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The effect of two thermal tempering protocols on the surface roughness of heat-pressed lithium silicate ceramics

Sara Mehanna Foudah[1], Iman Haggag El-Naggar[2], Amr Saleh El-Etreby[3]

Aim: This in vitro study is designed to evaluate the effect of two thermal tempering protocols on the surface roughness of heat-pressed lithium silicates ceramics (LSCs).
Materials and methods: 40 LSCs discs (15×1mm) were heat-pressed and divided into 4 main groups according to type of LSCs with ten discs in each of the main groups; Gp (E): LSC (IPS e.max Press, Ivoclar Vivadent).Gp (L): LSC (GC initial LiSi Press, GC).Gp (C): Zr reinforced LSC (Celtra Press, Dentsply Sirona).Gp (A): Zr reinforced LSC (VITA Ambria, VITA Zahnfabrik). After heat pressing discs were finished and polished according to manufacturer instructions. Surface roughness of each specimen was measured using (SJ-210 surface roughness tester) for Ra(nm) parameter values and surfaces were further analyzed with AFM (ThermoMicroscope, Bruker, Santa Barbara, CA, USA). Discs of each group were subdivided equally according to thermal tempering protocol (n=5); T1 subgroup were subjected to temperature of 9%below pressing temperature, T2 subgroup were subjected to temperature of 5% below pressing temperature. Roughness measurements and AFM analysis were repeated. Data were analyzed statistically with two-way Anova tests.
Results: LSCs type had no statistically significant effect on mean Ra. Thermal Tempering also had no statistically significant effect on mean Ra. The interaction between the two variables had no statistically significant effect on mean Ra.
Conclusions: The surface roughness of LSCs after polishing are almost equal in values with no significant difference related to the type of ceramic, exposing such materials to the suggested tempering protocols have no significant effect on its surface roughness.

Keywords: roughness, tempering, glass, ceramics

1. Lecturer of Fixed Prosthodontics, Faculty of Dentistry, Ain Shams University.
2. B.D.S Faculty of dentistry,6th of October University.
3. Professor of Prosthodontics, Faculty of Dentistry, Ain Shams University.
Corresponding author: Sara Mehanna Foudah, email: sarafoudah@dent.asu.edu.eg

Introduction

Lithium-silicate based ceramics (LSCs) are being widely used in dental practice as a material that combines high levels of esthetic pleasing outcomes with relevant mechanical performance.[1] Today, LSCs are open for both CAD/CAM and heat pressing manufacturing.[1]They represent a sort of a "gold standard" choice whenever monolithic anterior or even posterior restorations are targeted and to a great extent in full rehabilitation situations.[2.] Trials to extend its range of applications to get maximum mechanical benefits of such a material were targeted by dental manufacturers since the early introduction of the first lithium disilicate predecessor named as Empress II, launched in 1998.[3] The unique successor for this predecessor named as IPS e.max Press was launched in 2005 by Ivoclar Vivadent followed by the CAD version that introduced the chair side glass-ceramic option in a year later.[4] Exclusive domination of the dental market as the only lithium disilicate glass ceramics was evident until 2013 when the Glidewell Laboratories introduced Obsidian with the lithium silicate predominating its structure.[5] Few months later, biphasic forms of this material started to invade the market enriching the chemical structure of these forms by oxides such as zirconia oxides to approach mechanical excellence of oxide ceramics[6] with launching of Suprenity PC (Vita Zahnfabrik)[7] and Celtra Duo (Dentsply Sirona).[8] Subjecting this material to firing cycles is an essential step that may be required inherently during the manufacturing process of such material.[9] As the CAD/CAM version of the material was introduced, it was supplied in a partially crystallized state that needs a crystallization cycle to reach its final form and properties.[10] While the hot-pressed forms are essentially subjected to the melting temperature to be pressable inside its mold.[10] Additional firing cycles may be needed for many reasons such as corrections, glazing or sometimes for mechanical healing of microcracks formed during processing.[9] Heat treatments and subjecting the material to additional firing cycles with variable temperatures, heating rates, holding times, or cooling rates have been evidenced to affect the material structure, crystallization formation and growth.[11] Thus, it can have an impact on material's behavior.[9] The outcomes of applying heat to this type of ceramic material repeatedly have generated controversy in the literature. While some discuss how applying heat can cause stress,[12,13] others have noted that it can also have a strengthening impact.[9,14] In 2021 Vita Zhanfabrik launched

