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THE EFFECTS OF RE-PRESSING ON BIAXIAL FLEXURAL STRENGTH AND MICROSTRUCTURE OF CELTRA PRESS (AN INVITRO STUDY)

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ABSTRACT

INTRODUCTION: Celtra® Press is a zirconia-reinforced lithium silicate (ZLS) glass-ceramic containing 10 % zirconium oxide (ZrO2). This material is used to fabricate dental restorations by pressing technique. After heat pressing procedures leftover buttons are usually discarded. these leftover buttons are suggested to be re-pressed with concerns regarding changes in microstructure and mechanical properties.

OBJECTIVES: To evaluate the effects of re-pressing on the mechanical properties and microstructure of Celtra® Press.

METHODOLOGY: A total of 24-disc specimens; 14mm in diameter and 1.5 in thickness were fabricated by the lost wax technique. Specimens were divided into two parallel groups, Group I: Pressed specimens (n=12) and Group II: Re-pressed specimens (n=12). The biaxial flexural strength (BFS) was measured using a piston-on-three-ball test. X-ray diffraction (XRD) was used to identify the crystalline phases. Scanning electron microscope (SEM) photomicrographs (5000x) were used for microstructure analysis.

RESULTS: The mean and standard deviation values of BFS for the pressed specimens (Group I) and the re-pressed specimens (Group II) were (136.72 ± 29.41) and (167.24 ± 36.46) MPa respectively. BFS of the repressed group was significantly higher than the pressed group (p=0.001). SEM photomicrographs (5000x) showed an increase in the grains size after repressing. X-ray diffraction revealed lithium silicate as the main crystalline phase and the peak intensities of the re-pressed specimens were lower than the pressed specimens.

CONCLUSION: Re-pressing of Celtra® Press improved the BFS values significantly. Lithium silicate was the main crystalline phase in the pressed and re-pressed specimens. Celtra® Press grains were larger after re-pressing under SEM.

KEYWORDS: Glass-ceramics, Celtra® Press, Repeated heat-pressing, Mechanical properties, Microstructure

RUNNING TITLE: Effects of re-pressing on biaxial on Celtra Press.

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INTRODUCTION

All-ceramic dental restorations have gained much interest due to their strength, esthetics, and ease of fabrication (1). Ceramics can be shaped to full-contour restorations thus eliminating the challenges of combining different materials synergistically (2).

Moreover, glass ceramics can still be shaped to a core and then layered for maximum esthetics with some strength compromise (3,4).

Ceramic restorations are fabricated by sintering, slip casting, heat pressing, and milling (5). Moreover, heat pressing has advantages over sintering and slip casting in terms of porosity and marginal fit (6). Pressable ceramics are categorized into two generations, the first-generation is leucite-based while the second generation is lithium disilicate based (7,8). Lithium disilicates have received importance as the flexural strength and the fracture toughness are higher than other crystalline forms of pressable ceramics. However, lithium disilicates are still brittle and do not have enough strength to be used in high-stress areas (9).

The heat pressing procedure utilizes the lost wax technique and involves pressing ceramic ingots into a mold cavity inside a pneumatic press furnace by a plunger. The sprues and buttons are removed after pressing and cooling. The buttons used in the pressing cycles should be discarded. A new glass-ceramic ingot is used for each new pressing cycle. Re-pressing of leucite-based and lithium disilicate-based glass-ceramics has been reported in the literature with concerns regarding changes in microstructure and mechanical properties (10-16).

Efforts to increase the durability of glass-ceramics were directed towards enhancing their mechanical properties and microstructure while maintaining their optical properties for maximum esthetics. During fabrication procedures of glass ceramics, the glassy phase is transformed into the crystalline phase, and the resulting materials are composed of a glassy matrix with several crystalline phases (7).

The final crystalline form depends on the glass composition, nucleating agent, and method of heating (17,18). Also, the morphology and size of the crystals play a significant role in the determination of mechanical properties. Strengthening methods of glass-ceramics include ion exchange to form surface

compressive stresses and the addition of ZrO_2 with different concentrations to form zirconia-toughened glass-ceramics (19-22).

Celtra® Press is a newly introduced zirconia-reinforced lithium silicate (ZLS) with the addition of 10% wt. zirconium oxide (ZrO_2) as a nucleating agent. During heat pressing, ZrO2 promotes volume crystallization of glasses and hinders crystal growth (23). Consequently, smaller crystalline phases are present which improve optical properties and decrease surface roughness (24,25). However, the incorporation of ZrO_2 and crystal growth hindering affected the flexural strength (26).

