

**EVALUATING THE EFFECT OF REPEATED HEAT** PRESSING ON STRENGTH AND HARDNESS OF A NEW LITHIUM DISILICATE CERAMIC MATERIAL

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#### ABSTRACT

Objective: To investigate the effect of repeated heat treatments on flexural strength and hardness of a new lithium disilicate ceramic.

Materials and Methods: Thirty specimens of pressable lithium disilicate were prepared in the form of discs 15 mm in diameter and 1 mm in thickness and were fabricated using heat pressing technique. The discs were randomly divided into three groups (n=10); Press group (P1), 1<sup>st</sup> Repress group (P2), and 2<sup>nd</sup> Repress group (P3). For (P1), disc-shaped wax patterns were invested and heat-pressed according to the manufacturer's recommendations. For (P2) group, 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 (P1). The same procedure was repeated for (P3) group specimens fabrication. All specimens were tested for biaxial flexural strength. For the Vickers Hardness test, one fractured segment of each specimen was chosen randomly for the test and hardness was measured using Vickers hardness tester. Data was statistically analyzed by ANOVA and Tukey's post hoc analysis ( $\alpha = 0.05$ ).

Results: For biaxial flexural strength, there was a significant difference (P < 0.05) between tested groups. (P2) group showed the highest flexural strength value (403.69) while (P3) group showed the lowest value (353.22). For hardness, there was no significant difference between tested groups (p=0.055).

Conclusions: The optimum properties for LiSi Press are obtained with the first pressing. However, multiple heat repressing could significantly affect the strength of LiSi Press, but with no noticeable effect on surface hardness.

KEYWORDS: Flexural strength, Heat pressing, Lithuim disiicate, Surface hardness.

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# INTRODUCTION

With the revolutionary wave of esthetics taking over the whole world in the past recent years, the demand for dental esthetics prevailed as well. Accompanied by this wave were the rising concerns regarding metal allergies, all of which called for the introduction and use of metal-free all-ceramic restorations for prosthetic rehabilitations <sup>(1,2)</sup>.

Several all-ceramic materials and processing techniques have been introduced over the years, yet these ceramics differ in their composition and crystalline structure, which consequently influence their mechanical and physical properties <sup>(3)</sup>. Almost two decades ago, lithium disilicate glass ceramics appeared to hold a peculiar place in the scene of prosthetic dental materials thanks to their good mechanical performance and natural esthetic appearance. Consequently, they were able to surmount most competitors within that range of glass ceramics <sup>(4,5)</sup>.

For lithium disilicates, the ratio of  $\text{Li}_2\text{O}$  and  $\text{SiO}_2$  is considered essential for the formation of  $\text{Li}_2\text{SiO}_3$ ,  $\text{Li}_2\text{Si}_2\text{O}_5$ , or  $\text{Li}_4\text{SiO}_4$ . In fact, the increase in the crystalline content up to 60-70% led to the appearance of glass ceramics having flexural strength values about two to three times greater than that of unreinforced glass ceramics. However, the variation of processing parameters such as temperature can influence the crystals' amount, type, orientation as well as porosities, and shrinkage, which consequently influences the mechanical properties like, for instance, the fracture toughness or flexural strength <sup>(3,6-8)</sup>.

Dental lithium-based glass ceramics are considered non-stoichiometric where they are not highly stable when considering their solubility. Rather, they are based on multi-component compositions, where oxides such as  $Al_2O_3$ ,  $K_2O$ , and  $CeO_2$ , Na2O, are added to enhance resistance to solubility and reduce melting. On the other hand, 2-6 wt % of diphosphorus pentoxide ( $P_2O_5$ ) is added to the base glass as almost exclusively the single nucleation agent used to enhance the nucleation and crystallization kinetics. However, though increasing the amount of  $P_2O_5$  leads to decreasing the activation energy for the crystallization of  $Li_2SiO_3$ , yet it promotes the formation of a crystalline lithium orthophosphate phase which is regarded as being stable at higher temperatures <sup>(9,10)</sup>.

Currently, available lithium disilicates can be fabricated through either heat-pressing or CAD/ CAM milling. Lithium disilicate pressing using the lost wax technique has been reported to result in production restorations with a good marginal fit, high strength, and better fracture toughness in comparison to those obtained through milling technique. Such findings might be attributed to the different heating parameters that are known to probably affect the growing lithium disilicate crystals and modify the overall percentage of residual glasses <sup>(2,11,12)</sup>.

For heat pressing, lithium disilicate ingots are subjected to pressing under pressure into a mold in a press furnace with both the sprue and the button parts being discarded and ditched following the first press process. However, for economic reasons, some dental laboratories consider such parts useful and reuse them. They recycle such leftovers and reuse them for new pressings. Consequently, assessing the microstructure as well as the mechanical properties of such re-pressed glass ceramics to ascertain the risk of repeated heat pressing is considered crucial <sup>(13)</sup>.

