



Assessment of Chemical Composition and Mechanical Properties Effect on Marginal Chipping after Repeated Heat Pressing of Dental Ceramic Materials

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Abstract

Objective: The aim of this study is to evaluate the effect of ceramic repressing on marginal chipping of two different pressable ceramic materials.

Materials and Methods: Two commercially available glass ceramic materials were used in this study; IPS E-max press (Ivoclar, vivadent) and Celtra press (Dentsply, Sirona). The two materials were used to fabricate veneer samples as which a total of forty veneers were constructed. The samples for each material (n=20) were randomly divided into two equal groups; Group A: Pressed specimens (n=10) veneer shaped wax patterns were invested and heat-pressed according to the manufacturer's recommendations and Group B: Re-pressed specimens (n=10) the leftover material from 1st pressing was recovered and the buttons were adjusted to fabricate the specimens by repeated heat-pressing using the same procedures as for (A). A stereomicroscope was used to measure the amount of marginal chipping of each veneer in microns (L). The chipping factor (CF) was calculated using the following equation: $CF = L/P \times 100$, where (L) is the amount of marginal chipping and (P) is the marginal circumference of each veneer. A sharp conical head indenter was fixed on a universal testing machine (Instron, 3345, UK). Chipping factor were recorded and mean values for each group determined. Data was statistically analyzed.

Results: For IPS E.max or Celtra press; there was no statistically significant difference between mean values of press and repress conditions ($P < 0.05$) between tested groups. Chipping factor mean values was recorded for Celtra press (14 ± 1.1) while E-max press was recorded (13.9 ± 0.5), Celtra repress recorded (13.8 ± 0.7), however E-max repress recorded (13.5 ± 1) which have the lowest chipping mean value.

Conclusions: The optimum properties for lithium disilicate Press ceramic materials are obtained with the first pressing. However, multiple heat repressing could affect the microstructure and mechanical properties but with no statistically significant difference on surface marginal chipping.

Keywords: Chemical Composition, Marginal Chipping, ceramic-fixed prostheses.

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1. INTRODUCTION

Increasing interest in ceramic-fixed prostheses has followed improvements in aesthetics, strength and ease of fabrication. More recently, a multiphase glass-ceramic system was introduced with a high degree of crystallinity and reinforced with lithium disilicate.

The persistent demand for obtaining all ceramic restoration that merges between excellent esthetics and optimum mechanical properties has participated to the elaboration of reinforced glass ceramics ,for example zirconia reinforced glass ceramics (Celtra

Duo, Celtra Press), manufactured by CAD/CAM system and Pressing technology respectively⁽¹⁾.

Glass-ceramics exhibit some compositional and micro-structural differences and combine properties that are typical for both ceramics and glasses⁽²⁾. Ceramic restorations are fabricated by sintering, slip casting, heat pressing, and milling⁽³⁾. Heat pressing has advantages over sintering and slip casting in terms of porosity and marginal fit⁽⁴⁾.

Pressable ceramics are categorized into two generations; the first-generation is leucite-based while the second generation is lithium disilicate

based^(5, 6). 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⁽⁷⁾.

Leucite-reinforced and lithium disilicate reinforced glass-ceramic materials are available in ingots with different shades to match various clinical requirements. During laboratory procedures, these ingots are heat pressed into a mold by an alumina plunger under pressure within a pneumatic press furnace.

After pressing and cooling, the button and sprue portions are removed and usually discarded. However, these residual materials are found to be useful for re-pressing in some dental laboratories as it is more cost effective for them to reuse what is often wasted material to press multiple restorations, thereby reducing the quantity of wasted material. In addition, repressing leftover material will reduce treatment expenses for the patient and preserve environmental resources.

So, it is important to evaluate the properties of repressed glass ceramic material to determine the feasibility of repeated heat-pressing treatment. It is hypothesized that after repeated heat pressing, the recycled materials will maintain the same microstructure and mechanical properties as that of the original pressed material.

The aim of this study is to investigate the effect of repressing IPS e.max Press and Celtra press on the marginal chipping of ceramic veneers. The hypothesis is that ceramic repressing will not affect the marginal chipping as those of one heat-pressing.

2. MATERIAL AND METHODS

Two commercially available glass ceramic materials were used in this study; IPS e.max press (Ivoclar, vivadent) and Celtra press (Dentsply, Sirona). The two materials were used to fabricate veneer samples as which a total of forty veneers were constructed. The samples for each material (n=20) were randomly divided into two equal groups; Group A: Pressed specimens (n=10) veneer shaped wax patterns were invested and heat-pressed according to the manufacturer's recommendations and Group B: Re-pressed specimens (n=10) the leftover material from 1st pressing was recovered and the buttons were adjusted to fabricate the specimens by repeated heat-pressing using the same procedure as for (A).

