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THE EFFECT OF REPEATED HEAT-PRESSING ON THE BIAXIAL FLEXURAL STRENGTH AND SURFACE ROUGHNESS OF LITHIUM DISLILICATE GLASS-CERAMICS

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ABSTRACT

Statement of problem: During heat-pressing of lithium dislilicate glass-ceramics, It is more economical to press several restorations from one ingot at the same time. However, this is often not possible and may result in a considerable amount of leftover material. It has been reported that the leftover materials after heat-pressing are reused in some dental laboratories. Sufficient knowledge about the consequences of such procedure is not available. The issue is thereby raised whether the leftover material should be discarded or reused.

Purpose: The aim of the present study was to evaluate the effect of repeated heat-pressing on biaxial flexural strength and surface roughness of lithium dislilicate pressable glass-ceramics (IPS e.max Press). As well as to describe the microstructural features present in fresh-pressed, and repressed material using Xray diffraction, EDAX and SEM

Materials and methods: Twenty pressed and repressed samples were prepared following the manufacturers' recommendations measuring 15 mm × 1 mm per material. Biaxial flexure (piston on 3-ball method) was used to assess strength. X-ray diffraction was performed to identify the crystalline phases, and a scanning electron microscope was used to disclose microstructural features. Also surface roughness was evaluated

Results: BFS for pressed and repressed respectively; (375.8± 4.55) and (389.4±12.12) MPa; no significant difference was found between the Pressed and Repressed groups. Surface roughness for pressed glazed and unglazed respectively (0.27±0.05, and 1.14±0.16) – for repressed glazed and unglazed respectively (0.21±0.06 and 1.33±0.20) - for both groups, significant difference was found between Glazed and Un-Glazed ceramics. No statistical significant difference was found between Pressed and Repressed groups. Xray diffraction revealed the material is predominantly crystalline structure; lithium disilicate was identified to be the main crystalline phase, peaks after pressing and repeating pressing are similar, the crystalline phase assemblage did not change; however their radiation intensities (height) has, the dominant peak (highest peak) for the repressed

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sample is smaller compared to the pressed. EDAX results showed no change in composition between pressed and repressed samples. Microstuructural features SEM displayed a noted increase in crystal dimension after repressing

Conclusions. IPS e.max Press left over pressed buttons can be safely reused since repressing does not display consequent negative effects on Biaxial flexural strength and surface roughness, as well as microstructure.

KEYWORDS: Pressable ceramics, Lithium disilicate, Heat pressing, Glass ceramics

INTRODUCTION

The appearance of natural teeth is best mimicked by ceramic materials.[1] In recent years the popularity of all-ceramic dental restorations has increased due to their high esthetic qualities and metal-free structure. Significant developments in all-ceramic materials have created wonderful opportunities for the fabrication of lifelike restorations that provide reliable, long-term results [2] However, all-ceramic dental materials, are inherently fragile in tension, affected by microcracking, flaws, and defects that may be introduced during thermal treatment or fabrication procedures. The fabrication process precision, and skills of individual dental technicians, may affect the reliability and clinical performance of all-ceramic restorations. Mechanical properties such as strength is the first parameter assessed to understand the clinical potential and limits of dental ceramics. [3]

Heat-pressing has become a common technique to produce glass-ceramic dental restorations. In addition to its simplicity, this technique promotes better crystalline dispersion within a glass matrix, less porosity, and better marginal adaptation compared to sintering technique. IPS Empress was the original heat-pressed glass ceramic and leucite (SiO_2, Al_2O_3, $4K_2O$) is the main crystalline phase in this system. IPS Empress 2 has lithium disilicate ($Li_2O_2SiO_2$) as its main crystalline phase and is 60% crystalline when processed [2]

IPS e.max Press material has now replaced IPS Empress 2; it has improved mechanical properties and has significantly higher translucency. The microstructure consists of 70% lithium disilicate crystals embedded in a glassy matrix. These crystals are circular in morphology and measure 3 to 6 mm in length. IPS e.max Press is supplied for heat pressing in 2 sizes, a small ingot that weighs 3.2 g or a larger ingot that weighs 6.1 g. These ingots are pressed into a mold by an Alumina plunger under pressure from a pneumatic press furnace. After pressing and cooling, the sprues are removed, along with the remaining material (button). The buttons should be discarded and a new ingot should be used for a new pressing.[4]

It is more economical to press several restorations from one ingot at the same time. However, this often is not possible and may result in a considerable amount of leftover material (The remaining sprues and button). It has been reported that these remaining materials are recycled in some dental laboratories; sufficient knowledge about the safety and consequences of such treatment is not available. The issue is thereby raised whether these buttons can be repressed and recycled or should be discarded. Concerns have also been expressed regarding the change in the microstructure and possible degradation of the mechanical properties of these materials, as a result of multiple processing and subsequent heat firing.[3, 5]

Extensive research into the mechanical and optical properties as well as clinical performance of heat-pressed glass ceramics has been carried out over the past two decades.[6-18] In spite of this, only 3 studies that examined the mechanical and microstructural properties of re-pressed ceramics were identified by the present authors.