Although multiple studies have investigated the result multiple heat pressings could have on the mechanical properties of those repressed ceramics <sup>(14-16)</sup>, lately, several manufacturers have introduced novel lithium disilicate glass ceramic systems to the market with no enough data. However, it was reported that different lithium disilicate ceramics from different manufacturers exhibited different sizes and distributions of lithium disilicate crystals

that could typically affect their mechanical and physical properties <sup>(17)</sup>.

In light of the aforementioned data, this study aimed to evaluate the effect of repeated heatpressing on the biaxial flexural strength and hardness of a novel lithium disilicate glass ceramic. The null hypothesis was that repeated heat pressing will not affect the flexural strength nor hardness of the lithium disilicate tested.

# MATERIALS AND METHODS

Thirty specimens of pressable lithium disilicate (GC Initial LiSi Press GC America) were used in this study. Specimens were prepared in the form of discs 15 mm in diameter and 1 mm in thickness and were fabricated using heat pressing technique. The discs were randomly divided into three groups (n=10); Press group (P1), 1<sup>st</sup> Repress group (P2), and 2<sup>nd</sup> Repress group (P3). For (P1), disc-shaped wax patterns were first sprued and attached to the base former with a silicone ring. Discs were invested and heat-pressed according to the manufacturer's recommendations. After pressing, the investment molds were moved out from the furnace and left to air cool. The specimens were then devested through air abrasion using 50 µm alumina particles at 3 bar pressure. For (P2) group, the leftover material was recovered where the buttons were separated from the pressed discs using a diamond-cutting saw (Isomet, Buehler Ltd.Lake, IL, USA). The buttons were then tailored through grinding to facilitate their placement into the refractory mold to fabricate the specimens by repeated heat-pressing using the same procedure as for (P1). The same procedure was repeated for (P3) group specimens fabrication. After pressing and repressing, specimens were finished and glazed according to the manufacturer's recommendations.

All specimens were then tested for biaxial flexural strength using piston-on-three ball test. Specimens were placed concentrically on a custommade 10 mm diameter metallic platform with three symmetrically spaced steel balls of 3.2 mm diameter each and load was applied with a universal testing machine (Instron Co., Canton, Mass.), through a flat punch of tip diameter 1.4 mm at cross-head speed 0.5 mm/min. 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:  $(1 \pm n) \ln(r_2/r_3)^2 \pm [(1 n)/2] (r_2/r_3)^2$
- Y:  $(1 \pm n) [1 \pm \ln(r_1/r_3)^2] \pm (1 n) (r_1/r_3)^2$

Where (n) is the Poisson's ratio,  $(r_1)$  is the radius of the support circle(mm),  $(r_2)$  is the radius of the tip of the piston (mm),  $(r_3)$  is the radius of the specimen (mm). Poisson's ratio is 0.24 for lithium disilicate ceramic material.

For the Vickers Hardness test, a fractured segment from each specimen was randomly chosen for the test. Hardness was measured using micro-hardness tester (Tukon 1102 Wilson, Buehler, Germany) with a load of 1000 gm applied without impact, forcing the indenter into the test specimen. The indenter was held in place for 15 seconds. Following the removal of the load, the indentation was observed with a magnifying eyepiece and the two impression diagonals were measured, usually to the nearest 0.1- $\mu$ m with a micrometer, and averaged. The Vickers hardness (HV) was then calculated through the following equation;

### $HV = 1854.4L/d^2$

Where, (L) is the load applied and (d) is the average diagonal.

Numerical data were presented as mean and standard deviation (SD) values. In order to test for

normality, Shapiro-Wilk's test was implemented while Levene's test was used to test for homogeneity of variances. Data showed parametric distribution and variance homogeneity and were analyzed using one-way ANOVA followed by Tukey's post hoc test. The significance level was set at p<0.05 within all tests.

## RESULTS

Results of intergroup comparisons for biaxial flexural strength values presented in table (1) showed that there was a significant difference between tested groups (p<0.05). (P2) group showed the highest flexural strength value (403.69±22.15) while (P3) group showed the lowest value (353.22±39.31). There was no statistically significant difference between group (P) and both (P1) and (P2) groups.

On the other hand, the results of intergroup comparisons for hardness values displayed in table (2) showed that there was no statistically significant difference between tested groups (p=0.055).

