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THE EFFECT OF THERMAL TEMPERING ON FRACTURE RESISTANCE AND SURFACE CHARACTERIZATION OF LITHIUM DISILICATE AND ZIRCONIA REINFORCED LITHIUM SILICATE CROWNS (IN VITRO STUDY)

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ABSTRACT

Statement of problem: The manufacturer of newly introduced pressable ceramic claimed that the strength can be increased by subjecting the material to a thermal tempering cycle at 9% below pressing temperature after its pressing. However, the effect of this thermal tempering protocol on the fracture resistance of different heat-pressed glass ceramics is still not known.

Purpose: This in-vitro study aimed to investigate the effect of thermal tempering on the fracture resistance and microstructural features of two heat-pressed glass ceramics.

Materials and methods: A total of 28 glass-ceramic crowns were assigned into two groups according to the ceramic type (n =14): Group (A): Lithium Disilicate glass-ceramic crowns and Group (B): Zirconia Reinforced Lithium Silicate glass ceramic crowns. Crowns of each group were divided into two equal subgroups (n=7) according to the subsequent thermal tempering temperature: Subgroup (T0): crowns were not subjected to thermal tempering and subgroup (T1): crowns were subjected to a temperature at 9% below pressing temperature. Fracture resistance of all samples were tested. A two-way ANOVA test was used to study the effect of ceramic type, thermal tempering and their interactions on fracture resistance.

Results: When no thermal tempering was applied, IPS E.max press showed statistically significantly higher mean fracture resistance value than Celtra press (1819.04 N and 1286.4N respectively). Thermal tempered Celtra press crowns showed the highest fracture resistance value (1951.7 N).

Conclusions: Lithium disilicate crowns demonstrated higher fracture resistance than Zirconia-Reinforced Lithium Silicate (ZLS) crowns without tempering. Thermal tempered ZLS crowns demonstrated increase in fracture resistance.

KEYWORDS: Pressable ceramics, Lithium disilicate, Lithium silicate, Fracture resistance, Thermal tempering.

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INTRODUCTION

In the 1960s, John Maclean introduced all ceramic dental restorations, which marked a substantial transformation in the dental field[1]. Recently, novel ceramic materials have been developed in an effort to combine polycrystalline ceramics mechanical properties and glass ceramics' superior esthetics[2]. IPS E.max was launched in 2007 as an updated generation of lithium disilicate ceramics, with improved physical properties and excellent translucency using different firing processes[3]. Its microstructure contains 3 to 6 μm in length needle-shaped lithium disilicate crystals, approx. 70wt%, embedded in a glassy matrix. It has a flexural strength of 400 Mpa[4,5].

Zirconia-reinforced lithium silicate (ZLS) ceramic: Celtra press, is an alternative strategy to enhance translucency and strength. It is composed of a glassy matrix, a homogeneous crystalline structure made of lithium silicate crystals and reinforced with tetragonal zirconia fillers (about 10% by weight) that results in higher strength values than lithium disilicate glass ceramics[6]. ZLS ceramic is a promising restorative material that exhibits a unique behavior when subjected to aging[7]. Their physical, chemical, and mechanical properties are adjustable by changing the composition and heat treatment process[8]. It showed a lower brittleness index providing superior machinability with high edge strength and better surface finish than lithium disilicate glass-ceramic[9].

There are different factors affecting the fracture resistance of lithium disilicate crowns such as composition of ceramic material, ceramic microstructure, crown thickness and thermal tempering[10-13]. The heat-pressing technique has become a common technique to produce glass-ceramic dental restorations. Heat-pressed glass-ceramic restorations have better fitting accuracy, marginal edge quality, low porosity, as well as high mechanical properties when compared to computer aided design/computer aided manufacture (CAD/CAM) milled materials[14]. Due to the limitations in ingots shades, the heat-pressed glass ceramic restorations may need to undergo additional firing cycles to improve the esthetic outcome, either for glazing, characteristic dying or even surface veneering. The consequent heat treatments of glass ceramic restoration were associated with changes in material mechanical and optical properties, which can affect the final clinical results[15] .