Albakry et al [3] (2004) evaluated the biaxial flexural strength (BFS) and identified the crystalline phases and the microstructural features of pressed and repressed materials of the glass ceramics (Empress I and Empress II). They concluded that the second pressing had no significant effect on the biaxial flexural strength of both glass-ceramics. However, higher strength variations among the repressed samples of the materials may indicate less reliability of these materials after second pressing.

Chung et al [19] (2009) studied the properties of four heat pressed glass-ceramic materials after repeated heat-pressing. Optimal pressable glass-ceramic OPC, 3G, Empress I and Empress II were evaluated. They stated that, repeated heat pressing treatment produced a statistically significant increase in the flexural strength of Empress II glass-ceramic material and Empress II secondary electron imaging (SEI) showed a densely packed, interlocking microstructure and an increase in size with preferred orientation of lithium disilicate crystals.

Gorman et al [5] (2014) investigated the effect of repeated pressing on the biaxial flexural strength, hardness and flexural toughness of lithium disilicate glass-ceramic (IPS e.max Press). They found no significant difference neither in BFS nor in flexural toughness but the hardness of the material decreased. Using x-ray diffraction analysis, lithium dislilicate was identified as the main crystal phase and no difference in crystalline composition was found with repeated pressing.

With only 3 studies that examined the mechanical and microstructural properties of re-pressed ceramics of which 1 has studied lithium dislilicate IPS e.max Press. Concerns whether repeated heat pressing affects the biaxial flexural strength and surface roughness of IPS e.max Press are still valid. Thus, the question is to reuse or not the left over material (sprues and button) is not yet answered.

The aim of the present study was to evaluate the effect of repeated heat-pressing on the biaxial flexural strength and surface roughness of lithium dislilicate glass-ceramics (IPS e.max Press). As well as to describe the microstructural features present in fresh-pressed, and repressed material using **Xray diffraction, EDAX and SEM**

MATERIALS AND METHODS

Twenty IPS e.max press discs (Ivoclar, Vivadent, Schaan, Liechtenstein) were prepared according to the ISO 6872 specifications for testing ceramic materials; discs of 15mm diameter, 1 mm thickness, in shade A3 were fabricated using heat pressed technique. A special custom made Teflon mold was constructed for this purpose. The discs were divided into two groups, Pressed group and Repressed group (n=10). For the Pressed group, discs were heat-pressed according to the manufacture's recommendation, then the leftover material was recovered where **Only** the button parts were adjusted by grinding to allow proper insertion into the refractory mold to construct specimens of re-pressed group by repeated heat-pressing. Pressing procedure used was the same for both pressed and repressed specimen groups. All discs were finished an glazed according to the manufacture's recommendation.

Surface roughness evaluation

All discs were cleaned ultrasonically in 99% alcohol solution for 3 minutes then dried with air. The average surface roughness (Ra) for the specimens was measured using a 3D laser scanning microscope (Keyence VK-X100, Keyence GmbH, Neu-Isenbuerg, Germany). The wave length of the laser was 658 nm. Three separate areas were measured on each disc, the measured area was 500μm x 750μm and the distance between the separate scans was over 3μm. The mean Ra for each disc was later recorded before and after glazing.

Biaxial flexural strength testing

Discs were tested for biaxial flexural strength using piston-on-three ball technique on Universal testing machine, Instron, (model LRX-plus,

LIoyd Instrument Ltd, Fareham, UK), version 4.3 software (Nexygen- MT-4.6, Lloyd Instruments Ltd, Fareham, UK. A custom-made 10 mm diameter metallic platform with three symmetrically spaced steel balls of 3.2 mm diameter was used to support the disc samples. Load was applied by a piston of 1.4 mm diameter at 0.5 mm/min crosshead speed. A thin plastic sheet (polyethylene film) was placed between the piston and the specimen to facilitate even load distribution and minimize surface contact damage as well as maximize the probability of interface-initiated cracks.