According to the manufacturer, Celtra® Press has mechanical properties values that are comparable with lithium silicate glass-ceramics (17,27,28). However, there is a paucity of information in the literature about the effect of repressing on the microstructure and the mechanical properties of the recently introduced ZLS.

The null hypothesis was that re-pressing had no effect on the BFS and microstructure of Celtra® Press.

MATERIALS AND METHODS

1. Specimen preparation

Lost wax technique was used to manufacture a total of 24 Celtra® Press (Dentsply Sirona, NC, USA) disc-shaped ceramic specimens. A digital design of the disc-shaped specimen; fourteen millimeters in diameter and one and half-millimeter in thickness was designed by computer-aided design software (AutoCAD, Autodesk Inc., USA). The specimen design was dry milled in white CAD/CAM wax blanks (Ceramill® wax- Amann Girrbach AG, Austria). Twenty-four wax specimens were produced using a 5-axis milling machine (Ceramill Motion 2 - Amann Girrbach AG, Austria). Wax Specimens were then divided into 2 equal parallel groups. Group I: Pressed specimens (n=12) and Group II: Re-pressed specimens (n=12).

Each 3 wax specimens were sprued and invested in a 100gm ring system using phosphate-bonded investment (Bellavest® SH – BEGO, USA). **(Figure 1-A)** The ring was then placed in a burnout furnace (Yoshida burnout furnace. Sumida-Ku Tokyo, Japan) at room temperature and heated to 900℃. The temperature was maintained at 900℃ in the burnout for 30 minutes.

New Celtra® Press ingot was used for each pressing cycle. Pressing cycle parameters were adjusted following the manufacturer's instructions for pressing. Pressing was achieved under vacuum using a pneumatic furnace (Programat® EP 3010 - Ivoclar Vivadent, Schaan, Liechtenstein). **(Figure 1-B)** For re-pressed specimens (Group II), leftover buttons from the pressing cycles were adjusted and used as ingots for the repressing cycles following the same steps of the pressing procedure. **(Figure 1-C)** (**Table 1)**

Using silicon carbide sandpaper with grits from 100 to 2500, the ceramic specimens were wet polished. Finally, all the specimens were cleaned with distilled water at 55℃ for 10 mins utilizing an ultrasonic cleaner. Digital caliper (Hogetex digital caliber, Netherlands) with an accuracy of ± 10 µm was used to measure the final specimen dimensions at three different points. **(Figure 2)**

2. Testing of mechanical properties

2.1. Biaxial Flexural Strength (BFS)

The Piston-on-three-ball test was used to evaluate the BFS of ceramic specimens following the ISO 6872 specifications. The piston was flat-ended with a diameter of 1.5 mm. The three supporting balls were 3.2 mm in diameter and positioned apart at an angle of 120 degrees on a support circle with a diameter of 12.5 millimeters. The specimens were positioned on the three supporting balls concentrically and loaded by the flat-ended piston with a crosshead speed of 1mm/min using a universal testing machine (Instron 3345, Electromechanical, Norwood, MA, USA). To reduce the friction a polyethylene sheet was placed between the specimen and the piston (29). **(Figure 3)**

The BFS was calculated as follows:

$S = -0.2387\ P\ (X - Y)/d2$

S is the BFS (MPa), P is the total load required for fracture (N),

$X = (1 + n)\ ln\ (B / C)2 + [\ (1 - n) / 2]\ (B / C)2$

$Y = (1 + n)\ [\ l + ln\ (A / C)\ 2\ ] + (1 - n)\ (A / C)2$

n = Poisson's ratio

A = radius of supporting circle (mm).

B = radius of loaded area (mm).

C = radius of specimens (mm).

d = specimen thickness at fracture origin (mm).

ln = natural logarithm.

The ISO 6872 specifications recommend a Poisson's ratio for dental ceramics to be 0.25. Poisson's ratio measures the Poisson effect. This effect measures the expansion of the specimens in a perpendicular direction to the compression force directions (28).