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the Ambria Press lithium disilicate with a recommended definite thermal tempering protocol in a temperature of 9% below the firing temperature to increase the mechanical biaxial flexural strength.[15] Modifying this temperature is not addressed enough till now in dental literature according to its impact on different types of LSCs. Surface roughness for such monolithic restorations appears to be very important because it affects surface smoothness and lustrous.[16] Furthermore, surface roughness can have a significant impact on issues such as wear of the opposing tooth surface and plaque retention, which will compromise the restoration's long-term biocompatibility.[16] Thus, well-finished restoration surface can reduce mechanical and esthetic complications as the material is evidently tougher, with higher gloss, and more resistant to color and translucency shifts.[17] The effect of these different tempering protocols on the surface roughness of such restorations is not thoroughly examined in dental literature. This study was designed to evaluate the effect of specific thermal tempering protocols of four different heat pressed LSCs on the surface roughness of these materials. The null hypothesis was that no significant difference would be found in the surface roughness values with four types of heat pressed LSCs under both thermal tempering protocols.

Materials and methods

A total of 40 rounded discs with a diameter of (15mm) and a thickness of (1mm) LSC specimens were fabricated out of 40 pressed ingots; and divided into 4 groups according to material; Gp (E) (IPS e.max Press; Ivoclar Vivadent AG), Gp (L) (GC initial LiSi Press, GC), Gp (C) (Celtra Press, Dentsply Sirona) and Gp (A) (VITA Ambria, VITA Zahnfabrik). Wax patterns (Elastiwax; Keram & Keramik) were invested (IPS e.max Special Investment Material; Ivoclar Vivadent AG), the wax eliminated at 850 ˚C for 1 hour, and the ingots were pressed in a furnace (EP600; Ivoclar Vivadent AG) as per the protocol recommended by each manufacturer (Table 1).

Table 1: recommended pressing temperature T0

Material used	Thermal pressing temperature T0
Vita Ambria	880˚ C
Celtra Press	870˚ C
IPS e.max Press	917 ˚C
Lisi Press	910˚C

The specimens were divested by sandblasting with 50 µm alumina particles at 4 bars of atmospheric pressure. Then smooth divesting was used at 2 bars till complete removable of investment material from discs. After sprues were separated by separating

diamond discs, each attachment point was smoothened by a diamond disc. The samples were finally finished and polished using (Optrafine Ceramic Polishing System) sequentially; using the first tip for 60 seconds with a speed of 10000 rpm. While the next tip was used for the exact same duration of 60 seconds, but with a slower speed of 6000 rpm to simulate the clinical situation. SJ-210 surface roughness tester was used to measure the surface roughness in terms of Ra values. Each sample was put into its designated holder, which moved vertically up to the specimen surface until it barely touched the measuring tip. The surface to be measured was positioned horizontally. Prior to usage, calibration is carried out using the standard calibration specimen.

Testing specifications;

1. Distance of measuring: ten millimeters.
2. Stylus description: tip inclination of 60 degrees, tip diameter of 2 microns.
3. Speed measurement: 0.5 mm/s. delivering 1 mm/s.
4. Measuring 0.75 MN of force.