The manufacturer of newly introduced ZLS ceramic claimed that the strength of the material can be increased by subjecting the material to a thermal tempering cycle at 9% below the pressing temperature after its pressing[16,17]. Thermal tempering procedure greatly influences the crystal size and its morphology[18]. The growth of the grain size indicates that the crystallization process continues during the thermal tempering procedure and more crystals are precipitated leading to an increase in the fracture resistance [5].

This in-vitro study aimed to investigate the effect of thermal tempering and ceramic type on the fracture resistance and microstructural features of two types of heat pressed glass ceramics. The null hypothesis of this study was that neither the type of the heat pressed ceramic nor the thermal tempering temperature would affect the fracture resistance of the ceramic material tested.

MATERIALS AND METHODS

The materials used in this study are presented in Table (1).

TABLE (1) Materials used in this study

Material	Trade name	Manufacturer	Description
Lithium disilicate glass ceramic	IPS E.max Press	Ivoclar vivadent, Schaan, Liechtenstein.	Lithium disilicate crystals (approx. 70%), Li2Si205, embedded in a glassy matrix. Top of Form Bottom of Form Top of Form Bottom of Form
Zirconia reinforced lithium silicate glass ceramic	Celtra Press	Sirona Dentsply, Milford, DE, USA.	1.5 um plus nano-scale lithium phosphate, Li20 and Si03 and 10% zirconia (Zr02), which is completely dissolved in the glass phase

A computerized numerical control lathe-cut milling machine (C.N.C premium 4820, imes-icore Eiterfeld, Germany) was used to prepare acrylic lower first molar following the manufacturer's recommendation to receive a posterior crown. Using a tapered stone with round end, occlusal reduction 1.5 mm and axial reduction 1mm were obtained. A 1mm supragingival circumferential heavy chamfer finish line was prepared. Silicon duplicating material (Replisil 22N, dentecon, Germany) was used for making seven impressions of the master model. After setting, the molds were poured with chemical cured epoxy resin (Chemapoxy 150, MBC, Egypt) and left for 24 hours for complete curing. This step was repeated 4 times to produce 28 epoxy resin dies.

For standardization of the crown's anatomy, contour, and thickness, the preparation was scanned by Identica blue scanner (MEDIT corp., Seoul, Korea), then the data was transferred to an Exocad computer software version 2017 (Exocad GmbH, Darmstadt, Germany) to design the crowns. A 5-axis milling machine (VHF, CAM 5-S1, Germany) was used for milling of wax patterns from wax blocks (YAMAHACHI, Japan). A total of 28 wax patterns were milled and tried gently over their corresponding dies to check their accuracy. All wax patterns were sprued and invested following manufacturer's instructions with IPS PressVEST (Ivoclar Vivadent, schaan, Liechtenstein). The investment was left to set for 30 minutes, and then wax elimination was performed with wax burn out furnace (Ney, US Dental Depost, USA) according to the manufacturer's recommendation. Half of the patterns (n=14) were pressed using IPS e.max Press ingots (Group A) while the other half (n=14) was pressed using Zirconia Reinforced Lithium Silicate glass ceramic (Celtra Press) ingots (Group B). All crowns were pressed following manufacturer's recommendations in a heat press furnace (EP 3000, Ivoclar Vivadent, schaan, Liechtenstein). After pressing, half of the crowns in each group (n=7) were left without any further heat treatment (Subgroup T0), while the other half (n=7) were subjected to thermal tempering 9% below the pressing temperature (Subgroup T1). The pressing temperature and the calculated thermal tempering temperature for each ceramic material are presented in Table (2).

TABLE (2) Comparison between pressing temperature and tempering temperature of each ceramic material

Material	Maximum pressing temperature without tempering (°C)	Calculated tempering temperature with 9% below maximum pressing temperature (°C)
IPS E.max Press	917	834
Celtra Press	865	787

Each crown was checked over it's corresponding dies for seating and marginal accuracy using a dental probe and magnifying loops (2.5X). Any defective or unseated crown was discarded. The internal surface of each crown was etched with hydrofluoric acid gel 9.5% (Porcelain Etchant, BISCO, USA) for 20 seconds, rinsed with water, and then dried with oil-free moisture-free air. Silane coupling agent (Porcelain Primer, BISCO, USA) was applied to the internal surface of each crown for 1 minute, then air-dried for 5 seconds. All crowns were cemented using dual cure self-adhesive resin cement (Duo-Link, BISCO ,USA). Using gentle finger pressure, all crowns were seated on their corresponding dies. An axial load of 5 kg was applied using loading device for 10 minutes. Light curing of the margins for 3 seconds using SDI plus light-curing unit (SDI, Australia) was applied. Then, excess luting material was removed. The luting material was light cured for 20 seconds per surface.

