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Effect of Repeated Heat Pressing on Microstructure, Hardness and Surface Topography of Two Pressable Ceramics

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ORIGINAL STUDY

Effect of Repeated Heat Pressing on Microstructure, Hardness, and Surface Topography of Two Pressable Ceramics

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Abstract

Purpose: To evaluate the impact of frequent heat pressing of Lithium disilicate (IPS e.max press) and zirconia reinforced lithium disilicate (Celtra press) on their microstructure, hardness, and surface topography. **Patients and methods:** Two marketable pressable glass ceramics; E.max (Ivoclar, vivadent) and Celtra (Dentsply, Sirona) (pressable types) were utilized in this study. The two materials were used to construct a total of 40 discs with 2 mm thickness and 10 mm diameter representing two groups (n = 20) group I: E.max discs and group II: Celtra discs. Every group was split up into two even subgroups regarding the number of pressing cycles; subgroup (A): Pressed ceramic discs and subgroup (B): Repressed ceramic discs, (n = 10) each. Then the discs were subjected to thermomechanical fatigue using a chewing simulator, resembling 3 and 6 months. Microstructure, hardness, and surface topography were examined at baseline, before thermomechanical fatigue representing 3 and 6 months. Data were collected, employing Pearson's correlation tests and One-way analysis of variance for statistical analysis. The threshold for significance was fixed at *P* less than 0.05. **Results:** At baseline: no statistically significant difference existed between the mean value of all subgroups in hardness and surface topography. Hardness decreases and surface topography increases gradually after aging. **Conclusions:** The repressing of both materials has no impact on their surface hardness and roughness. Thermo-mechanical aging negatively affects the surface hardness and topography of E.max and Celtra in fresh and repressed states.

Keywords: Repressing, Microstructure, Hardness, Topography, Celtra press, E.max press

1. Introduction

Numerous varieties of ceramic systems have been developed recently. These ceramics can be used in construction of different indirect restorations, ranging from the ultra-conservative no preparation veneers reaching to the posterior multi-unit fixed partial denture [1].

The outstanding development in all ceramic materials facilitated the opportunity to fabricate life like restorations that showed long-term and reliable results. Unfortunately, all ceramic dental materials are vulnerable to stress and could be harmed by thermal treatment during fabrication procedures in the form of micro-cracking, flaws, or surface defects. The reliability and clinical performance of the restoration may be affected by the precision of the fabrication process and even by the skill of the individual dental technician [2].

To understand the clinical potential and the dental ceramic limit, mechanical properties such as hardness and strengths are evaluated.

Different methods have been utilized for ceramic restoration fabrication ranging from the simple conventional technique to new methods using computer software for designing and fabrication. Another method which combines the application of high temperatures and external pressure (pressable ceramics) has been used [3].

Lithium disilicate has been regarded as the most well-liked and esthetic material to be created in the laboratory using the hot-pressing technique. Many developments in glass ceramics that improved their qualities, by improving the crystalline structure,

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caused the development of zirconia-reinforced lithium silicate glass-ceramics (ZLS) [4].

ZLS contains lithium silicate crystals (the crystalline phase) and a glassy matrix which is strengthened by zirconium oxide fillers. These fillers' percentage is 10% by weight to function as a nucleating agent that stops crack spread [5].

The dental labs aim to expedite as many restorations as possible in each time to decrease the expenses and the wasted material as much as they can. Unfortunately, this can not be applied every time, some dental labs may repress the residual material to be used in new restorations. Sufficient knowledge about this procedure should be studied to clarify whether leftover material should be discarded or reused [6].

Aging of dental restoration is a process that is enhanced by saliva, masticatory stresses and chemical deterioration. It is important to simulate the oral environmental conditions extraoral to study the restoration's clinical effectiveness in the research. For evaluation of the change in the surface properties of the dental materials, a chewing simulator is used. This device exerts forces similar to the masticatory forces and applies different temperature fluctuations [7].

The esthetic quality of the all-ceramic restorations is essentially affected by the degree of smoothness and surface topography of the material [8]. Improperly finished surface increases the tendency of restoration discoloration, plaque accumulation, and wear of the opposing restoration or the dental structures, moreover, the translucency is decreased and the mechanical properties are compromised leading to failed dental restoration [9].

Mean surface roughness (Ra) of the dental restoration should be to a minimum. It has been stated that Ra should be equal to or less to 0.2 μm otherwise excessive plaque will be accumulated. Smooth restorations also facilitate the maintenance of good oral hygiene [8].