Parameter of evaluation; three measurements were taken of each sample at a distance of 500 microns, with Ra values given in microns. Then a representing sample of each main group was further examined under AFM (ThermoMicroscope, Bruker, Santa Barbara, CA, USA) for roughness and surface topography. Each specimen's polished surface was washed with 70% alcohol and left to dry at room temperature prior to the AFM examinations. All specimens had polished surfaces, and the AFM was run in the contact mode at room temperature. The polished specimen side was continuously in touch with the cantilever, which had a radial diameter of 10 nm. The sensor sensed the movement of the cantilever as a result of vibrations operating between the cantilever and the sample's contact surface. The cantilever's continual flexing over the heights of uneven surface roughness was measured and observed by software. The various prepared samples' 3D surface topography were accurately recreated. 20 µm × 20 µm digital pictures were captured and scanned gradually. The 40 discs of the 4 major groups of ceramic materials used were subdivided into 2 groups, each group of 5 discs and were subjected to suggested thermal tempering temperature. Subgroup (T1) was subjected to thermal tempering at a temperature 9% below recommended pressing temperature. While the other subgroup (T2) was subjected to thermal tempering at temperature 5% below

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recommended pressing temperature as shown in (Table 2).

Table 2: pressing and thermal tempering temperatures for the four LSCs

Material used	T0	T1	T2
Vita Ambria	880 C	800 C	836 C
Celtra Press	870 C	790 C	826 C
IPS e.max Press	917 C	835 C	872 C
LISI Press	910 C	828 C	865 C

The surface roughness was then remeasured after thermal tempering with the exact above methods of measuring and examined under AFM.

Using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests) and examining the data distribution, numerical data were examined in preparation for statistical analysis. The data were all parametrically distributed. The mean and standard deviation (SD) values of the data were displayed. Two-way ANOVA testing and repeated measures ANOVA were employed. When the ANOVA test is significant, pairwise comparisons were performed using Bonferroni's post-hoc test.

A significant threshold of $P < 0.05$ was established. With IBM SPSS Statistics for Windows, Version 23.0, statistical analysis was carried out. NY / Armonk: IBM Corp.

Results

Before thermal tempering, there was no statistically significant difference between mean Ra (µm) measured with the surface roughness tester between the four lithium silicate ceramics (P-value = 0.142, Effect size = 0.154) as shown in (Figure 1), (Table 3)

Table 3: Mean and Standard deviations Ra(µm) values of four LSCs before thermal tempering

IPS e.max Press		LiSi Press		Celtra Press		VITA Ambria		*P*-value	Effect size
Mean	SD	Mean	SD	Mean	SD	Mean	SD		*(Partial eta squ*
2.95	0.43	2.99	0.45	3.19	0.34	3.28	0.21	0.142	0.154

Significant at $P \leq 0.05$

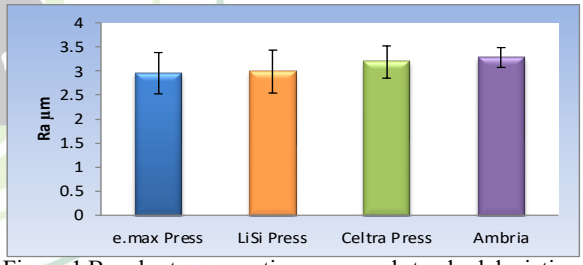

Figure 1 Bar chart representing mean and standard deviation values for surface roughness Ra values of ceramic types at T0

After thermal tempering procedures, there was no statistically significant difference between mean Ra (µm) values of all specimens after any of the thermal tempering protocols whether T1 or T2. As shown in (Table 4).

Table 4: mean and standard deviation of Ra(µm) values for LSCs at T1 and T2

perature	e.max Press		LiSi Press		Celtra Press		Ambria		*P*-value (Between ceramic types)	Effect size *(Partial eta squared)*
	Mean	SD	Mean	SD	Mean	SD	Mean	SD		
T1	2.75	0.26	2.79	0.2	3.16	0.39	3.28	0.23	0.068	0.197
T2	3.14	0.5	3.2	0.57	3.21	0.34	3.29	0.21	0.926	0.014
value tween ratures)	0.104		0.086		0.817		0.946			
ct size tial eta ared)	0.081		0.09		0.002		0.0001			

The AFM images at nanolevel are displayed in Figure 2, Figure 3 showing almost similar surface topography for ceramic samples before and after thermal tempering.