The fracture load for each disc was recorded and the biaxial flexural strength was calculated using the following equation:

$$S= [-0.2387P(X-Y)]/d^2$$

Where S: maximum tensile stress (Mpa), P: fracture load (N), d: specimen disk thickness at fracture origin (mm). X and Y were determined as follows:

$$X: (1 \pm n) \ln(r2/r3)^2 \pm [(1 - n) / 2] (r2/r3)^2$$

$$Y: (1 \pm n) [1 \pm \ln(r1/r3)^2] \pm (1 - n) (r1/r3)^2$$

Where n is the Poisson's ratio, r1 is the radius of the support circle(mm), r2 is the radius of the tip of the piston (mm), r3 is the radius of the specimen (mm). Poisson's ratio for tested materials, which is 0.24 for lithium disilicate ceramic material was taken from a previous report.

Xray diffraction and EDAX

For each group, the discs were submitted to XRD to determine the crystalline phases. Samples were placed on the holder of the diffractometer (X pert pro, USA; PW 3040/60) and scanned using Cu Kα xray angle from 20-40 degrees, 2θ with a step size of 0.04 degrees and 5s-step interval. EDAX (energy dispersive x-ray analysis) was carried out to quantify elements by x-ray microanalysis (FEI Czech SEM - USA).

Microstructure by SEM

For each group, the discs were submitted to scanning electron microscopy (SEM). Samples were cleaned and etched with 9.8% Hydrofluoric acid for 90 seconds, cleaned in an ultrasonic cleaner, steamed, then dried and sputter coated with gold. SEM (Quanta 250 FEG) was carried out to examine the microstructure and assess grains at magnification of 6000x

Statistical Analysis

The data were collected, tabulated then analyzed using two-way analysis of variance (ANOVA), followed by Tuckey's HSD test at a significance level of $p< 0.05$.

RESULTS

Surface Roughness

Regarding surface roughness, for both groups, significant difference was found between Glazed and Un-Glazed ceramics. No statistical significant difference was found between Pressed and Repressed groups. No interaction between surface glazing and type of ceramic (Pressed and Repressed) was found (Table-1).

TABLE (1) Mean Ra and standard deviation of test groups before and after glazing

Pressed				Repressed			
Glazed		Unglazed		Glazed		Unglazed	
Mean	SD	Mean	SD	Mean	SD	Mean	SD
0.27 [a]	0.05	1.14 [b]	0.16	0.21 [a]	0.06	1.33 [b]	0.20

Means with different superscript letters are statistically significant (p<0.05)

Biaxial Flexural Strength

Regarding Biaxial flexural strength, no significant difference was found ($p>0.05$) between the two tested groups (Pressed and Repressed) (Table-2).

TABLE (2) Mean BFS and standard deviation of test groups

Pressed		Repressed	
Mean	SD	Mean	SD
375.80 [a]	4.55	389.40 [a]	12.12

Means with different superscript letters are statistically significant (p<0.05)

X-Ray Diffraction Analysis (XRD)

X-Ray Diffraction Analysis (XRD) investigates crystalline material structure, including atomic arrangement, crystallite size, and imperfections. The X-ray analysis (XRD) of both pressed and repressed samples detected diffraction peaks that correspond to crystalline phases present indicating that the material is predominantly crystalline structure; lithium disilicate was identified to be the main crystalline phase. Major peaks for lithium disilicate (Li2Si2O5) were observed at 2θ values of 24.7 degrees, 24.2 degrees, and 40 degrees. The dominant peak (highest peak) was at 24.7 degrees, which corresponds to the (040) crystallographic plane of this monoclinic phase, Corresponding to the standard peaks for lithium disilicate (Fig. 1).

The XRD data showed that peaks after pressing and repeating pressing are similar, the crystalline phase assemblage did not change; however their radiation intensities (height) has, the dominant peak (highest peak) for the repressed sample is smaller compared to the pressed.

Microstructure by SEM

The SEM image observations (6000x); in pressed samples the length of lithium disilicate crystals averaged 3.05 μm in length while they averaged 473 nm in width, compared to repressed that averaged 6 μm in length while they averaged 500 nm in width. There is a noted increase in dimension after repressing (Fig-2).

