a scanning electron microscope (SEM).

## Materials and Methods

Two of fully crystallized block of CAD/CAM ZLS were ground to cylindrical form. Then by microtome machine under water irrigation they were sliced into 20 disc forms of the diameter with 12.0 mm and 1.2 mm thickness. Then they were examined by SEM in order to illustrate any defects and following that 6 defected specimens were excluded, and other 14 specimens were divided into two equal groups, including group I as polished (n=7) and group II as glazed and fired (n=7).

### Polishing

Group I specimens were polished by the Celtra Duo polishing set as follows: 1 sintered diamond grinder, wheel, black; 1 diamond grinder each cone flat, yellow, reversed cone, yellow, cone pointed, yellow, and ball, yellow; 1 diamond polisher each wheel, green, wheel, violet, and wheel, yellow, and 1 diamond polisher point, yellow (Table I). Table II described the materials brand and manufacturer used in this study.

### Glazing

Group II specimens were cleaned for 3 min with an ultrasonic cleaner in water bath to eliminate any remaining residue on the surface. Then they were glazed at 820°C following the manufacturer's instruction. Glazing the specimens was done after finishing smoothing the surface after slicing. The universal Celtra glazing paste was mixed with distilled water on a plastic slab, and then the mix was applied on the specimens with a brush to produce a uniform thickness. Glaze firing was conducted on a honey-combed firing tray, and then it was introduced into the Ivoclar Vivadent (Programat Furnace) using the stipulated firing parameters (Table III). The specimens were allowed to cool at room temperature after the completion of the firing cycle and removed from the furnace. Finally, the thicknesses of the specimens were checked using a digital caliper.

**Table I.** Composition of material

Material	Manufacturer	Composition	Young's modulus E (GPa)	Poisson's Ratio $\nu_a$
Celtra Duo	Dentsply Sirona	<ul style="list-style-type: none"> <li>• 10% zirconia</li> <li>• 58% silica, lithium metasilicate and phosphate crystals</li> <li>• SiO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, Al<sub>2</sub>O<sub>3</sub>, LiO, ZnO, 10% ZrO<sub>2</sub></li> </ul>	107.9	0.222

**Table II.** Materials brand and manufacturer used in this study

Product name	Manufacturer
CELTRA DUO	Dentsply Sirona, Germany
Universal Celtra Duo glaze	Dentsply Sirona, Germany
Celtra Duo polishing paste	Dentsply Sirona, Germany

**Table III.** Crystallization parameter of post-milling heat treatment

	First glaze firing
Pre-drying	2 min plus 2 min when using SuperPeg II
Drying (depending on type of furnace)	2 min
Preheating	2 min
Start temperature	500°C
Heating rate	60°C/min
Final temperature	820°C
Vacuuming	Off
Holding time	1.5 min
Cooling	3 min

## Results

The tests were used for normal distribution of quantitative variables and compare the results of the two studied groups.

Table IV showed  $P < 0.001$  and  $t = 7.058$ , which proved that there was a significant difference in biaxial flexural strength between group I (polished) and group II (glazed and fired).

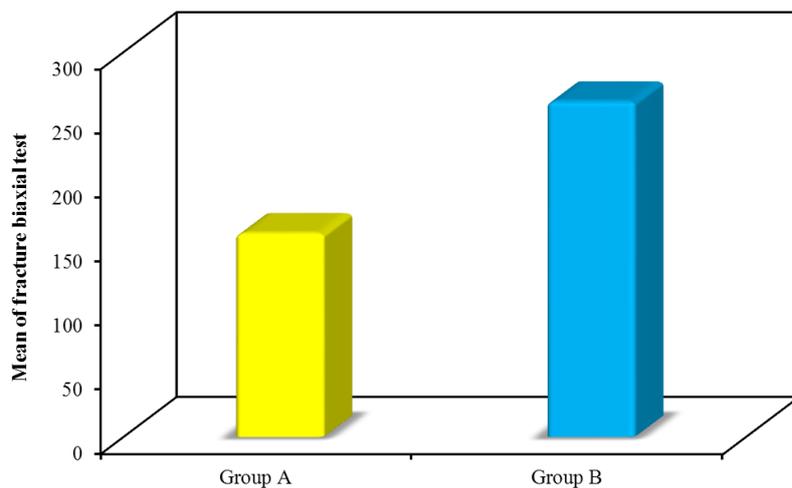
**Table IV.** Comparison between minimum, maximum, and mean  $\pm$  standard deviation in two studied groups according to fracture biaxial strength in MPa