A natural extracted maxillary central incisor which free from any pathosis, the tooth which collected was kept in thymol 0.1% to avoid dehydration. The tooth

was mounted in epoxy resin blocks using a special device (parallometer).

Self-limiting depth-cutting of 0.5 mm (NTI, Kerr dental, USA, 0.5 mm, .03mm) were used to define the depth cuts, followed by a diamond bur (Kerr dental, USA). to refine the preparation. Thicknesses of the labial surface were prepared 0.5 mm. No incisal reduction, but 0.5 mm facio incisal surface of the tooth was reduced and 0.2 mm bevel was placed at the expense of the labial surface.

Forty resin dies of yellow shade were fabricated to act as replica for a prepared upper central incisor. The resin dies (Resin ABS-V2.0 yellow, power resins, 3BFAB, Teknoloji A.Ş., Istanbul, Turkey) were obtained by scanning the prepared teeth by extraoral scanner (T310, Medit, Korea) and then printed by 3D Printer (Mars 3, Elegoo, china) which used for duplication and making an exact replica for the prepared status.

The resin dies scanned using laser scanner. The wax-patterns (power resins, 3BFAB, Teknoloji A.Ş., Istanbul, Turkey) were produced with 3D printer using laboratory cast scanner to digitize the dies, after they were sprayed with scan spray, then forty standardized wax patterns designed were fabricated on ready dies in which the wax patterns were designed with a thickness of 0.5mm.

The wax patterns were sprued (Kerr, Orange, CA) and then Sprues were attached to the IPS silicon investment ring System. The ring was filled with investment material and was allowed to set for 35 minutes. The investment ring was placed in the preheated furnace (*Vulcan 3-130, Degussa-Ney Yucaipa, CA, USA*). The ceramic ingots of IPS E.max (*Ivoclar-Vivadent, Schaan, Liechtenstein*), Celtra (*Dentsply Sirona, NC, USA*) Press were then plastified and pressed under vacuum into them old of the investment in a press furnace (*EP600 combi, Ivoclar-Vivadent, Schaan, Liechtenstein*).

The heat-pressing conditions were as the manufacturer instructions. After pressing, the investment molds were removed from the furnace and allowed to air cool.

The specimens were then carefully divested using an air abrasion unit with 50 µm glass beads at a pressure of 3bar. The button and sprue portions were cut; 20 specimens were selected randomly. For the remaining specimens, the button and sprue portions were adjusted by grinding to allow proper insertion into the refractory molds for repeated heat-pressing. With the same heat-pressing conditions an additional 20 specimens were fabricated.

Bonding protocols were followed in cementation of all veneers according to the manufacturer's recommendations to avoid any variables during

bonding procedures. The veneers were cemented using Bisco Bissem dual cure self-adhesive resin cement (*Choice 2, Bisco, USA*). Luting procedures followed the clinical protocols to ensure a close simulation of clinically relevant conditions.

All specimens were subjected to thermocycling procedures in automated thermocycling machine to mimic the oral conditions. Samples of retention test were thermocycled for 5000 cycles, between 5°C-55°C with a dwell time 15 seconds, but specimens of microleakage test the number of cycles used was 500

cycles between 5°C-55°C with a dwell time 25 seconds.

A stereomicroscope was used to measure the amount of marginal chipping of each veneer in microns (L). The chipping factor (CF) was calculated using the following equation: $CF = L/P \times 100$, where (L) is the amount of marginal chipping and (P) is the marginal circumference of each veneer.

A sharp conical head indenter with a diamond tip at 120 degrees and tip sharpness under 5 μm (*Gilmore Diamond Tools, Inc*) was fixed on a universal testing machine (*Instron, 3345, UK*) as shown in **Figure (1)**.