3. Observation of microstructure

3.1. X-ray diffraction (XRD)

For each group, the ceramic specimens were submitted to XRD for crystalline phase determination. specimens were placed on the diffractometer system holder (EMPYREAN, Malvern Panalytical, UK). Cu Kα x-rays were used for scanning with 2θ angles from 4.0131 to 79.9591 degrees, the step size was 0.0260 degrees, and 21.42 seconds scan step time. 45 kV and 30mA, respectively, were chosen as voltage and current. HighScore Plus software (Malvern Panalytical, UK) was used for qualitative and quantitative crystallographic analysis. Crystalline phases were determined by comparing the peaks with the ICDD (International Center of Diffraction Data) files.

3.2 Scanning electron microscope (SEM)

The microstructure of pressed and repressed specimens was evaluated using SEM. Specimens were etched with hydrofluoric acid with a concentration of 9.5%. Specimens were etched for 60 seconds then rinsed for 60 seconds thoroughly with air-water sprays. Specimens were cleaned with distilled water using an ultrasonic cleaning device. After ultrasonic cleaning, specimens were gold-sputtered using an ion sputtering device (JFC-1100E FINE COAT ion sputtering device; JEOL, Tokyo, Japan) and the microstructure was observed under 5000x magnification using SEM (JSM-

IT200 InTouchScope™ Scanning Electron Microscope; JEOL, Tokyo, Japan).

4. Statistical analysis.

The collected data were analyzed statistically by the Statistical Package for Social Science program (SPSS 21.0; SPSS, Inc., IL, Chicago, USA). The parametric statistics were used as a Kolmogorov-Smirnov test of normality revealed no significance in the distribution of the variables. Data were described using minimum, maximum, mean, standard deviation, and 95% CI of the mean. Paired t-test was used to compare between the two studied dependent normally distributed variables. An alpha level was set to 5% with a significance level of 95%, and a beta error accepted up to 20% with a power of study of 80%.

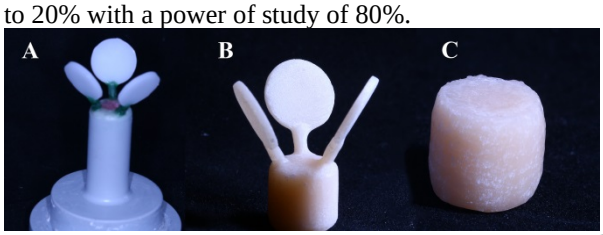

Figure 1: (A): CAD-CAM milled wax specimens after spruing. (B): 3 Celtra® Press pressed ceramic specimens. (C): Pressed Celtra® Press ingot used in repressing after adjustments.

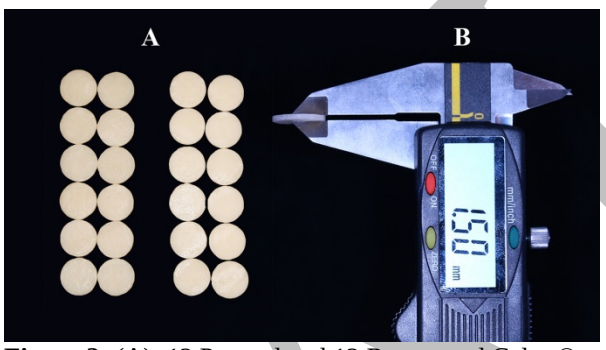

Figure 2: (A): 12 Pressed and 12 Re-pressed Celtra® Press ceramic specimens. (B): Thickness of polished pressed ceramic specimen.

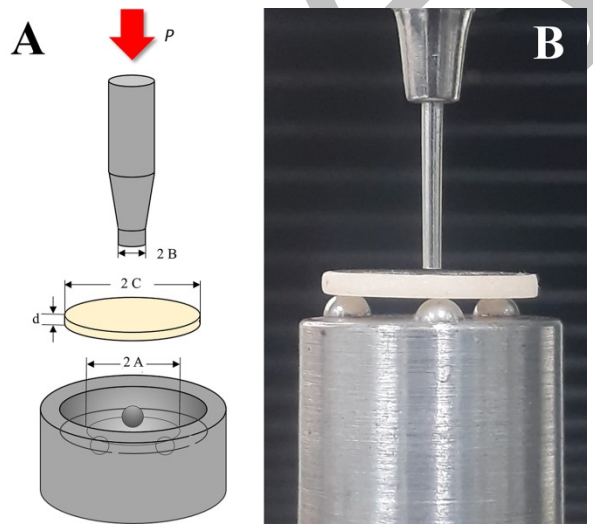

Figure 3: BFS testing method by piston-on-three-ball test following ISO 6872.

Table 1: Celtra® Press composition and pressing parameters.

