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EFFECT OF HEAT TEMPERING ON THE BIAXIAL FLEXURAL STRENGTH OF FOUR HEAT PRESSED GLASS CERAMICS (AN IN VITRO STUDY)

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ABSTRACT

Objectives: The aim of this study was to evaluate the effect of thermal tempering on the biaxial flexural strength of four types of heat pressed Lithium disilicate glass ceramics.

Methods: Sixty heat pressed glass ceramic discs were fabricated (diameter = 15 mm; thickness = 1mm) and allocated among four groups (n = 15): IPS e.max Press (E), GC initial LiSi Press (L), Celtra Press (C) and VITA Ambria (A). Each group was subdivided into three subgroups (n=5) according to suggested thermal tempering temperature: NO thermal tempering (T0), thermal tempering at a temperature 9% below the recommended pressing temperature (T1) and thermal tempering at a temperature 5% below the recommended pressing temperature (T2). Biaxial flexural strength was measured. Scanning electron microscope (SEM) was performed for microstructural characterization. To determine the crystalline phases of Lithium disilicates glass-ceramics, X-ray diffraction (XRD) was used. Statistical analysis was then performed.

Results: IPS e.max Press showed the highest mean strength. There was no difference in strength between Celtra Press and VITA Ambria; both showed the lowest mean strength values. Regardless of ceramic type 5% below pressing temperature showed the highest mean strength and No thermal tempering showed lowest mean strength.

Conclusion: Regarding flexural strength; heat tempering at 5% below the pressing temperature of IPS Emax press showed the highest values. Vita Ambria without heat tempering showed the lowest values.

KEYWORDS: Lithium disilicate, Heat tempering, glass ceramics, Biaxial flexural strength, heat pressed

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INTRODUCTION

Recent advances in lithium disilicate glass ceramics lead to increase in flexural strength, fracture toughness, aesthetics and chemical resistance. These materials have been used to fabricate single anterior, posterior and three units fixed partial denture up-to the second premolar as a monolithic or layered restoration[1]. Heat pressing became an effective and common technique to fabricate glass ceramic restorations, as they produced a glass ceramic with an increased flexural strength, excellent marginal fit and decreased porosity [2–4].

The era of heat pressed glass ceramics started with IPS Empress, which has leucite (SiO2, Al2O3 and 4K2O) crystals as the main crystalline phase and a flexural strength of 185 MPa, followed by IPS Empress 2, which has the lithium disilicate (Li2O2SiO2) crystals as the main crystalline phase and a 360 MPa flexural strength[1]. IPS Emax press has replaced IPS Empress 2, which contains 70% lithium disilicate crystals in a glass matrix and a 450 MPa flexure strength[5]. In 2018, GC Initial LiSi Press was introduced. It's a high-strength lithium disilicate ingot fabricated using HDM (High Density Micronization) technology and has a 500 MPa flexural strength. It combines strength and aesthetics to the highest degree[6]. Recently, Celtra press and Vita Ambria were introduced as hybrid zirconia reinforced lithium disilicate ceramics to combine high optical properties with improved mechanical properties by adding 8-12% zirconia crystals into the glass matrix of lithium silicate/ disilicate glass ceramics[7,8].

It was reported that flexural strength of IPS Empress increased during heat-pressing procedure and further increased with the subsequent heat treatments required for a final restoration, the subsequent heat treatments are very important to obtain the maximum strength of the glass ceramics[9]. Regarding the need to improve the mechanical properties of lithium disilicate restorations to be used in high stresses areas, it could be strengthened by heat tempering which increases the flexural strength by increasing the size of crystals and the microstructure becomes more interlocking[10,11].

Differences in mechanical properties for materials with similar chemical compositions are primarily due to their microstructure. Flexural strength is one of the most important properties to assess the strength of brittle materials and the limitation of the dental ceramics [1,4,8,12]. Flexural strength of glass ceramics depends on many factors like the type of crystalline phase, grain size, additives, microstructure, glass matrix, way of manufacturing and chemical composition[1]. Studies showed that heat treatment of glass ceramics increased the dimensions of the crystals; length and width .The microstructure became more interlocking and the biaxial flexural strength increased [8,13].