In addition to esthetics, optimum mechanical properties are essential for long-term dental restoration. One of the most critical characteristics of the mechanical properties is the surface hardness. Surface hardness describes the amount of abrasiveness exerted on the opposing natural teeth and it measures the material resistance to penetration, scratching and permanent indentation.

The mechanical strength of the material is usually correlated to its surface hardness [10].

As previously mentioned, the clinical performance of dental ceramics is affected by microstructure, hardness, and surface topography. Therefore, the present investigation aims to investigate the consequence of frequent pressing of E.max and Celtra on their microstructure, hardness and surface topography.

2. Patients and methods

2.1. Sample size calculation

To calculate size of samples of the study, Independent *t*-test was helped to link the effect of various crown materials and pressing cycles according to a previous study [11].

Using a two-sided assumptions test and a significance level (1-β error) of 0.05 for the data, a total specimen size of 20 (10 for every group) was enough to determine an effect size of 1.44 with a power (1-error) of 0.8. To determine specimen size, G power Program version 3.1.9.2 was employed.

2.2. Ethical approval

The Research Ethics Committee, Faculty of Dental Medicine for Girls, Al-Azhar University approved our study under code: (REC-CR-21-01).

2.3. Samples grouping

Two marketable ceramic materials were utilized in the current investigation; E.max (Ivoclar, vivadent) and Celtra (Dentsply, Sirona) (pressable types). The two types were utilized to produce discs. Fabrication of forty discs with 2 mm thickness and 10 mm diameter to represent two groups in accordance with the kind of ceramic.

Group I: E.max discs.
Group II: Celtra discs.

The discs for each type were divided at random into two groups of equal size in accordance with the number of pressing cycles into:

Subgroup A: Pressed ceramic discs, (n = 10).
Subgroup B: Repressed ceramic discs (*n* = 10).

2.4. Ceramic discs fabrication

2.4.1. Fabrication of pressed ceramic samples, subgroup (A)

To make a standardization in disc-shaped multiple wax patterns, a split custom made Teflon mold was created (2 mm thickness, 10 mm diameter).

After fabrication of disc-shaped wax patterns (BEGO Bellavest, Begosol, BEGO Co, Germany), spruing and investing were made following the

manufacturer's instructions. The investment casted aside to set for an hour before starting wax elimination. Wax eradication was done using wax-burn out furnace (Ney, US Dental Depot, USA) in accordance with the advice of the manufacturer.

Pressing of ceramic discs was done in an EP3000 press furnace (Ivoclar, Schaan, Liechtenstein, Germany) by using new ingot of IPS emax following manufacturer's instructions. Immediately after the program was finished, the investment ring was taken out from the oven and casted aside to chill at lab temperature for approximately 1 h on a wide-entangled grid (E.max cooling rack, Ivoclar, Schaan, Liechtenstein, Germany), this made sure that the investment ring cooled quickly and uniformly [12].

Discs were removed from the investment ring using an abrasive disc (Ivoclar, Schaan, Liechtenstein, Germany), polished using polishing beads (Ivoclar, Schaan, Liechtenstein, Germany) at 4 bar (58 psi), maintaining a safe distance to avoid damage of the freshly pressed discs. Once the discs were exposed, fine divestment was obtained at 2 bar (29 psi) pressure.

The ceramic residue on the Alox Plunger was removed using type 100 Al_2 O_3. Then the pressed discs were sunken in Invex liquid (Ivoclar, Schaan, Liechtenstein, Germany) for 30 min to move away the reaction layer of the investment, then rinsed under running water and dried. After that, they were blasted with glass beads at 2 bar pressure to remove any remaining reaction layer. For Celtra Press, all the procedure of investment and pressing IPS e.max discs were repeated while adjusting the pressing parameters following the advice of the manufacturer [12,13] as shown in Table 1.

2.5. Fabrication of repressed ceramic samples, subgroups B

The fresh discs were separated from the sprues and buttons using abrasive disc mounted on straight angle hand piece. The sprues were discarded and the buttons were kept for further use. The buttons were finished and adjusted to look like the shape of a new ingot using diamond discs and stones. The previously mentioned steps for construction of subgroup A samples were carried out again using the trimmed left-over buttons to produce repressed discs (subgroup B).

2.6. Testing procedures

2.6.1. Microstructure analysis of ceramic samples using scanning electron microscope (SEM) at baseline

The microstructure of the samples of both groups was assessed using scanning electron microscopy (SEM). Samples were etched by 9.5% hydrofluoric acid for half min then rinsed perfectly for half min with air-water spray.