Material	Composition	Lot.	Translucency	Shade	Processing technique
Celtra® Press	SiO_2: 59.3%, Al_2O_3: 3%, Li_2O: 14.5%, K_2O: 1.2%, Na_2O: 0.2%, P_2O_5: 4.9%, B_2O_3: 2%, MgO: 0.01%, ZrO_2: 9.3%, SrO: 0.0003%, CeO_2: 0.83%, V_2O_5: 0.61%, Tb_2O_3: 3.3%, Er_2O_3: 0.73%, HfO_2: 0.21%	18025848	MT	A3	Heat-pressing
Celtra® Press pressing parameters	Stand-by temperature	Temperature increase/ min	Pressing temperature		Holding time
	700℃	40℃/min	860℃		30 mins

RESULTS

1. Mechanical properties

The mean and standard deviation values of BFS were 136.72 ± 29.41 MPa for the pressed group and 167.24 ± 36.46 MPa for the repressed group. The BFS values of the repressed group (Group II) were significantly higher than the pressed group (Group I) were (P=0.001). **(Table 2) (Figure 4)**

*2. **Microstructural evaluation***

2.1. XRD analysis

The XRD data revealed the lithium silicate crystals as the main phase in both pressed and repressed specimens. The peak intensities of re-pressed specimens were lower than the peak intensities of the pressed specimens. **Table (3). Figure (5)**

2.2. SEM analysis

The SEM photomicrograph (5000x) showed needle-shaped particles presented in both pressed and re-pressed ceramic specimens. There was a noted increase in the average size of the particles after re-pressing. The mean particle dimensions in the pressed specimens were 1.68 microns in length and 0.301 microns in width while the mean particle dimensions in re-pressed

specimens were 2.32 microns in length and 0.468 microns in width. **(Figure 6)**.

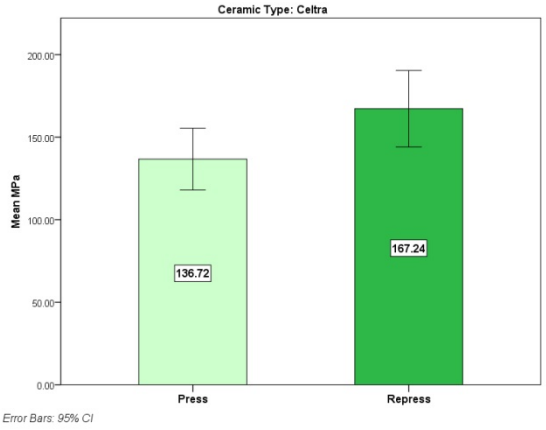

Figure 4: BFS of pressed and re-pressed specimens.

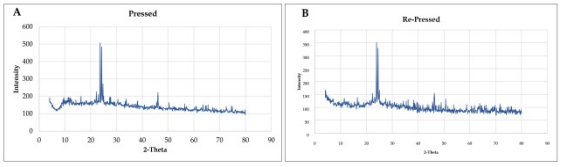

Figure 5: (A) XRD peaks for pressed Celtra® Press ceramic specimens. (B): XRD peaks for re-pressed Celtra® Press ceramic specimens.

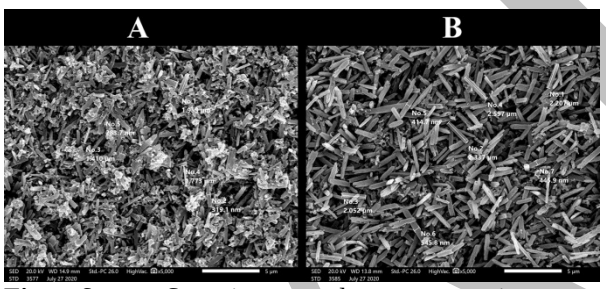

Figure6: Scanning electron microscope photomicrograph (5000X) after etching. (A): Pressed Celtra® Press specimen. (B): Re-pressed Celtra® Press specimen.

Table 2: BFS of pressed and re-pressed Celtra® Press ceramic specimens.

Data	Celtra Press	Celtra Re-press	Test of significance
n	12	12	
Min-Max	103.50-211.79	128.49-242.99	t=4.463
Mean ± Std. Deviation	136.72 ± 29.41	167.24 ± 36.46	*p*=0.001*
95% CI for mean	118.03 – 155.40	144.07 – 190.40	

n: Number of specimens
Min-Max: Minimum – Maximum
CI: Confidence interval
*: Statistically significant (p<0.05

Table 3: XRD pattern list for pressed and re-pressed ceramic specimens.