The present study was performed to evaluate the properties of four heat pressed lithium silicate/ disilicate glass ceramics; IPS Emax press, Initial LiSi, Celtra press and Vita Ambria before heat tempering and after heat tempering at a temperature below their pressing temperature by 5% and 9%. The null hypothesis of this study was that heat tempering would not affect the flexural strength of the tested Lithium Silicate/disilicate materials.

MATERIALS AND METHODS

1. Specimen preparation

Four heat-pressed lithium silicate/disilicate glass ceramics were used in the study; IPS Emax Press(Ivoclar Vivadent AG, Schaan/Liechtenstein), Initial LiSi Press(GC, Tokyo, Japan), Celtra Press(Dentsply Sirona, Hanau-Wolfgang, Germany) and Vita Ambria(Vita Zahnfabrik, Bad Säckingen, Germany). Sixty heat pressed glass ceramic discs were prepared according to the ISO 6872 specifications for testing ceramic materials. Discs of 15mm diameter, 1 mm thickness were fabricated using heat pressed technique. The discs were divided into four groups(n=15);(E,L,C andA) according to type

of heat pressed glass ceramic. All discs were heat pressed and finished according to the manufacture's recommendation. Each group was subdivided into three subgroups (n=5); according to suggested thermal tempering temperature; Subgroup (T0): Samples with no thermal tempering, Subgroup (T1): Samples were subjected to thermal tempering at a temperature 9% below the recommended pressing temperature and Subgroup (T2): Samples were subjected to thermal tempering at a temperature 5% below the recommended pressing temperature. (Table-1).

2. Biaxial flexural strength testing

All discs of each material were tested for biaxial flexural strength (MPa) according to the guidelines of ISO standard 6872 for dental ceramics. The test was done using piston-on-ball technique in a universal testing machine* together with Instron Bluehill universal software, 3 hardened steel balls with a diameter of 3.2 mm forming an equilateral triangle 60 degree, resting on a custom-made metallic platform with a diameter of 10 mm, was used for supporting the discs. Each disc was placed centrally on the steal balls. The load was applied from above the center of the discs by a piston 1.4 mm diameter and 0.5 mm/min crosshead speed until fracture occurred. All the discs were fractured into two pieces. The biaxial flexural strength was calculated using the following equation:

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

where, σ is the maximum center tensile stress (in MPa)

P is the total load at fracture (in N)

d is the specimen thickness at the fracture origin (mm)

$X = (1 + v) \ln (r2 /r3) 2 + [(1 - v)/2] (r2 /r3) 2$

$Y = (1 + v) [1 + \ln (r1 /r3) 2] + (1 - v) (r1 /r3) 2$

In which,

v is Poisson's ratio (0.25) the standard value for conventional ceramics

r1 is the radius of the support circle (mm)

r2 is the radius of the loaded area (mm)

r3 is the radius of the specimen (mm)

The results for the specimens in MPa were tabulated

3. Scanning electron microscope (SEM):

In preparation for SEM, specimens were cleaned, etched with 9.8% Hydrofluoric acid for 90 seconds and then dropped immediately in an ultrasonic cleaner for 15 seconds, then dried and sputter coated with gold. SEM (Quanta 250 FEG) was carried out to examine the microstructure at magnification of 15000x. It also served as a means of assessing a change in the grain width or length within the specimens after Heat treatment.

TABLE (1) Showing pressing and heat tempering temperatures

Tempering temp. / Material	Pressing temperature (T0)	9% below the recommended pressing temperature. (T1)	5% below the recommended pressing temperature. (T2)
IPS E.max Press (E)	915 C	832 C	869 C
GC Initial LiSi (L)	900 C	819 C	855 C
Celtra Press (C)	860 C	782 C	817 C
Vita Ambria (A)	880 C	800 C	836 C

* Instron-3345 universal testing machine.

4. Xray diffraction (XRD)

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 5-second step interval.