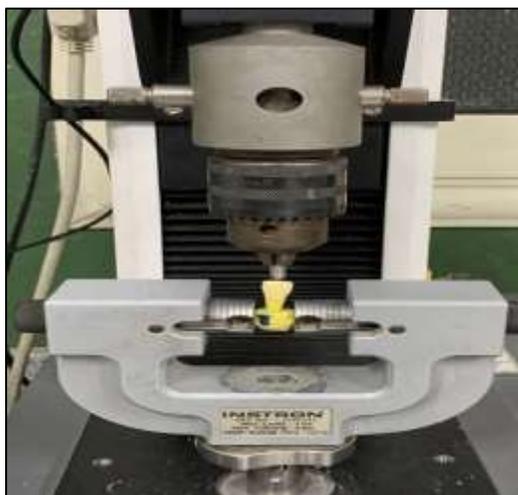


Figure (1): Indentation of the veneer by a sharp conical head indenter attached to Universal testing machine.

All distances were from the specimen edge to the center of the point of load application, the loads was 100 (N) which causing the edge of the specimen to spall and marginal chipping of the edges of specimens were measured using stereo microscope (MA 100Nikon stereomicroscope, Japan with omnimet image analysis software) as shown in figure (2).

All testing was done in laboratory ambient conditions. When a chip popped off, a sudden force drop off was detected by the break-load detection circuitry of the machine and the indenter extracted automatically and the peak load recorded.

A top view image of the margins of each veneer was taken using a digital camera connected to a PC to

measure the peripheral circumference (P) of the veneer using the Adobe Photoshop software (Adobe Inc. system V5. 0. Ltd. Europe). The images were then imported into image software. The average periphery of a veneer margins was calculated.

Analysis of the marginal quality on marginal after heat repressing of each veneer from incisal aspect through distribution of the chipping defects was performed. The length of the chipped margins of each veneer was measured using a stereo microscope at magnification 25X and the total amount of each specimen was calculated in microns (L)

The data of the chipping factor of each veneer margins were recorded and tabulated for statistically analyzed with Two-way ANOVA Samples.



Figure (2): Edge chipping under stereo microscope

3. RESULTS

The results showed that ceramic material, technique and the interaction between the three variables had a statistically insignificant effect on mean retention.

Table 1: Range, mean and standard deviation between different techniques in different material (E-max, Celtra).

Material	Marginal chipping	Technique		P value
		Press	Repress	
		N=10	N=10	
Celtra	Range	(12.1-16)	(13-14.9)	0.624
	Mean \pm SD	14 \pm 1.1	13.8 \pm 0.7	
E-max	Range	(13.2-14.6)	(12-14.9)	0.256
	Mean \pm SD	13.9 \pm 0.5	13.5 \pm 1	

- Independent Samples T test; Significant level at P value < 0.05

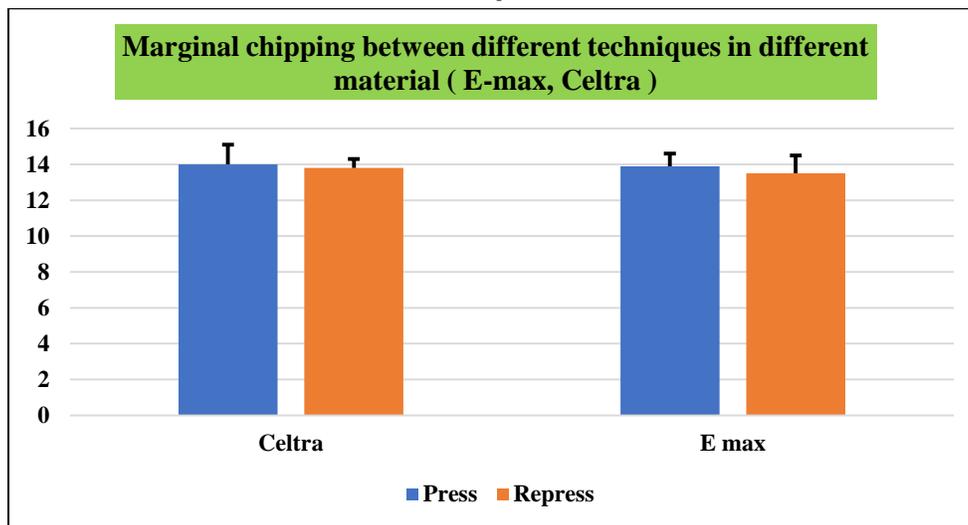


Figure (3): Histogram showing chipping mean values for press and repress in two materials (E-max and Celtra).

It was found that chipping factor mean values was recorded for Celtra press recorded (14 \pm 1.1) which was the highest chipping value then E-max press was recorded (13.9 \pm 0.5) , Celtra repress recorded

(13.8 \pm 0.7) , however E-max repress recorded (13.5 \pm 1) which was the lowest chipping value . The difference between groups was statistically

insignificant as indicated by two way ANOVA test P value < 0.05 .