Fracture biaxial test	Group I (n=7) (Polished)	Group II (n=7) (Glazed)	t	p
Minimum– Maximum	126.2–184.5	206.3–293.6		
Mean $\pm$ Standard deviation	158.7 $\pm$ 21.67	261.5 $\pm$ 31.89	7.058*	<0.001*
Median	159.1	263.5		

t: Student's t-test

p: P-value to compare two studied groups

\*: Statistically significant at  $P \leq 0.05$



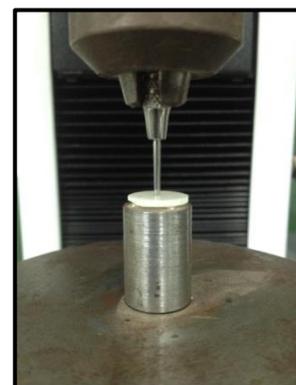
**Figure 1.** Comparison between two studied groups according to fracture biaxial test



(2A)



(2B)

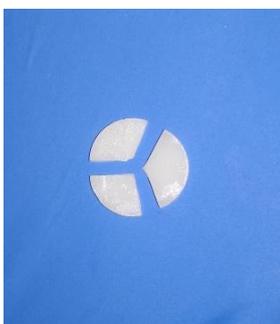


(C)

**Figure 2.** (2A,2C) Specimens under testing in universal testing machine . (2B) Piston-on-three-ball



(3A)



(3B)



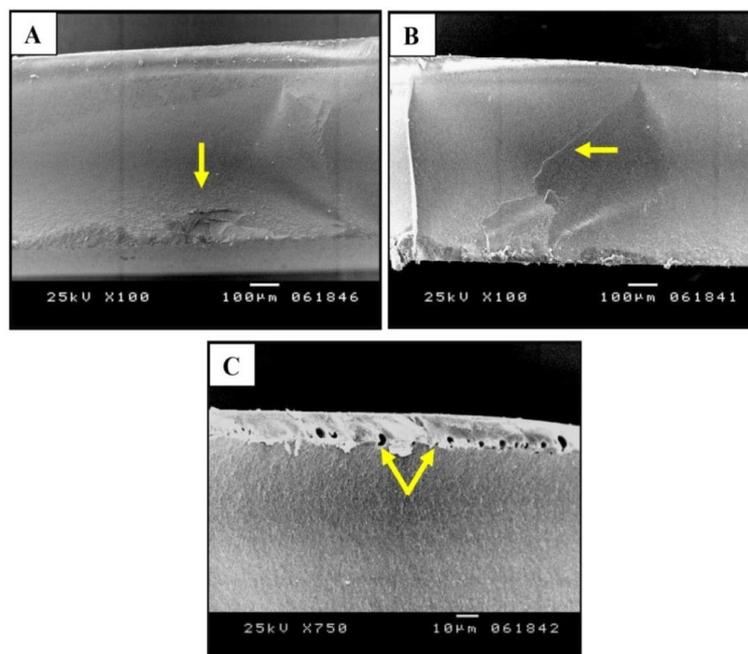
(3C)

**Figure 3.** Fracture pieces of two studied groups

**Table V.** Percentage of fractured pieces in two studied groups

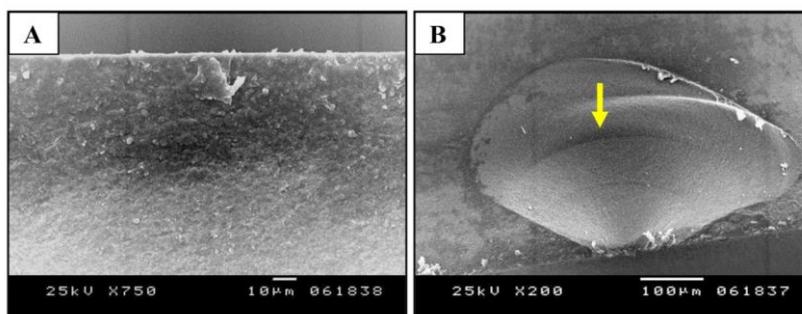
	Celtra Polished	%	Celtra Glazed	
Two pieces	4	55	5	70
Three pieces	1	15	2	30
Four pieces	2	30	0	0