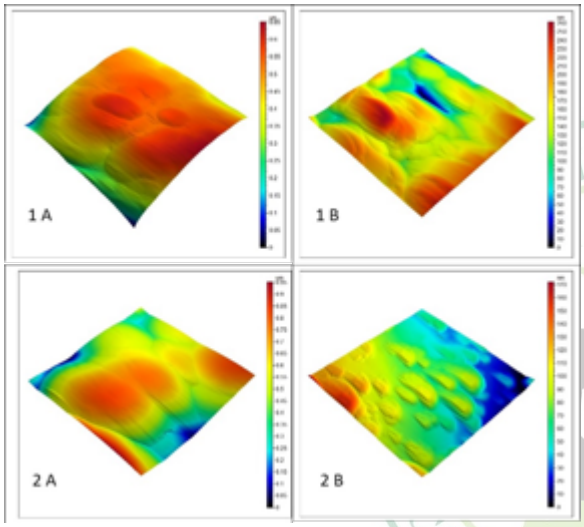

Figure 2: AFM images for IPS e.max Press and LISI Press before and after Tempering; 1=IPS e.max Press, 2=LISI Press, A=before tempering, b=after tempering

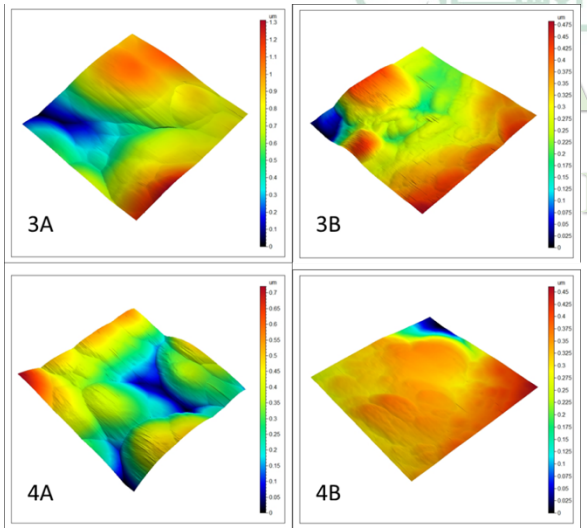

Figure 3: AFM images of Celtra Press and VITA Ambria before and after tempering; 3=Celtra Press, 4=VITA Ambria, A=before tempering, B=after tempering

Table 5: specifications of the selected polishing system OptraFine, NR= not specified by the manufacturer

instrument	Grit	Contents	manufacturer
OptraFine F (coarse)	NR	Synthetic rubber Diamond granulate Titanium dioxide	Ivoclar Vivadent AG Schaan Liechtenstein
OptraFine P (fine)	NR	Synthetic rubber Diamond granulate Titanium dioxide	Ivoclar Vivadent AG Schaan Liechtenstein

Discussion

Every effort is made to target a dental restoration's smooth, polished surface. Increased surface roughness prevents the material from reaching its optimum flexural strength,[18] accelerates the wear of opposing surfaces which consequently increases the area of rough surfaces. That in turn results in a restoration more prone to picking up stains and influences the restorations' final aesthetic results over time,[19] rather than the multiplication of the rates of calculus and plaque accumulation.[20] Assessment of surface quality after polishing procedures can be done through screening and analysis of the surface roughness of the ceramic material.[21,22] Numerical values for surface roughness can be expressed by linear parameters (Ra, Rq, Rz) and three-dimensional parameters (Sa, Sq, Sz).[23,24] In this study, the linear parameter (Ra) was selected for assessment. It is the most roughness parameter commonly assessed in literature for finishing procedures evaluation, it is usually described as the mean arithmetical value of all the absolute

distances of the profile inside of the measuring length.[21,25] At T0 after pressing and before any further thermal tempering procedures all LSCs used showed no statistically significant difference between Ra values despite different microstructure and crystalline content. Literature pointed out to surface roughness as a material-dependent characteristic.[26] The insignificant difference between examined LSCs used could be attributed to the standardized single polishing protocol applied for all specimens using the OptraFine polishing system (Table 5), while specimens were done by the same skillful operator.