Statistical Analysis

Numerical data were explored for normality by checking the distribution of data and using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). All data showed normal (parametric) distribution. Numerical data were presented as mean and standard deviation (SD) values. Two-way ANOVA test was used to study the effect of ceramic type, tempering and their interactions on biaxial flexural strength. Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM SPSS Statistics for Windows, Version 23.0. Armonk, NY: IBM Corp.

RESULTS

1. Biaxial flexural strength

With no thermal tempering; there was a statistically significant difference between ceramic types (*P*-value <0.001, Effect size = 0.422). Pair-wise comparisons between ceramic types revealed that there was no statistically significant difference between (E) and (L); both showed the statistically significantly highest mean biaxial flexural strength values. (A) showed statistically significantly lower mean value. (C) showed the statistically significantly lowest mean biaxial flexural strength.

With 5% below pressing temperature; there was a statistically significant difference between ceramic types (*P*-value <0.001, Effect size = 0.444). Pair-wise comparisons between ceramic types revealed that there was no statistically significant difference between (E) and (L); both showed the statistically significantly highest mean biaxial flexural strength values. There was no statistically significant difference between (C) and (A); both showed the statistically significantly lowest mean biaxial flexural strength values.

With 9% below pressing temperature; there was a statistically significant difference between ceramic types (*P*-value = 0.009, Effect size = 0.214). Pair-wise comparisons between ceramic types revealed that (E) showed the statistically significantly highest mean biaxial flexural strength. There was no statistically significant difference between (L), (C) and (A); all showed the statistically significantly lowest mean biaxial flexural strength values.

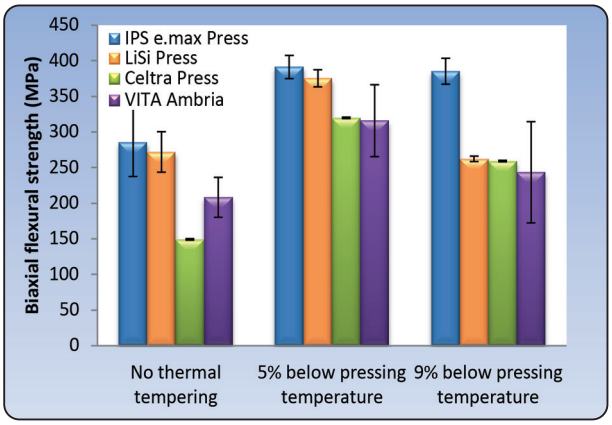

Fig. (1) Bar chart representing mean and standard deviation values for biaxial flexural strength without and with heat tempering

2. Microstructure

The (ET0) specimens exhibit typically multilayered rod-shaped crystals. The crystals of (ET0) group is aligned parallel to the pressing direction. The average length of the crystals is 3 um and the average width of the crystals is 680 nm (fig 2). whereas with (ET1) group, the length increased to 4.5 um and the width increased to 700 nm (fig 3). With (ET2) group, the crystal length increased to 4 um and the width increased to 870 nm (fig 4)

The microstructure of (LT0) group is significantly different compared to the (ET0) group. It showed multilayered platelet-shaped crystals embedded in the glass matrix forming an interlocking microstructure. Compared to (ET0), the crystals weren't aligned parallel to the pressing direction. The average length of the crystals in the (LT0) group is 2 um and the width is 600 nm (fig 5). The (LT1) group showed 2.5 um length of the crystals and 630 nm width (fig 6). The (LT2) group showed 2.4 um length of the crystals and 985 nm width (fig 7).

The microstructure of (CT0) group showed interlocking microstructure with the average crystals length of 1.8 um and 500 nm in width (fig 8). The (CT1) group showed decrease in the crystal's length to 1.3 um and the width increased to 600 nm (fig 9). The (CT2) group showed increase in the crystal's length to 2 um and a significant increase in width to 770 nm (fig 10).