4. DISCUSSION

Despite of the continued evolution of dental material sciences, masticatory and para functional occlusal forces can cause mechanical failure and degradation of dental restorative materials⁽⁸⁾.

Previous studies have shown that chipping is a relatively frequent mechanical problem for dental hard tissues^(8, 9), ceramic materials⁽¹⁰⁻¹⁸⁾, and polymer-based materials^(13, 14, 16, 19, 20), especially when subjected to excessive masticatory forces⁽²¹⁻²³⁾.

Clinically, minor chipping often causes marginal infiltration and/or discoloration of the tooth-restoration interface, which may result in restoration loss⁽²⁴⁾.

More specifically, a chip is a small broken or spall-off piece from a brittle material. Chipping can either be the primary mode of fracture or a secondary, minor resultant from the fracture process. In clinical dentistry, chipping usually occurs when a load near an edge of a tooth or restoration causes to chip off a portion of it.

Such fracture process initiates beneath a concentrated contact by subsurface crack formation that propagates unstably towards a free edge (adjacent surface) to form the chip. *in vitro* researches have used the edge-chipping test that applies an increasing force near the edge of a sample until a chip forms^(17, 24).

The edge chipping test was originally developed at the National Physical Laboratory (NPL), London, UK, in the 1980s to evaluate hard metals⁽²⁵⁾.

Quinn et al.,⁽²⁶⁾ introduced it to Dentistry to evaluate brittle structures, such as human teeth and restorative dental materials, with the purpose of measuring the force necessary to generate a chip. A crack is intentionally created near the edge of a structure using an indenter, which is linked to a load cell chips by loading the structure with an indenter at a determined distance from the edge and recorded the force required to create the chip^(12, 27).

Chipping is often reported as the clinical failure cause for failed ceramic restorations⁽²⁸⁻³⁰⁾, which may explain the amount of edge chipping studies on ceramic materials (45%). Multilayer ceramic structures are more susceptible to chipping, regardless of the infrastructure composition or fabrication technique⁽³¹⁻³⁷⁾ compared to monolithic ceramic restorations⁽¹⁷⁾, which often offer greater fracture resistance⁽¹²⁾. Thus, chipping of the veneering ceramic remains the main reason for the failure of multilayer restorations^(30, 31).

For posterior restorations, chipping fracture is usually a typical contact damage failure mode, starting from a

wear facet on the occlusal surface. Consequently, microcracks develop under the contact zone propagate as a single crack within the veneer material⁽³¹⁾.

Yet, the conditions and mechanisms of chip formation and the resultant chip size are dependent on material properties. For example, materials with similar *to* but different elastic moduli result in differences in strain energy release rates⁽²⁶⁾.

The chipping factor varies according to the material used, this might be the case for the IPS e.max ceramic whose physical properties are improved by subsequent firing.

The result in this study revealed that E-max repress group (C) recorded (13.5±1) which was the lowest chipping mean value as which statistically insignificantly with chipping mean values of Celtra press group (B) which was recorded the highest chipping mean value (14±1.1).

These results may be due to the brittleness index (BI) of the brittle materials and the chipping factor (CF) of the tested materials when were compared, it was clear that there was a correlation between them as the chipping factor was increasing as the brittleness index increased. Correlation analysis verified this, giving a perfect positive correlation relationship between BI and CF ($r_s = 1$)⁽³⁸⁾.

Besides various combinations of hardness, stiffness, fracture toughness and other parameters have been used to indicate a degree of brittleness as which the correlation between brittleness and hardness and fracture toughness as mentioned with the equation

$$B = \frac{H}{K_{Ic}}$$

as quantification of the brittleness of materials (B) that can be derived from the hardness (H) and fracture toughness (K_{Ic}) of the material⁽³⁹⁾.

Accordingly with the last two relations, the more the hardness of the material, the more the brittleness of the material and so on more liable for edge chipping defect. Therefore Celtra ceramic material which is harder than E-max ceramic material as Stawarczyk B, et al., in 2020 concluded⁽⁴⁰⁾, and so having more chance to chipping.

Regarding the material of construction; there was statistically insignificantly when comparison between the two materials in different heat pressing cycles (pressing new ingots and after one repeated heating).

When comparing the two materials in the same heat pressing cycle as in new heat pressing by E-max have statistically insignificant mean chipping values (13.9±0.5) while veneers constructed by Celtra (14±1.1). Also, after repeated heat pressing by Emax have statistically insignificant mean chipping values (13.5±1) while veneers constructed by Celtra (13.8±0.7).