A universal testing machine with a load cell of 5kg was used and data were recorded using computer software (Instron 3345, Instron, USA). Fracture resistance test was done by compressive mode of load applied to the center of the occlusal surface, in such way the load applicator tip only touches the buccal incline of the lingual cusp and lingual incline of the buccal cusp using a metallic rod with spherical tip (5.6 mm diameter) with a tin foil sheet in-between to achieve homogenous stress distribution and minimize the transmission of localized force peaks. Failure load was manifested by an audible crack and confirmed by a sharp drop at load-deflection curve recorded using computer software. The load required to fracture was finally recorded in Newton and the mode of failure for each restoration was observed. Fragments of broken crowns were retrieved and further prepared for Scanning electron microscopic (SEM) analysis (Quanta 250 FEG, Australia). Each Scanning electron microscopic image was captured at magnification of 10.000x.

Statistical analysis was performed with R statistical analysis software version 4.1.3 for Windows (R Core Team, Vienna, Austria). Shapiro-Wilk's test was used to test fracture resistance data for normality. Homogeneity of variances was tested using Levene's test. Data showed parametric distribution and variance homogeneity and were analyzed using two-way ANOVA. Comparison of simple main effects was done utilizing the error term of the two-way model with p-values adjustment using Bonferroni correction. The significance level was set at $p<0.05$ within all tests. Categorical data was presented as frequency and percentage value and was analyzed using Fisher's exact test.

RESULTS

1- Fracture resistance results:

Comparison of simple main effects presented in table (3) showed that, When no thermal tempering was applied, IPS E.max press crowns showed statistically significant higher mean fracture resistance value than Celtra press crowns ($p<0.001$). While with thermal tempering 9% below pressing temperature; Celtra press crowns demonstrated statistically significant higher fracture resistance values than Emax press ($p=0.010$).

TABLE (3) Comparisons of simple main effects

Temperature	*Fracture resistance (N) (Mean±SD)*		*p-value*
	Emax Press	*Celtra Press*	
T0	1819.04±51.22	1286.40±11.50	**<0.001***
T1	1754.94±210.75	1951.72±151.52	**0.010***
p-value	**0.374**	**<0.001***	

****significant (p<0.05)***

Celtra press crowns with thermal tempering 9% below pressing temperature showed statistically significant higher mean fracture resistance value than without thermal tempering (p<0.001). While IPS E-max press showed no significant difference between tempered and non-tempered crowns (p=0.374).

Results of intergroup comparisons of failure mode presented in table (4) showed that the majority of non tempered Celtra Press crowns had cracks, while most of the samples in other groups had either chipping or partial fractures, yet the difference between tested groups was not statistically significant (p=0.741).

Table (4): Intergroup comparison of failure mode

Failure mode		*Emax Press (T0)*	*Emax Press (T1)*	*Celtra Press (T0)*	*Celtra Press (T1)*	χ^2	*p-value*
Crack	**n**	3	2	4	2	**4.95**	**0.741**
	%	42.9%	28.6%	57.1%	28.6%		
Chipping or partial fracture	**n**	4	3	3	4		
	%	57.1%	42.9%	42.9%	57.1%		
Catastrophic or fragments fracture	**n**	0	2	0	1		
	%	0.0%	28.6%	0.0%	14.3%		

2. Scanning electron microscope results:

IPS E.max press SEM image showed needle shaped elongated crystals that were highly interconnected with sharp and pointed edges forming highly interlocking microstructure as shown in Figure (1). While, Celtra press SEM image showed shorter and flatter crystals with less interlocking microstructure as shown in Figure (2).

IPS E.max press samples subjected to tempering 9% below pressing temperature showed elongated needle shaped crystals with rounded edges and highly interlocking as shown in Figure (3). Celtra press samples subjected to tempering 9% below pressing temperature: showed broader, randomly oriented and highly interlocking crystals than non-tempered Celtra press samples as shown in Figure (4).