The microstructure of heat pressed (AT0) showed a needle like crystals with average length of 3 um and a 380 nm width (fig 11), The (AT2) group showed solidification and shrinkage in size of the lithium disilicate crystals (fig 13) that made it difficult to perform reliably accurate measurements of grain sizes. The (AT1) group showed increase crystals length and width to be 4.5 um and a 530 nm respectively (fig 12)

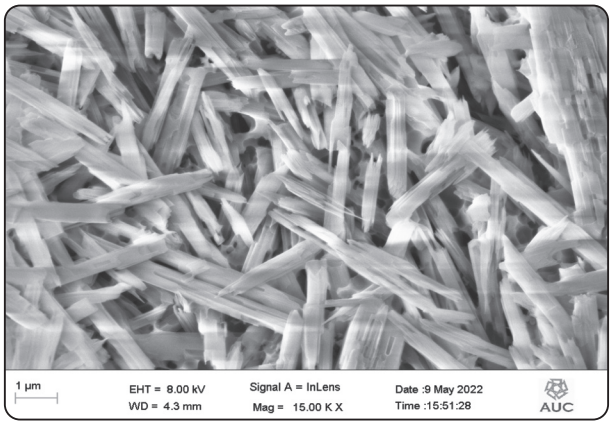

Fig. (2) IPS Emax press without tempering

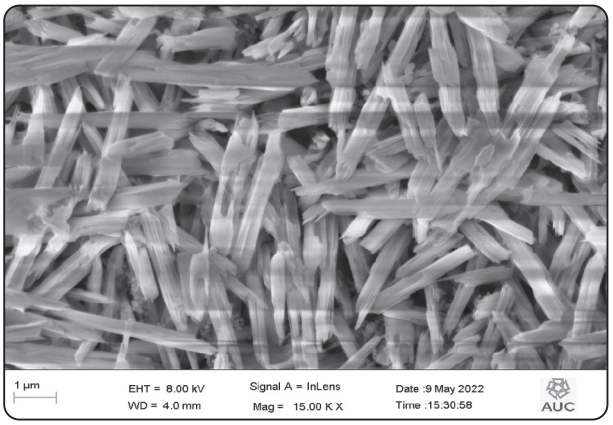

Fig. (3) Showing SEM of IPS Emax after the first heat tempering 9%

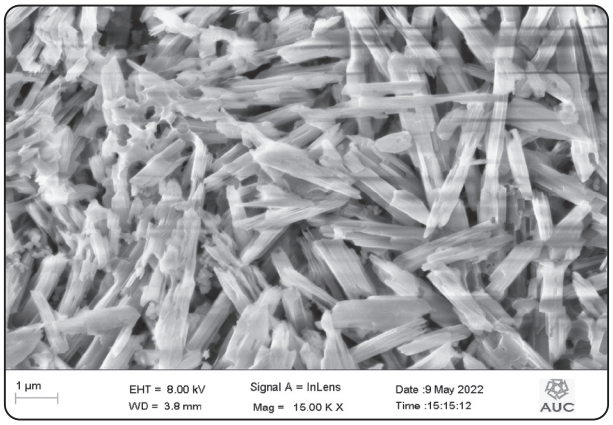

Fig. (4) Showing SEM of IPS Emax after the first heat tempering 5%

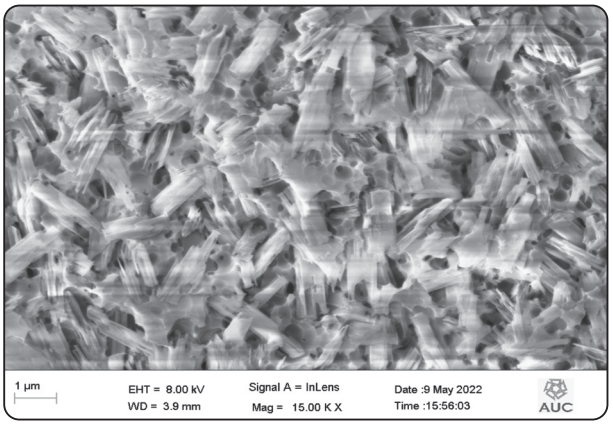

Fig. (5) Showing SEM of LiSi press without tempering