These results may be attributed to the difference in compositions and material properties of E-max press and Celtra press which may influence the hardness of restoration.

Besides repeat heat pressing there were changes in the micro-structural of the material and mechanical properties. As the original glass composition as well as the presence, volume fraction, crystal size, distribution and morphology of the crystalline phases may account for the variations in the ceramics' mechanical properties⁽⁴¹⁾. As the final crystalline form depends on the glass composition, nucleating agent, and method of heating⁽⁴²⁾, the morphology and size of the crystals play a significant role in the determination of mechanical properties⁽⁴³⁾.

As which the crystal alignment during the heat treatments depends on the crystal shape, the proportion, size and viscosity of the remaining glass phase⁽⁴⁴⁾.

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⁽⁴⁴⁾.

Hallmann L, et al 2019 founded when analyzing IPS e.max Press(IE), Celtra Press, and Initial LiSi Press (IL) that the transformation of lithium silicate to the lithium disilicate phase was completed for IE and IL but no for Celtra Press. As which after pressing, rod-shaped crystals were aligned parallel to the direction of extrusion, whereas platelet shaped crystals, having an interlocking microstructure, were not⁽⁴³⁾.

Consequently, larger grains are expected to grow at the expense of small particles. This may be the result of the phase transition between lithium metasilicate and lithium disilicate⁽⁴¹⁾.

The orientation of the lithium disilicate crystals was probably a result of plastic deformation of the glass matrix phase and occurred during sprue extrusion⁽⁴⁵⁾.

And according to the manufacturer, Celtra® Press has mechanical properties values that are comparable with lithium silicate glass-ceramics^(40, 43).

Celtra® Press is a newly introduced zirconia-reinforced lithium silicate (ZLS) with the addition of 10% wt. zirconium oxide (ZrO₂) as a nucleating agent. During heat pressing, ZrO₂ promotes volume crystallization of glasses and hinders crystal growth⁽⁴⁶⁾.

Strengthening methods of glass-ceramics include ion exchange to form surface compressive stresses and the addition of ZrO₂ with different concentrations to form zirconia-toughened glass-ceramics^(47, 48).

The absence of ZrO₂and/or ZrSiO₄reflections in the X-ray diffractograms and in the Raman spectra for the Celtra Press specimens indicates that ZrO₂is

dissolved in the glass matrix and serves as a network modifier⁽⁴³⁾.

By consideration the later changes in the morphology and size of the crystals after repeated heat pressing which alter the microstructure and mechanical properties of the materials as among these properties the hardness and fracture toughness which responsible of cracks origination and propagation as our results in this study revealed that there was statistically insignificant decrease in hardness after repeated heat pressing of the ceramic materials and so on become less liable to chipping.

Results showed that statistically insignificant decrease in chipping mean values after repeated heat pressing as E-max press recorded (13.9±0.5) while E-max repress recorded (13.5±1), also pressing group in Celtra material noted (14±1.1) but Celtra repress recorded that (13.8±0.7).

Also, these results may be due to a, there is a possibility of increased porosity, cracks and decreased density during repressing procedures 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⁽⁴⁵⁾. These results and observations were later supported by Tang X et al. who also reported a reduction in flexural strength of repressed lithium disilicate. Through their observations, they declared that with repeated heat pressings there was a decrease in density together with increased porosity possibly because of the numerous nucleation sites during crystallization⁽²⁾.

Our findings are in agreement with Wahba M et al. assumed that it was clear that repeated heat pressing was associated with a decrease in Vicker's hardness values although this decrease was not significant⁽⁴⁹⁾.

And Gorman CM et al. which 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⁽⁵⁰⁾.

Some studies have shown that repeated heat-pressing significantly influenced the microstructure of lithium disilicate-reinforced glass-ceramic materials. These findings were not in agreement with our findings.

Such as Tang X. et al. who 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⁽²⁾.

Based on the obtained results, the hypothesis was accepted as there was no significant difference in marginal chipping between the first heat pressing and repeat heat pressing.

5. CONCLUSIONS

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

1-The optimum properties for LiSi Press are probably obtained with the first pressing. However, one heat repressing could affect the microstructural composition and mechanical properties of LiSi Press, but with no noticeable effect on marginal chipping defect.

2-Further investigation could be done for the effect of much heat repressing cycles and different weight percentage of new and repressed ceramics on chipping of the material.

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