Specimens	Compound Name	Chemical formula	Score	hkl	Ref. Code
Celtra Press	Lithium Silicate	$Li_2Si_2O_5$	42	040	00-040-0376
	Lithium Silicate	$Li_2Si_2O_5$	2	111	00-024-0651
	Lithium Phosphate	Li_3PO_4	4	040	00-045-0747
Celtra Re-press	Lithium Silicate	$Li_2Si_2O_5$	20	040	00-015-0637
	Lithium Silicate	$Li_2Si_2O_5$	11	111	01-070-4856
	Lithium Phosphate	Li_3PO_4	3	040	00-045-0747

DISCUSSION

The purpose of this study was to evaluate the effects of re-pressing on the mechanical properties and microstructure of Celtra® Press. Repressing of Celtra® Press significantly increased the BFS. Moreover, the microstructure of Celtra® Press specimens was changed in the x-ray diffraction and scanning electron microscope analysis. Therefore, the null hypothesis of this study was rejected.

BFS evaluates the strength of glass-ceramics which are brittle materials. These ceramics are much stronger in compression than in tension (30). In flexural tests, pure tensile stress is applied on one side of the specimen. Moreover, this tensile stress is balanced on the opposite side by compression stress. Failure of the specimen occurs on the side with pure tensile stresses.

The most common flexural test methods used are uniaxial and biaxial bending tests. (31). Uniaxial bending tests have a significant disadvantage as there are difficulties in preventing failures of the edges. The biaxial strength testing has some advantages, over the uniaxial testing. These advantages include simple preparation of the specimens and the load is applied centrally on the specimens. Moreover, it is not necessary to round the edges of the specimens. Following the ISO standard 6872, disc-shaped specimens were prepared, and a piston-on-three-ball test was used to evaluate the BFS of the pressed and re-pressed ceramic specimens (29,32).

The BFS values of Celtra® Press were less than the BFS of conventional lithium disilicate glass-ceramics. According to Apel et al, the incorporation of the glassy matrix with ZrO2 did not increase the BFS values (25). Moreover, high ZrO2 content increased the viscosity of the glass-ceramic and reduced the growth of lithium disilicate crystals (33).

The BFS values were lower than the values reported by the manufacturer (above 400 MPA). The

manufacturer values were after glaze application and additional firing cycle which was not applied to the specimens. The lower BFS values can be related to residual stresses due to the manufacturing process and the usage of carbide sandpaper grits from 100 to 2500 (13,14). These results were in agreement with Gorman et al findings (14).

Residual stresses are normally present due to the manufacturing process and can be released by annealing. Annealing involves exposing the specimens to an additional firing cycle that can significantly increase the BFS values (34,35). Specimens of this study were tested without annealing so the residual stresses were not released, therefore these stresses might have been the reason for the lower BFS values.

The surface finish is also responsible for the BFS values of the specimens (4,36). Gorman et al (14) reported lower BFS values of lithium disilicate specimens related to the surface finish compared to manufacturer values (7,13). The lower BFS values were related to a surface finish of the specimens using silicon carbide sandpaper 180 grits (14). In this study, specimens were wet polished using silicon carbide sandpaper grits from 100 to 2500. The usage of silicon carbide sandpaper grit 100 might have induced more stresses and microcracks that adversely affected the BFS values.

SEM photomicrographs (5000x) showed a noted increase in the mean grain size of Celtra® Press after re-pressing. Larger grains composed of lithium silicate crystals were observed after the re-pressing. This increase in size is considered as Ostwald ripening phenomenon as described by Albakry et al.. Ostwald ripening is a phenomenon that is observed in solid solutions. It describes the changes in inhomogeneous structures where small crystals dissolve and redeposit onto larger crystals to form larger grains. This occurs because larger crystals more energetically stable than small crystals and the crystals systems tend to lower their overall energy. As a result, the small high-energy crystals dissolve and redeposit onto the larger more stable crystals. (7,13). Consequently, larger grains are expected to grow at the expense of small particles. This is due to the phase transition between lithium metasilicate and lithium silicate (21).

XRD showed that both pressed and repressed samples were composed of crystalline and amorphous phases. The main crystalline phase was lithium silicate in both pressed and repressed samples. However, the peak intensities after repressing were lower than the peak intensities of pressed samples.