**Glazed specimens**



**Figure 4.** A: Scanning electron microscope (SEM) of fractured surface of zirconia-reinforced lithium silicate (ZLS) glazed specimens, crack origins mainly semi-elliptical flaws on surface at  $\times 100$ ; B: SEM of fractured surface of ZLS glazed specimens; arrows indicating crack direction propagation at  $\times 100$ ; C: SEM of glazed Celtra Duo samples with some micropores (arrows) present at interface between glaze layer and upper surface of disc at  $\times 750$

**Polished specimens**



**Figure 5.** A: Scanning electron microscope of fully-crystallized polished Celtra Duo (zirconia-reinforced lithium silicate) material showing fine grain structure of material at  $\times 750$  magnification; B: another photomicrograph of zirconia-reinforced lithium silicate Celtra Duo polished specimens with visible hackle lines (arrow) at site of crack origin at  $\times 100$

## Discussion

Several types of ceramic materials, such as zirconia-based core ceramic, have been utilized for chair-side fabrication of all-ceramic restorations using CAD/CAM (6). The ZLS glass ceramic maintains a relatively high strength, which is high enough for full-coverage crowns in the posterior area. The ceramic blocks are fully crystallized and contain both lithium silicate and zirconia. Celtra Duo is a new class of ceramic, which is called ZLS. The inclusion of 10% zirconia dissolved into the lithium silicate glass matrix results in 4 times smaller silicate crystals implying a high glass content and higher translucency than conventional LiSi ceramics (Celtra Duo, Dentsply Sirona, Germany).

Surface defects and cracks are among threats that exist in dental ceramics. These surface cracks depend on the manufacturing technique. Using CAD/CAM technology decreases the risk of inducing cracks while shaping the ceramic restoration. One important aspect in the critical cracks of the ceramics is the fact that they may exhibit a slow and stable crack growth (SCG) or may be called static fatigue when subjected below the critical value.

The critical value is the stress at which the ceramic material fractures. The SCG occurs especially in the presence of water as present in the oral environment. This phenomenon will lead to the strength of degradation over time and decrease of lifetime of dental ceramics (8, 9). In this study, the glazed specimens of Celtra Duo showed more cracks than the polished ones. Stating the strength of ceramic materials definitively seems infeasible due to multiple factors influencing measurements, such as the testing method, specimen dimensions, test environment, polishing procedures, stress rates, and stress area (10).

The 3-point biaxial flexural strength test utilizes a bar sample with two supports, while the biaxial test requires more force to break the sample, compared to the 3-point biaxial flexural test that results in higher and reliable results. Test data using the piston-on-three-ball method suggest that the biaxial strength is generally higher than the uniaxial strength. This relationship was assessed by the general relationship stated by Schatz et al. (11).

The piston-on-three-ball test has shown to be a reliable and simple method for determining the biaxial strength of restorative materials supplied as small CAD/CAM blocks. Moreover, it applies the force into the specimens directly on the center and does not affect their peripheries, which make it more reliable.

The Size of the disc diameter in this study was 12 mm with thickness of 1.2. The present study used the biaxial flexural test with piston-on-three-ball method as

a reliable technique for studying brittle materials standardized by ISO.

In the present study, we showed that glazed ZLS had higher mean value than the polished ZLS using piston-on-three-ball method that is in line with the findings of a study by Riquieri et al. (12). In the aforementioned study, it was shown that ZLS was higher than the e.max CAD. However, by SEM the broken specimens and piston-on-three-balls test an important change was found in the microstructure of the fully crystallized lithium silicates that is mainly the growth of lithium silicate grains. The results of the aforementioned study are consistent with the findings of the present study indicating a higher mean value for the fully crystallized ZLS.