Different polishing systems could have led to significant differences in surface roughness between the four different LSCs examined in this study as literature is rich in comparisons between different surface finishing procedures.[27,28] Differences in the grit of polishing tips, substance and elements of polishing tips, or using variable polishing pastes may have a significant impact on the surface roughness results.[27,29] Further investigations may be needed to fully address the effect of such variables on these materials.

All the four LSCs tested in this study were heat pressed which means that these materials exposed to the high temperature for pressing with almost full maturation and growth of the crystals so the repeated firing through thermal tempering may not cause significant extra growth of the crystal size. Using other CAD/CAM LSCs could have recorded different values of surface roughness that may have significantly impacted the results.[16,30]

Exposing samples to thermal tempering with either tested protocol T1 or T2 showed no significant difference in values of surface roughness. This may be attributed to the selection of heat pressed LSCs that all are heated to a very high pressing temperature that allow full maturation of crystals around the already formed nucleating centers.[31] As for lithium silicate-based materials mainly two stages are reached during formation. the first stage is at lower temperature levels (700˚C) in which the nucleation centers are formed and the deposition of lithium disilicate crystals in parallel with lithium metasilicate ones occurs, while the second stage is at higher temperature levels (900˚C) in which growth and maturation occur and the main composition is lithium disilicate after dissolution of lithium metasilicates into lithium disilicates. [32,33] The chosen tempering procedures were slightly below the pressing

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temperature, therefore there might not have been a noticeable change in the surface microstructure that would have affected the samples' surface roughness. While literature is rich with proofs that repeated firings impact the surface roughness of LSCs; Gonuldas et al.[34] reported reduction in surface roughness mean values with increase in number of firing cycles. They referred the cause of this reduction in surface roughness to the modification of pores sizes and geometry inside the dental ceramic with multiple firings. Meanwhile, ÖZDEMİR and ÖZDOĞAN[35] found out a kind of proportional increase in surface roughness with the increase in thermal cycles, they pointed out the microstructural dissipation of different sizes of lithium disilicate crystals into the glassy matrix as the main cause behind such an increase. Miranda et al [36] also pointed out changes in surface roughness with the modifications of number of firing cycle however the significant increase in roughness was related to the higher viscosity of the staining paste they used in the single extra firing cycle. In the present study only two thermal tempering temperatures were selected. Modifying temperatures, tempering time and cooling rates are evidenced to modify structure of such materials that may or may not affect the surface roughness,[37] further investigations to explore the effect of changing such variables on the roughness of the LSCs are needed.

The quantitative numerical data for surface roughness values in this study was measured with the surface roughness tester. Despite simplicity, being user friendly, utility and effectivity of such method, it does not document the silhouette; it automatically processes the movement over the surface profile to display the numerical data. [38] While the AFM analysis in this study was mainly for qualitative image of the surface roughness profile, numerical data couldn't be used either for statistical analysis nor for correlation between the two methods. A random single specimen from each group was selected for AFM analysis which cannot be relied on as a sample size to be statistically analyzed. However, the higher accuracy and sensitivity of AFM at nano-level may have led to significant differences in surface roughness measurements if it was mainly used to record the numerical qualitative and quantitative roughness parameters.

The current investigation is unable to rule out the null hypothesis and the results failed to reject it that no significant difference was statistically found in values of surface roughness between the four types of LSCs

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under any of the tested thermal tempering protocols.

Conclusions

1. For the heat pressed LSCs, polishing can be relied on to produce smooth surface after processing or clinical adjustments.

2. Thermal tempering procedures mentioned can be used safely without affecting the surface roughness of such monolithic restorations.

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