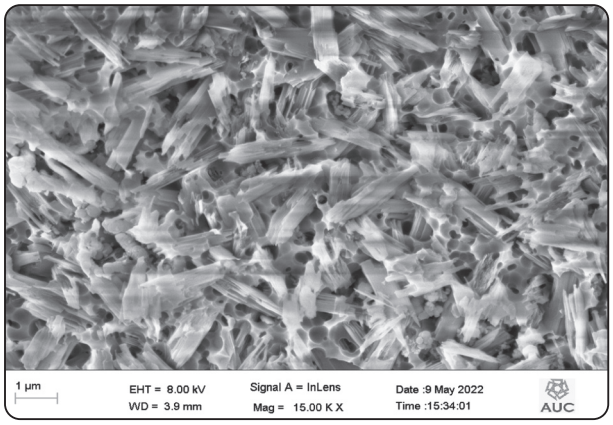

Fig. (6) Showing SEM of Initial LiSi after the first heat temperong 9%

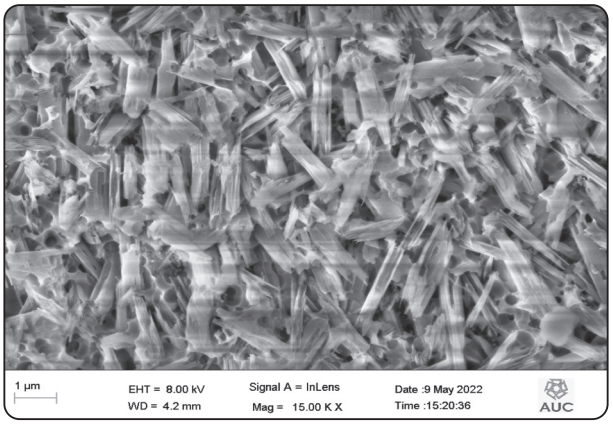

Fig. (7) Showing SEM of Initial LiSi after the first heat temperong 5%

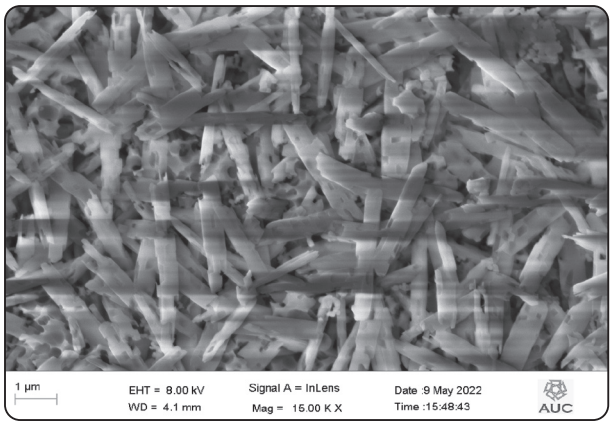

Fig. (8) Showing SEM of Celtra press without tempering

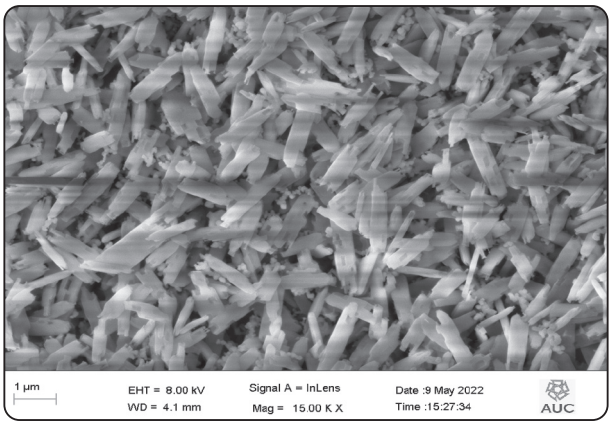

Fig. (9) Showing SEM of Celtra press after the first heat temperong 9%

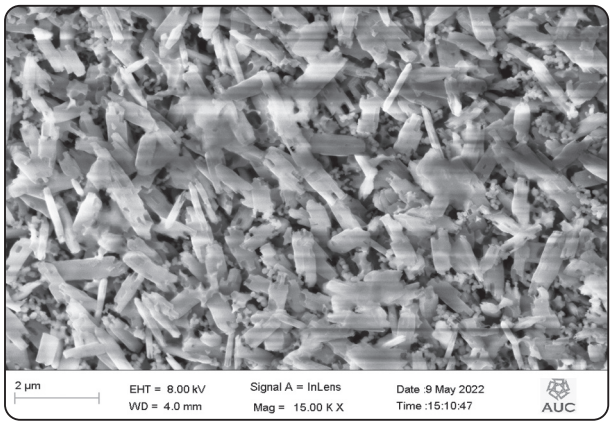

Fig. (10) Showing SEM of Celtra press after the first heat temperong 5%

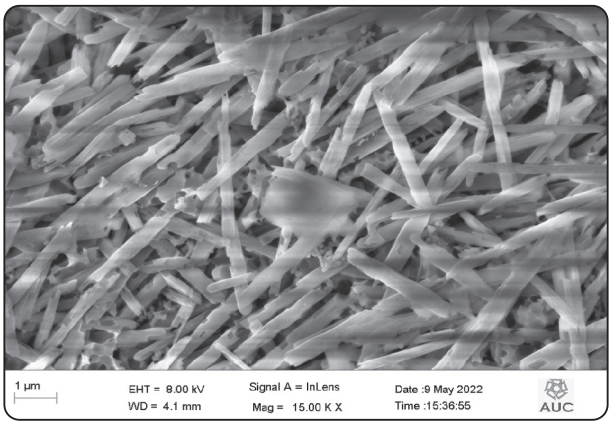

Fig. (11) Showing SEM of Vita Ambria without tempering

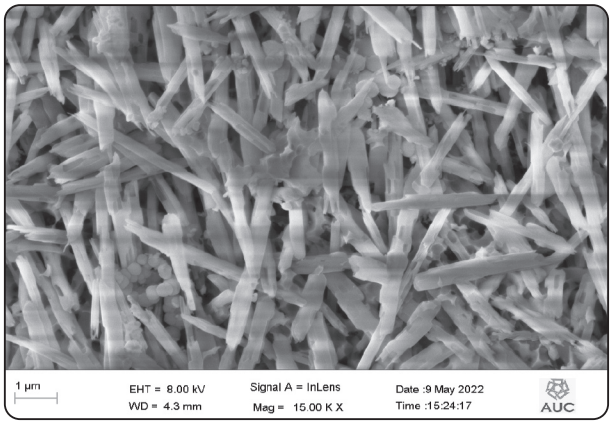

Fig. (12) Showing SEM of Vita Ambria after the first heat temperong 9%

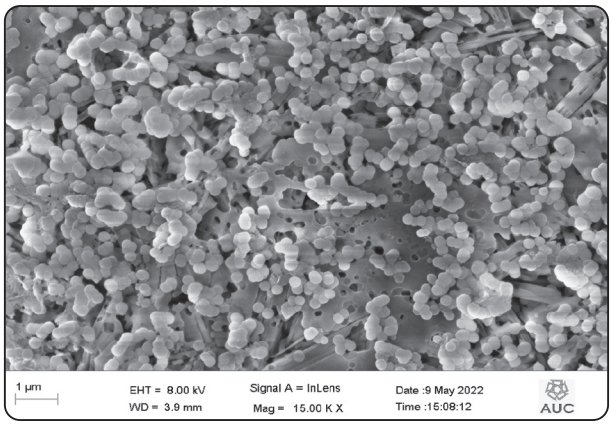

Fig. (13) Showing SEM of Vita Ambria after the first heat temperong 5%

3.3. XRD

The main phase of all pressed lithium reinforced glass-ceramics groups was lithium disilicate (LS2). Except for the (CT0), (CT1) and (CT2) group, the main crystalline phase was Lithium Silicate. The XRD data of (ET1), (ET2), (LT1), (LT2), (AT1) and (AT2) groups showed that, after heat tempering, the main crystalline phase did not change. However lower intensities of the main peaks of the LS2 phase were observed for the (ET1) group compared to (ET0) and (ET2). Same was observed for Ambria groups; the crystalline phase of (AT1) showed lower intensities than that of (AT0) and (AT2) groups. The crystalline phase of (LT1) and (LT2) groups showed higher intensities than that of (LT0) group. The crystalline phase of (CT1) and (CT2) groups showed lower intensities than that of (CT0) group.