Regarding aging, the glazed specimens showed higher mean value of biaxial flexural strength than polished specimens in this study. However, Beuer et al. (13) and Kang et al. (14), who studied the strength of CAD/CAM fabricated all-ceramics with similar aging procedure, reported that no difference was found in the strength mean values with or without aging. They also showed that For lithium disilicate and ZLS, the initial biaxial flexural strength increased greatly after heat treatment. With regard to the fractured pieces of the specimens, it was shown that the Celtra Duo glazed specimens had higher strength than the Celtra Duo polished specimens.

## Conclusion

Based on the results of this in vitro study, the glazed ZLS Celtra Duo had higher biaxial flexural strength than the Celtra Duo polished specimens.

## Conflicts of interest

The authors declare that there is no conflict of interest.

## Acknowledgments

The authors would like to thank Dean of faculty of dentistry, pharos university, Alexandria, Egypt, Prof.Dr. Ahmed Yehia Ashour. And Vice dean of faculty of dentistry, pharos university, Alexandria, Egypt, Prof.Dr. Fayza Eldaly for their continuous support and encouragement.

## References

1. Preis V, Behr M, Hahnel S, Rosentritt M. Influence of cementation on in vitro performance, marginal adaptation and fracture resistance of CAD/CAM-fabricated ZLS molar crowns. *Dent Mater.* 2015;31(11): 1363-1369.

2. Kelly JR, Benetti P. Ceramic materials in dentistry: historical evolution and current practice. *Aust Dent J.* 2011;56(Suppl 1): 84-96.
3. Bader JD, Rozier RG, McFall WT Jr, Ramsey DL. Effect of crown margins on periodontal conditions in regularly attending patients. *J Prosthet Dent.* 1991;65(1): 75-79.
4. Jin J, Takahashi H, Iwasaki N. Effect of test method on flexural strength of recent dental ceramics. *Dent Mater J.* 2004;23(4): 490-496.
5. ISO-Standards ISO 6872 Dentistry-Ceramic materials. 3rd ed. Geneva: International Organization for Standardization; 2008.
6. Miyazaki T, Hotta Y. CAD/CAM systems available for the fabrication of crown and bridge restorations. *Aust Dent J.* 2011;56(Suppl 1): 97–106.
7. Awad D, Stawarczyk B, Liebermann A, Ilie N. Translucency of esthetic dental restorative CAD/CAM materials and composite resins with respect to thickness and surface roughness. *J Prosthet Dent.* 2015;113(6): 534-540.
8. Southan DE, Jorgensen KD. The endurance limit of dental porcelain. *Aust Dent J.* 1974;19(1): 7-11.
9. Bergmann CP, Stumpf A. *Dental Ceramics: Microstructure, Properties and Degradation.* New York: Springer; 2013.
10. Albakry M, Guazzato M, Swain MV. Biaxial flexural strength, elastic moduli, and x-ray diffraction characterization of three pressable all-ceramic materials. *J Prosthet Dent.* 2003;89(4): 374–380.
11. Schatz C, Strickstock M, Roos M, Edelhoff D, Eichberger M, Zylla I, et al. Influence of specimen preparation and test methods on the flexural strength results of monolithic zirconia materials. *Materials.* 2016;9(3): 180.
12. Riquieri H, Monteiro JB, Viegas DC, Campos TMB, de Melo RM, de Siqueira Ferreira Anzaloni Saavedra G. Impact of crystallization firing process on the microstructure and flexural strength of zirconia-reinforced lithium silicate glass-ceramics. *Dent Mater.* 2018;34(10): 1483-1491.
13. Beuer F, Steff B, Naumann M, Sorensen JA. Load-bearing capacity of all-ceramic three-unit fixed partial dentures with different computer-aided design (CAD)/computer-aided manufacturing (CAM) fabricated framework materials. *Eur J Oral Sci.* 2008;116(4): 381-386.
14. Kang SH, Chang J, Son HH. Flexural strength and microstructure of two lithium disilicate glass ceramics for CAD/CAM restoration in the dental clinic. *Restor Dent Endod.* 2013;38(3): 134-140.

**Corresponding Author**

Alaa M. Attaallah  
 Fifth year student, Faculty of Dentistry, Pharos University  
 Mobile: 01285347991  
 E-mail: alaaabdefata7@gmail.com