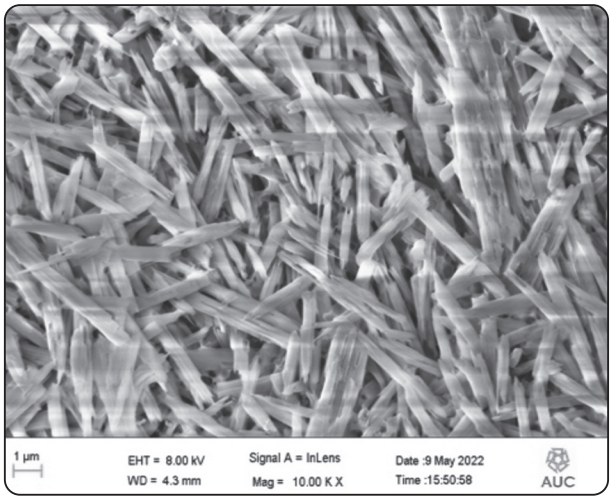

Fig. (1) SEM images (10.000x) of IPS E.max press control group specimens

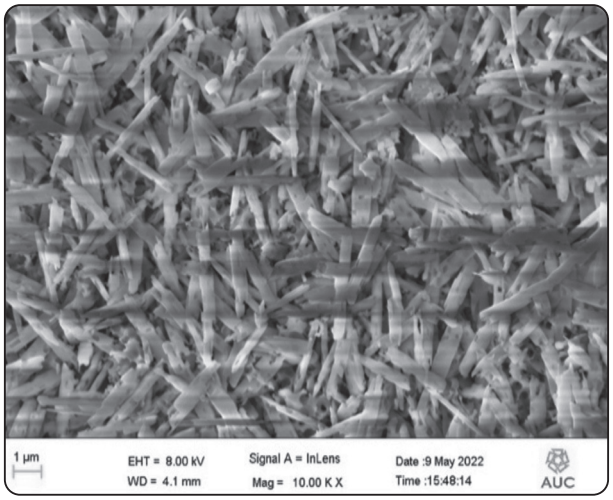

Fig. (2) SEM images (10.000x) of Celtra press control group specimens.

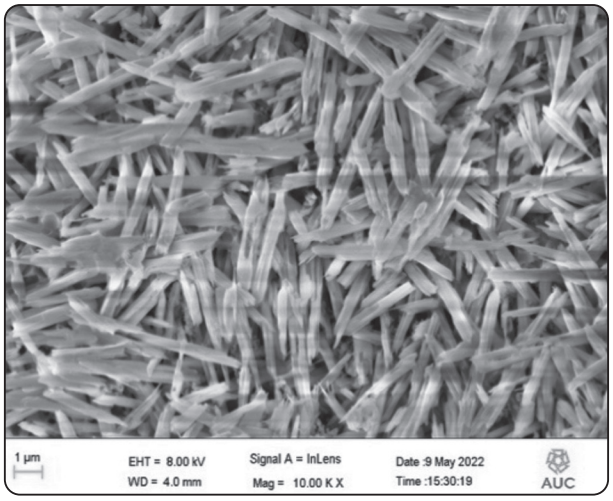

Fig. (3) SEM images (10.000x) of IPS E.max press tempering -9% specimens.

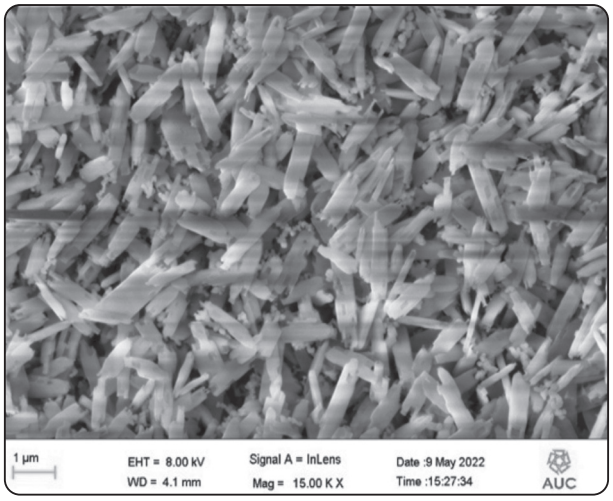

Fig. (4) SEM images (10.000x) of Celtra press tempering -9% specimens.