DISCUSSION

The Lithium Disilicate glass ceramics have high translucency and optical properties that made it highly recommended for anterior and posterior single restorations, on the other hand it has limited clinical indications when it comes to high stress. The introduction of hybrid ceramic materials as Celtra press by means of the addition of zirconia particles as second phase crystalline particles to lithium silicate glass ceramics, lead to a new generation of ceramics which allegedly combine glass-ceramic esthetic performance and Zirconia improved mechanical properties[7,14]. Celtra manufacturer recommended that Celtra press requires a 'Power cycle' before veneering or glazing that will repair small flaws in the glass matrix brought on by sandblasting which is done during the divesting of the investment or polishing and boost the flexural strength [7].

A heat pressed zirconia reinforced lithium disilicate glass ceramic material (VITA Ambria) has been recently introduced. The manufacturer (VITA Zahnfabrik) claims that subjecting this material to thermal tempering (at 800ºC), would improve its flexural strength form 400 MPa to 550 MPa. The effect of thermal tempering on the biaxial flexural strength of this material is still to be investigated. Also, the exact temperatures for tempering of each material and whether this thermal tempering is effective with other types of heat pressed lithium disilicate glass ceramics is still unknown[15].

In this study 60 discs with 15 mm diameter were used .1 mm thickness was chosen to simulate the crown thickness. The discs were fabricated by heat pressing technique using Ivoclar Programat EP3010 furnace which involves the use of a special porcelain furnace with a pneumatic ram, which presses the ceramic material into the mold at high temperatures under vacuum. The benefits of heat-pressed ceramics as opposed to the more traditional method

of sintering are, net-shape processing, decreased porosity and increased flexural strength. [16,17].

Heat tempering is a well-known method for glass ceramics strengthening. Studies showed that heat tempering of Lithium disilicate increase crystals size and the crystals microstructure becomes more interlocking and highly packed so the crack propagation follows more tortuous path[18–20]. Testing the mechanical properties of glass ceramics could be done in-vivo or in-vitro. However, in-vitro methods are more reliable and faster.

Biaxial flexural strength is one of the most important properties of dental ceramics to be tested[21]. There are many methods for flexural strength testing but in this study three points biaxial biaxial flexural strength test was done as it gives more reliable results and more recommended than four points bending tests and uniaxial flexural strength tests[21-23].

Heat tempering at 9% below pressing temperature was selected according to Vita ZahnFabrik which claimed that subjecting the material to thermal tempering at 800C improves the biaxial flexural strength[15] and heat tempering at 5% below pressing temperature was selected as it is near to the repressing temperature.

Biaxial flexural strength:

The null hypothesis that heat tempering would not affect the flexural strength of the tested Lithium Silicate/disilicate materials was partially accepted. **After pressing and without heat tempering,** (ETO) group (285 MPa) showed the highest biaxial flexural strength followed by (LTO) group (272 MPa), This result could be explained by SEM that showed high interlocking microstructure with rod shaped crystals that have an average length 3 um and average width 680 nm for (ETO) group. Its microstructure is aligned parallel to the pressing direction. Unlike (LTO) group that showed multilayered platelet shaped high interlocking crystals which have 2 um length and 600 nm width. These crystals weren't aligned parallel to the pressing direction. These results were also supported by XRD that showed higher intensities of lithium disilicate crystalline phase for (ETO) group compared to (LTO) group.

The biaxial flexural strength values of (ATO) and (CTO) groups were lower than (ETO) and (LTO) groups. The biaxial flexural strength of (ATO) was (208 MPa) and (CTO) was (149 MPa) which showed the lowest biaxial flexural strength values. These results can be explained by SEM of (ATO) and (CTO) groups that showed relatively smaller crystal width of 380 nm and 500 nm respectively that are aligned randomly in the glass matrix leading to high porosity microstructure that enhances the crack propagation.