Microstructural analysis showed that the crystallization process continued in the re-pressing procedure. This crystallization process deposited more crystals in the glassy matrix. Therefore, the grains size increased noticeably under SEM photomicrographs.

The composition of parent glass, nucleating agent, and heat treatment affects the final crystalline phases. volume nucleation by phase separation of the base glass leads to the development of glass ceramics with high mechanical properties. This controlled procedure prevents microcracks and flaws generated during fabrication that led to clinical failures (37).

Glass-ceramics are fabricated first by melting glass followed by a controlled heat treatment procedure by nucleating agents until the desired degree of crystallinity is produced. During these procedures, the glassy phase is transformed into the crystalline phase, and the resulting materials are composed of a glassy matrix into which several crystalline phases are embedded (38).

The main factor for controlling crystallization in glass-ceramics is nucleation. Volume nucleation and surface nucleation are two nucleation mechanisms utilized to form glass-ceramics. The predominant mechanism depends on the chemical composition of the nucleating agents and the parent glass (39).

The addition of ZrO_2 as a nucleating agent promoted volume crystallization of glasses and hindered crystal growth (23). Therefore, smaller lithium silicate crystalline phases were present in the pressed samples compared to ZrO2-free glass-ceramics. These smaller crystals adversely affected the mechanical properties of the glass-ceramic (25). However, after re-pressing there was a noted increase in the size of the grains under SEM. The growth of the grain size indicates that the crystallization process continues during the re-pressing procedure and more lithium silicate crystals are precipitated. The increase in the size of lithium silicate crystals leads to a significant increase in the BFS values of re-pressed ceramic specimens.

Studies have shown that re-pressing significantly influenced the microstructure of lithium disilicate-reinforced glass-ceramic materials and produced the blunt rod-like shape, larger grains, and orientation of the crystals (7,12,13). These findings are in accordance with this study. However, during repressing procedures, there is a possibility of increased porosity and cracks as well due to several nucleation sites through the crystallization process. These porosities and cracks represent flaws in the final restoration and might adversely affect the durability of such restorations (12).

Re-pressing of leucite glass-ceramics and lithium disilicate glass-ceramics was reported in the literature. However, there were controversies about the influence of re-pressing on the mechanical and physical properties of these glass-ceramics. Additionally, there was a paucity of information in the literature on the effects of re-pressing on the mechanical and physical properties of ZrO_2 lithium silicate glass-ceramics.

The findings of this study were in accordance with Albakry et al (13,40) and Chung et al (12). Albakry et al (13,40) evaluated the influence of repressing on IPS Empress and IPS Empress 2. They concluded that repressing resulted in significant growth of the lithium disilicate crystals in IPS Empress 2, but there was no change for the leucite crystals in IPS Empress. Additionally, the mechanical properties of IPS Empress 2 were not significantly affected by the size transformation of lithium disilicate crystals. Chung et al (12) also evaluated the influence of repressing on

IPS Empress and IPS Empress 2. They observed a significant increase in BFS and crystals size of IPS Empress 2 after re-pressing.

Repressing of IPS e.max Press was evaluated in two studies. In the first study, Gorman et al (14) investigated the effect of IPS e.max re-pressing up to four times. They concluded that the first pressing provided the optimum properties. Additionally, the mechanical properties did not differ significantly after subsequent pressings (14). In the second study, Tang X. et al (15) studied the effects of re-pressing on mechanical properties and microstructure of IPS e.max Press. They concluded that, after re-pressing, the microstructure was altered, and there was a noted increase in the porosity. Additionally, the density, hardness, flexural strength, and fracture toughness significantly decreased (15). These findings were not in agreement with our findings as the effects of repressing on the BFS and microstructure of Celtra® Press were significant.

Finally, re-pressing significantly improved the BFS and microstructure of Celtra® Press. However, additional studies on the effects of repressing on physical properties, fracture toughness, porosity, and Vickers hardness are important to provide a thorough analysis of the influence of re-pressing on the ZLS.

CONCLUSIONS

Within the limitations of the present study, the outcomes can be summarized as follows:

Repressed Celtra® Press specimens had a significantly higher BFS than pressed specimens. Moreover, lithium silicate was the main crystalline phase in both pressed and re-pressed specimens. Additionally, the crystal grains of repressed Celtra® Press specimens were larger than that of pressed specimens. Finally, further studies of other mechanical and physical properties are required.

CONFLICT OF INTEREST

The authors declare **that they have no conflict of interest.**

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