After that, samples were cleaned with ultrasonic cleaning equipment and distilled water. Following ultrasonic cleaning, the microstructure was examined by an environmental SEM (JSM-IT200 InTouchScopeTM Scanning Electron microscopy; JEOL, Tokyo, Japan) at a magnification of 10,000×.

2.6.2. Surface hardness evaluation at baseline

Using a Vickers diamond indenter and a 20× objective lens on a digital display Vickers micro-hardness tester (Model HVS-50, Laizhou Huayin Testing Instrument Co., Ltd., China), the surface micro-hardness of the samples was measured. A 100 g force was applied to the samples' surfaces for 20 s. Three indentations were made on the surface of each sample, each one being no more than 0.5 mm distant from the other and evenly placed around a circle.

The slanted length of the indentations calculated using a built-in scaled microscope, and Vickers values were transfigured to micro-hardness values. The following formula was used to calculate the micro-hardness: ($HV = 1.854\ P/d2$), where HV is the Vickers hardness expressed in Kg.f/mm^2, where P is the load in Kg.f and d is the diagonal length in mm.

2.6.3. Surface topography determination at baseline

The demand for noncontact, quantitative surface topography characterization is typically satisfied by optical approaches. Scope Capture Digital Microscope, from Guangdong, China, which has a built-in USB camera was used to take pictures of the samples. With a constant magnification equal (120×) its resolution equal (1280 × 1024 pixels per image), the camera was attached to an IBM suitable personal computer.

To specify/standardize the zone of topographical measurement, digital microscope images were cropped to 350 × 400 pixels by Microsoft Office Image Manager help. Utilizing WSxM software (Version 5 Develop 4.1, Nanotec, Electronica, SL), the clipped images were examined [14].

Table 1. Firing parameters for E.max press and Celtra.

	Standby temperature [B]	Temperature raises rate [t]	Final pressing temperature [T]	Holding time [H]	Stopping speed [E]
IPS e.max	700 °C	60 °C/min	917 °C	25 min	250 μm/min
Celtra Press	700 °C	40 °C/min	860 °C	30 min	250 μm/min

All limits, sizes, frames, and measured characteristics are uttered in pixels within the WSxM software. The pixels were therefore calibrated into absolute real-world units using this method. To calibrate, a known-size object (in this instance, a ruler) was compared with a scale produced by the software. Then, a three dimensional representation of the surface profile of the samples were produced.

Each sample had five three dimensional photos taken on its sides and center, the photos were 10 μm × 10 μm in size. The average of heights (Ra) reported in μm was calculated using the WSxM software, which is regarded as an accurate indicator of surface topography [15].

2.6.4. Thermo-mechanical fatigue

Samples of the tested groups and subgroups were exposed to thermo-mechanical fatigue using a programmable logic-controlled device chewing simulator (ROBOTA, AD-TECH Technology CO., Germany) incorporated with thermocycling protocol operated on servomotor.

The process included the application of a weight of 5 Kg, equal to 49 N of chewing force, in the middle of the occlusal surface for 37,500 cycles to clinically simulate 3 months chewing conditions. Simultaneously, samples were subjected to thermocycling between 5 and 55 °C with a dwell time of 60 s [16]. Chewing guidelines adjusted as shown in Table 2.

The procedure was conducted for 375,000 cycles to simulate 3 months chewing conditions followed by another 375,000 cycles to simulate a total of 6 months chewing conditions.

2.6.5. Measurements at 3 months and 6 months clinical simulation

Microstructure, surface hardness, and surface topography evaluations were done after 3 months and 6 months clinical simulations repeating the same criteria followed at baseline.

2.7. Statistical analysis

Data management and statistical analysis were performed using the Statistical Package for Social Sciences (SPSS) version 18. Numerical data were represented as means and standard deviation values.

By helping of the mean, standard deviation, and confidence intervals, numerical data were summarized. Data was distributed along the side of performing the KolmogoroveSmirnov and ShapiroeWilk tests, to examine its normality. A oneway analysis of variance test was used to examine differences across groups in terms of normally distributed numerical variables, after that; Bonferroni's post hoc test was done. The KruskalleWallis test and Wilcoxon signed rank test were used to collate nonparametric data on the difference and percent change within the same group. The formula: [(value after-value before)/value before 100] was used to calculate the percent change. All P values are two-sided, P values less than or equal to 0.05 were considered significant.