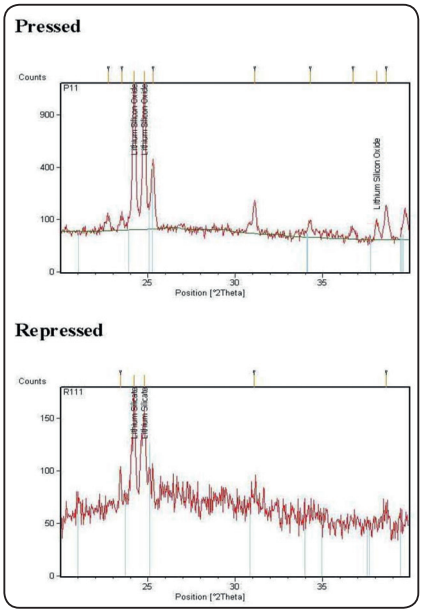

Fig. (1) X-ray diffraction (XRD) patterns of IPS e.max Press (Pressed) and (Repressed) showing peak positions in agreement with those of standard Lithium disilicate

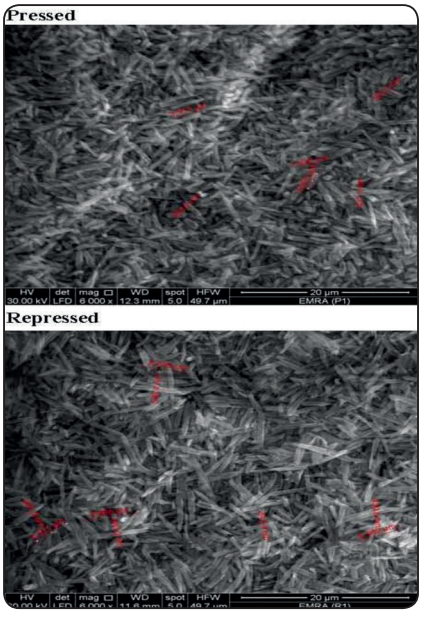

Fig. (2) SEM of IPS e.max Press (Pressed) and (Repressed) showing noted increase in dimension after repressing

EDAX

Energy Dispersive X-Ray Analysis (EDAX), is an x-ray technique used to identify the elemental composition of materials., it includes a lot of areas of applications. EDAX systems are attachments to Electron Microscopy instruments (Scanning

Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM) instruments where the imaging capability of the microscope identifies the specimen of interest. The data generated by EDAX analysis consist of spectra showing peaks corresponding to the elements making up the true composition of the sample being analyzed. It also allows elemental mapping of a sample and image analysis. It can be qualitative, semi-quantitative, and quantitative – it also provides spatial distribution of elements through mapping. The EDAX technique is non-destructive and specimens of interest can be examined in situ with little or no sample preparation. EDAX results showed no change in composition between pressed and repressed samples (Fig-3).

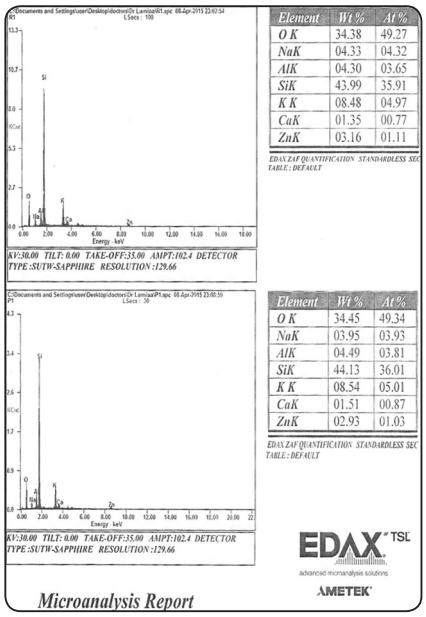

Fig. (3) Microanalysis by EDAX of IPS e.max Press (Pressed) and (Repressed)

DISCUSSION

Presence of pores in the bulk or surface of dental ceramic has a detrimental influence on the flexural strength of dental ceramics. [10, 20, 21] Therefore porosity control should be a fundamental consideration during fabricating dental restoratives using glass ceramic, in order to obtain optimal strength.

Chung et al [19] found by SEM scans (5000x) multiple small pores located in the glassy matrix and at the lithium disilicate crystal grain boundaries, and had a higher etching rate than the lithium disilicate phase. They said that it is possible that the small pores observed by SEM in the microstructure of the lithium disilicate reinforced glass-ceramic were precipitates of Li_3PO_4 that may act as sites for the nucleation of stable lithium disilicate. They added that, there is a possibility of having an increase in porosity and crack because of multiple nucleation sites during crystallization. In the present study, SEM scans (6000x) results for both groups (pressed and repressed) were found to be almost free of pores in the surface (Fig. 2). This pore-free microstructure was mainly attributed to using only the left over buttons and not using the left over sprues. This prevented trapping of air in-between the repressed material thus producing a repressed ceramic with nearly pore–free structure that is similar to that provided by the manufacturer and was used for the pressed group. This was not the case with **Chung et al** [19] who used both left over sprues and buttons to prepare the repressed samples.