The results of the present study were in agreement with **Hallmann et al (2018)**[8] who found that LiSi press and IPS Emax press have higher biaxial flexural strength than Celtra press. On the other hand **Ohashi et al(2013)**[24] found that the biaxial flexural strength of LiSi was higher than IPS Emax press.; this was explained by the densely distributed lithium disilicate crystals of LiSi group which can offer the resistance against crack propagation and inhibit crack growth.

With 5% below pressing temperature all tested ceramics showed statistically significantly higher mean biaxial flexural strength than with no thermal tempering. These results can be explained by the changes that showed in SEM of different ceramic materials. These results coincides with **El-Etreby et al(2017)**[13]**,** who concluded that the biaxial flexural strength of heat pressed lithium disilicate glass ceramics increased after heat tempering and this result was explained by the significant increase of crystal size after heat tempering.

With 5% below pressing temperature (ET2) group showed the highest biaxial flexural strength as the lithium disilicate crystals have the highest diameter values with increased microstructure's interlocking and packing. On the other hand, (CT2) and (AT2) groups showed the lowest biaxial flexural

strength values due to the presence of ZrO2 in the glass matrix which increases the viscosity of the heat pressed ceramic and decreases the crystal growth of lithium metasilicate and lithium disilicate during heat tempering. **Hallmann et al(2018)**[8] **,** found that Celtra press have the lowest biaxial flexural strength values after heat tempering at 860C when compared to IPS Emax Press and Initial LiSi press. On the other hand, **Albakry et al(2004)**[1] found that there was no significant effect on biaxial flexural strength after heat tempering of Lithium disilicate glass ceramics.

With 9% below pressing temperature the (ET1) group showed the highest significantly biaxial flexural strength, which increased from 285 MPa to 385 MPa, and (AT1, CT1) groups showed the lowest biaxial flexural strength, that increased from 208 MPa to 243 MPa and from 149 MPa to 259 MPa respectively. However, (LT1) group showed a decrease in biaxial flexural strength from 272 MPa to 262 MPa. These results could be explained by SEM; In (ET1) group the lithium disilicate crystals incearsed in length from 3 um to 4.5 um and the width increased from 680 nm to 700 nm. The microstructure of (LT1) group showed more porosity than the control group. For (AT1 and CT1) groups their microstructure showed shrinkage of the crystals accompanied with a solidification of some crystals, that could be explained by the presence of ZrO2 in the glass matrix which increases the viscosity and decreased the crystals growth during heat treatment. The microstructure became less interlocking.

These results coincides with **Sun et al (2021)** [25] who found that the flexural strength is increased when Lithium disilicate (IPS Emax press) glass ceramics were subjected to heat tempering at 820C to reach 325+/- 47 MPa, this increase in flexural strength was explained with change in the crystals morphology from spherical to rod shaped. Also **Oh et al (2000)**[11] concluded that heat tempering of IPS Empress 2 increased its flexural strength to 387 MPa, this was explained by SEM which showed highly packed, interlocking microstructure of many lithium disilicate crystals in the glass matrix**.** On the other hand **Tang et al(2014)**[25] concluded that flexural strength of IPS Emax press decreased when subjected to heat tempering and explained that by the increase in porosity after heat tempering.

CONCLUSIONS

Within the limitations of the present study, the following conclusions were drawn:

1. The IPS Emax press and LiSi press materials had the highest significantly mean biaxial flexural strength than Celtra press and Vita Ambria materials.
2. Heat tempering influenced the microstructure of lithium reinforced glass-ceramic materials and caused statistically significant change of BFS.
3. Heat tempering with 5% below pressing temperature increased the biaxial flexural strength of all tested glass ceramic materials.

RECOMMENDATIONS

It's recommended to use the heat tempering strengthening method to increase the flexural strength of glass ceramic restorations used in high stresses situations.

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