Table 2. Chewing simulator (ROBOTA) guidelines.

Chewing simulator guidelines	
Vertically motion: 1 mm	Horizontally motion: 3 mm
Accelerated speed: 90 mm/s	speed forward: 90 mm/s
Descending speed: 40 mm/s	rearward speed: 40 mm/s
Cycle recurrences 1.6 Hz	Weight for every sample: from 3 kg
Torque; 2.4 N m	

3. Results

3.1. Microstructural evaluation

3.1.1. At baseline

The SEM photomicrograph (10,000×) viewed needle-shaped fragments noticed in the two samples. Freshly pressed samples revealed a microstructure with many needle-like crystals packed closely together and interconnecting in multiple directions. However, after repressing, larger, wider crystals with a dispersed distribution became visible. The crystal ends with their pointed needle shapes vanished (Fig. 1).

The mean particle dimensions of IPS e.max press were (1.61 μm, 0.31 μm) in the pressed samples while the mean particle dimensions were (2.42 μm, 0.53 μm) in the repressed samples. The mean particle dimensions of Celtra press were (2.22 μm, 0.41 μm) in the pressed samples while the mean particle dimensions were (3.19 μm, 0.62 μm) in the repressed samples.

After thermo mechanical aging 3 and 6 months: There were more pores and fissures found between the crystals (Fig. 2).

Blue arrows are pointing to cracks and yellow arrows are pointing to pores.

3.2. Surface hardness evaluation

(a) At baseline; the repressed Celtra subgroup recorded the highest mean value followed by the repressed E.max press subgroup, followed by the freshly pressed E.max press, and finally freshly pressed Celtra subgroup, however, there was no statistically significant difference in the means of any of the subgroups.