TABLE (1): Intergroup comparison of biaxial flexural strength values (MPa)

Biaxial f	£			
First pressing	Second pressing	Third pressing	j-value	p-value
359.77±38.30 <sup>AB</sup>	403.69±22.15 <sup>A</sup>	353.22±39.31 <sup>B</sup>	4.52	0.034*

Means with different superscript letters within the same horizontal row are significantly different \*significant (p<0.05)

	Hardness (Mean±SD)			
First pressing	Second pressing	Third pressing	– <i>f-value</i>	p-value
585.60+15.12 <sup>A</sup>	608.68+22.26 <sup>A</sup>	606.60+13.96 <sup>A</sup>	3.73	0.055

TABLE (2): Intergroup comparison of hardness values

Means with different superscript letters within the same horizontal row are significantly different

# DISCUSSION

Recently, several manufacturers have introduced different lithium disilicate glass ceramic systems into the market, yet data regarding their microstructure and mechanical performance such as flexural strength and hardness is still limited (17). Among those relatively new lithium disilicate ceramic systems is LiSi Press which the authors chose for this study.

Ohashi K et al (17) have proved through their study comparing some mechanical and physical properties of LiSi to other lithium disilicate ceramics that LiSi has better physical properties, chemical stability, and higher flexural strength. They explained this through its characteristic microstructure where it showed fine lithium disilicate crystals  $(1.0-1.5 \,\mu\text{m})$ that were densely distributed. On the other hand,

the IPS e.max (Ivoclar Vivadent, Germany) specimens used in that study displayed much larger lithium disilicate crystals  $(1.0-4.0\mu m)$ . They suggested that further studies are needed for this ceramic system, consequently, that study aimed at testing the effect of repeated heat pressings on the biaxial flexural strength and hardness of GC Initial LiSi Press that is considered a relatively new lithium disilicate ceramic system <sup>(17)</sup>.

The null hypothesis was partially rejected since repeated heat pressing appeared to have a significant effect on biaxial flexural strength, but no significant effect on hardness.

In our study, we noticed an increase, yet a non-significant one, in the flexural strength of the LiSi discs when subjected to the first re-pressing. However, the value of flexural strength dropped significantly with the 2<sup>nd</sup> repressing. Such results agree with Eletreby A. and Ghanem L. <sup>(18)</sup> who showed that lithium disilicate discs displayed an increase in flexural strength when subjected to repressing only once. Additionally, Gorman C et al.<sup>(12)</sup> in a different study showed a similar pattern to the results obtained in our study where flexural strength increased non-significantly when subjected to the 1<sup>st</sup> repressing, yet the 2<sup>nd</sup> repressing caused a reduction in flexural strength values.

However, different studies in literature revealed a decrease in flexural strength of the lithium disilicate ceramic when subjected to repeated heat pressings. Albakry M et al. <sup>(16)</sup> noticed a decrease in flexural strength values after repeated heat pressings, additionally; they stated that scans obtained from scanning electron microscope showed that lithium disilicate crystals of the material subjected to repressing appeared to be larger than those of the pressed material with crystal length approximately 7.5 to 8.5  $\mu$ m. They explained this larger crystals size through the Ostwald ripening phenomenon <sup>(19)</sup>; a behavior happening when the microcrystals coarsen and liberate excess surface energy resulting

from the solubility of small particles happening as a result of the phase transition between lithium metasilicate and lithium disilicate <sup>(20,21)</sup>.

Their results and observations were later supported by Tang X et al. <sup>(13)</sup> 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 <sup>(20-22)</sup>. Moreover, SEM analysis showed that the densely packed and multidirectionally oriented interlocking multiple needlelike crystals seen in the glass matrix after the first heat pressing changed in microstructure after repeated pressings. They noticed that the crystals became wider, larger, and scantier in distribution <sup>(13)</sup>.

On a different scale, the effect that the repeated heat pressings had on the material's hardness was also interesting to investigate since it affects the success of the material clinically <sup>(23)</sup>. It has been stated that low surface hardness may cause a material to deteriorate easily, consequently causing fatigue to the material, and finally failure <sup>(24)</sup>.

When having a look at hardness values, it was clear that repeated heat pressing was associated with a decrease in Vicker's hardness values although this decrease was not significant. Our findings are in agreement with Gorman C et al. <sup>(12)</sup> and Tang X et al. <sup>(13)</sup>. Tang X et al <sup>(13)</sup> assumed that the drop in Vicker's hardness values could be associated with the decreased density and high porosity they noticed after repressings. They based their assumption on previous ceramic-related studies that confirmed that when the porosity decreases and the density increases for the ceramic bulk, its hardness values increase <sup>(25,26)</sup>.

In summary, our research has shown that multiple repressing would affect the material's strength without significant effect on its hardness; an observation that does not favor repressing the leftovers of lithium disilicate ingots more than once. However, more laboratory and clinical investigations are needed to determine the effect of multiple repressings on optical properties as well as the effect of thermal cycling together with repressing on both the mechanical and optical behavior of the material.

### CONCLUSIONS

The optimum properties for LiSi Press are probably obtained with the first pressing. However, multiple heat repressing could significantly affect the strength of LiSi Press, but with no noticeable effect on surface hardness.

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