DISCUSSION

IPS e.max press is currently considered as a gold standard reference for comparisons in many in vitro studies[19]. Celtra press is another innovative advancement in glass ceramic materials containing 10% zirconia providing high mean flexural strength in addition to its high glass Content[20]. Some manufactures claimed that the strength of ceramic material can be increased by subjecting the material to a thermal tempering cycle at 9% below pressing temperature[16,17]. This in-vitro study aimed to investigate the effect of thermal tempering on the fracture resistance of two heat pressed ceramics and examine their microstructural features using scanning electron microscope (SEM).

The crowns in this study were cemented and tested over epoxy resin dies to obtain fracture resistance values near to those cemented on dentin. Epoxy resin has a modulus of elasticity (12.9 GPA) which is similar to the reported modulus of elasticity of human dentin (14.7 GPA)[21].

Non-tempered IPS E.max press crowns showed statistically significant higher mean fracture

resistance values than non-tempered Celtra press crowns. This finding could be attributed to the microstructural features of E.max press samples. SEM images in this study revealed elongated needle shaped crystals with sharp and pointed edges forming an interlocking microstructure that might increase the fracture resistance. Also, the higher pressing temperature of Emax press (917 °C) might allow more crystal growth and interlocking. The results are in agreement with a study by **Hakim A et al.**[22], who discussed the flexural strength of Celtra press compared to E.max press. E.max press showed higher flexural strength value than Celta press.

The lower fracture resistance values of non-tempered Celtra press crowns can be attributed to the addition of ZrO_2 nucleating agent that hindered crystal growth. Therefore, smaller lithium silicate crystalline phases were present in the pressed samples compared to ZrO_2-free glass-ceramics. This finding was supported by SEM images of this study where Celtra press showed shorter and flatter crystals with less interlocking and more inter crystal spaces. These smaller crystals adversely affected the mechanical properties of the glass-ceramic. These results were in agreement with **Apel et al.**[23] **,** who stated that the incorporation of ZrO_2 in the glass matrix did not increase the flexural strength. This was explained by the increase in viscosity due to the high ZrO_2 content in the glass- ceramic and the associated reduction in the crystal growth of lithium silicate and lithium disilicate.

For Celtra press crowns, thermal tempering 9% below pressing temperature showed significant higher mean fracture resistance than without thermal tempering. This might be attributed to the effect of thermal tempering procedure on the crystal size and morphology . These results coincide with **Albakry et al.**[24]**,** they stated that this behavior is called "Ostwald ripening" . It takes place when the microstructure coarsens and liberates surface energy excess due to the solubility of small particles. As a consequence, larger crystals grow at the expense of smaller ones. Larger-sized crystals increase the interlocking effect which lead to an increase in the fracture resistance. This finding was supported by SEM images of this study where highly interlocking and broader crystals were presented in tempered Celtra press samples resulting in the highest fracture resistance mean value 1951.7 N.

Tempered IPS E-max press showed no significant difference between tempered and non-tempered crowns. This is can be attributed to the SEM images in this study where both presented nearly same features of elongated highly interlocking crystals.

The fracture resistance results of crowns in the current study are clinically accepted within the mean reported biting force of molars 600-900 N[25]. Therefore, the tested crowns are able to withstand the maximum posterior masticatory forces and presented favorable modes of failure which were mostly cracks and chipping fractures.

The null hypothesis in the present study was rejected as both ceramic type and thermal tempering had significant effect on the crowns fracture resistance.

Within the limitations of this study, the following conclusions can be drawn:

1. Lithium disilicate pressed crowns demonstrated higher fracture resistance than Zirconia reinforced lithium silicate pressed crowns without tempering.
2. The fracture resistance of tempered and non-tempered Lithium disilicate crowns are nearly equal.
3. Tempering protocol used in this study for pressed Zirconia reinforced lithium silicate crowns resulted in the highest fracture resistance in comparison to other tested groups.

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