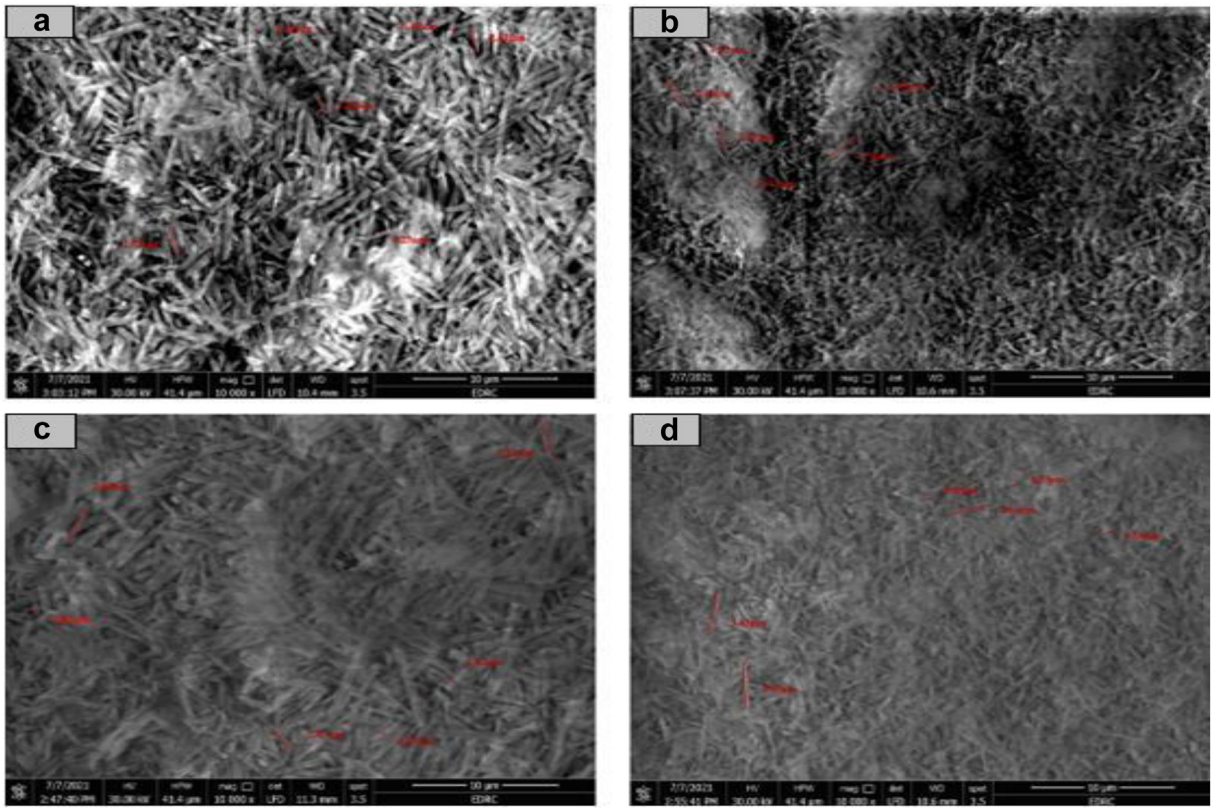

Fig. 1. Scanning electron microscopy photomicrograph at baseline (10,000×). (a): E.max freshly pressed, (b): E.max repressed, (c): Celtra freshly pressed, (d): Celtra repressed.

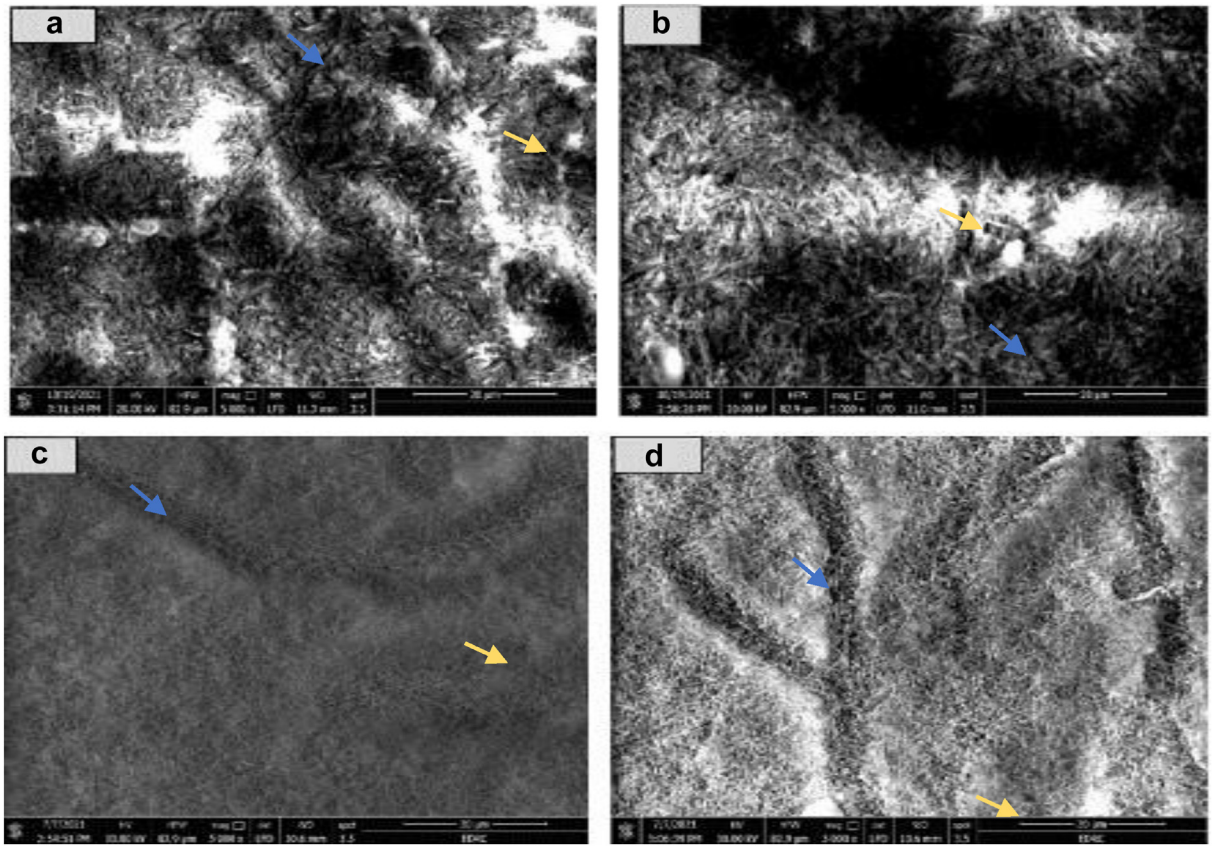

Fig. 2. Scanning electron microscopy photomicrograph after thermo-mechanical fatigue (10,000×). (a): E.max freshly pressed, (b): E.max repressed, (c): Celtra freshly pressed, (d): Celtra repressed.