The XRD and SEM data confirmed that the tested lithium disilicate-reinforced glass-ceramic materials contained lithium disilicate as a major crystalline phase, and that the amount of lithium disilicate did not increase as a result of repeated heat-pressing. Lithium disilicate elongated crystals were present in the glass matrix. They appeared to form an interlocking pattern in some sites; however, the lithium disilicate crystals in the repressed material were seen to be larger than those of the pressed samples. These results coincide with **Albakry et al** [3] **and Gorman et al** [5]**.** They stated that this behavior is called "Ostwald ripening" and is common for all precipitated materials. It takes place when the microstructure coarsens and liberates surface energy excess due to the solubility of small particles. As a consequence, larger grains are expected to grow at the expense of those small particles.

Surface roughness (Ra) refers to the finer irregularities of the surface texture that usually result from the action of the production process or material condition and is measured in micrometers (μm) [22]. Surface roughness of IPS e.max Press was not affected by repeated heat pressing. These results coincide with **Albakry et al** [3] who stated that no changes in the microstructure was found after repeated pressing, subsequently the second press has the same surface texture of the first one after finishing and glazing. However, Surface roughness was affected by glazing for both groups as it decreased significantly. This is due the fact that the purpose of glazing is to seal the open pores in the surface of fired porcelain decreasing its surface roughness. [23]

In the present study, Biaxial flexural strength (the mean ± SD BFS for Pressed group was 375.80 ± 4.55 MPa and Repressed group 389.40 ± 12.12 MPa) wasn't affected by repeated heat pressing. Although BFS was improved, yet it wasn't statistically significant. These results coincide with **Albakry et al** [3]. In their study; (the mean ± SD BFS for Pressed group was 340 ± 40 MPa and Repressed group 325 ± 60 MPa). They said that the improved mechanical properties of lithium disilicate ceramic material was attributed to the unique interlocking microstructure of densely packed high content of lithium disilicate crystals. The preferred orientation of these crystals after pressing may occur and cause fracture toughness anisotropy. This alignment may also result in overestimation of the fracture toughness and strength if stresses, during testing, are applied parallel to these aligned elongated crystals.

However, our results coincide with **Gorman et al**[5] and **Chung et al** [19] regarding BFS improvement without being statistically significant. Yet, our results as well as **Albakry et al** [3] were within the same range to those values reported by the manufacturer, while **Gorman et al** [5] and **Chung et al** [19] were lower.

Gorman et al [5] results were (The mean ± SD BFS for Pressed group was 243.4±45.8 MPa and Repressed group 252.7±23.7 MPa). They claimed that their samples surface finish may be responsible for the recorded lower values, because flexural strength measurements have been shown to depend on surface finish. Also residual stresses that are normally present as a result of manufacturing processes may have lead to decrease in BFS than that reported by the manufacturer (From 300-450 MPa) [24]. **Chung et al** [19] results were (The mean ± SD BFS for Pressed group was 281.2 ± 42.2 MPa and Repressed group 365.9 ± 35.5 MPa) They claimed that annealing may have contributed to the higher flexural strength values found in **Albakry et al** [3] compared to their study. They mentioned that all the specimens prepared in **Albakry et al** [3] study were fired again in a porcelain furnace to release stresses before the flexural strength test. The strength of glass-ceramic materials could be increased significantly after annealing because of relaxation of residual stress. Therefore, it was not surprising to find that there was no significant difference in strength between the pressed and recycled specimens after annealing.

In our study, the glazing cycle may have played this important role to release residual stresses. Not to mention that **Gorman et al** [5] and **Chung et al** [19] have used both leftover (sprues and buttons) to prepare the repressed samples which is not the case with our present study and **Albakry et al** [3]**,** hence the importance of pore–free structure for optimal strength results.

CONCLUSIONS

1- The microstructure of IPS e.max Press before and after re-pressing did not change. Indicating that lithium disilicate glass-ceramic materials have reached their maximum crystallinity prior to re-pressing.

2- Repeated heat pressing has no significant effect on Biaxial flexural strength and surface roughness of IPS e.max Press.

3- It is important to produce a pore–free structure by only repressing the left over buttons and not the left over sprues.

Therefore according to the present investigation the IPS e.max Press left over pressed buttons can be safely reused with no consequent negative effects on Biaxial flexural strength and surface roughness. Further investigations, regarding color reproduction and translucency are recommended.

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