(b) After thermo mechanical fatigue, the mean values showed a statistically significant gradual decrease from baseline to after 37,500 cycles to after 75,000 cycles in all subgroups as in Table 3.

3.3. Surface topography evaluation

(a) At baseline; freshly pressed IPS e.max press recorded the largest mean value followed by freshly pressed Celtra subgroup, followed by repressed E.max press, finally repressed Celtra subgroup, however, there was no statically significant difference between the mean value of all subgroups.
(b) After thermo mechanical fatigue, mean values showed a statistically significant gradual increase from baseline to after 37,500 cycles to after 75,000 cycles in all subgroups as viewed in Table 4.

4. Discussion

Pressable glass ceramics biocompatibility, great aesthetics, and superior mechanical qualities have made these restorations attract a lot of interest [6].

Furthermore, due to its ease of use, improved crystalline dispersion in the vitreous matrix, improved marginal adaptation, and lesser porosity, the heat-pressing technique has gained popularity for creating glass-ceramic restorations in contrast to the sintering method [17].

E.max was selected to be used in this study because of its high esthetic and mechanical properties. It is made up of 70% lithium disilicate crystals in a glassy matrix [18].

Additionally, Celtra press was utilized in this study because of its improved translucency and suitable flexural strength values [19]. It is made up of lithium silicate crystals and a glassy matrix that is strengthened with fillers that are weighting 10% of

Table 3. Descriptive statistics and comparison of Vickers Hardness (HV) between subgroups (analysis of variance test).

HV		Mean	SD	95% Confidence Interval for Mean		Min	Max	F	*P*
				Lower Bound	Upper Bound				
Baseline	C1	389.66	6.86	381.15	398.18	383.66	398.32	0.107	0.955 ns
	C2	392.85	4.51	387.25	398.46	387.81	397.82		
	E1	392.41	7.93	382.56	402.25	382.91	400.52		
	E2	392.62	16.92	371.61	413.62	365.81	410.82		
After 37,500	C1	364.45^{a}	6.17	356.80	372.11	356.59	370.26	3.33	0.046*
	C2	$350.49^{a,b}$	18.10	328.02	372.97	324.85	368.72		
	E1	$345.56^{a,b}$	11.19	331.66	359.45	333.55	357.71		
	E2	335.93^{b}	18.89	312.48	359.38	312.99	355.91		
After 75,000	C1	314.51^{y}	8.79	303.59	325.42	305.34	328.45	7.66	0.002*
	C2	$281.50^{y,z}$	39.55	232.39	330.61	243.17	345.50		
	E1	332.30^{x}	5.08	326.00	338.61	325.70	339.45		
	E2	270.04^{z}	22.42	242.20	297.88	242.35	304.24		

Significance level *P* less than or equal to 0.05, *significant, ns = nonsignificant, Means with different superscript letters are statistically significant ($P < 0.05$).
C1: Celtra freshly pressed, **C2**: Celtra repressed, **E1**: E.max freshly pressed, **E2**: E.max repressed.

Table 4. Descriptive statistics and comparison of topography between groups (analysis of variance test).

Topography		Mean	SD	95% Confidence interval for mean		Min	Max	F	*P*
				Lower bound	Upper bound				
Baseline	C1	0.2507	0.0016	0.2487	0.2526	0.2484	0.2527	0.988	0.424 ns
	C2	0.2490	0.0028	0.2455	0.2524	0.2469	0.2538		
	E1	0.2510	0.0020	0.2486	0.2534	0.2477	0.2528		
	E2	0.2497	0.0018	0.2475	0.2519	0.2478	0.2518		
After 37,500	C1	0.2506^{b}	0.0031	0.2468	0.2544	0.2473	0.2540	7.204	0.003*
	C2	$0.2548^{a,b}$	0.0055	0.2480	0.2615	0.2490	0.2629		
	E1	0.2540^{b}	0.0023	0.2560	0.2616	0.2563	0.2618		
	E2	0.2597^{a}	0.0021	0.2571	0.2623	0.2571	0.2618		
After 75,000	C1	0.2501^{y}	0.0021	0.2475	0.2528	0.2479	0.2529	1.808	0.016*
	C2	0.2575^{x}	0.0015	0.2556	0.2594	0.2550	0.2590		
	E1	0.2588^{x}	0.0025	0.2509	0.2570	0.2516	0.2570		
	E2	0.2603^{x}	0.0142	0.2427	0.2779	0.2401	0.2800		

Significance level *P* less than or equal to 0.05, *significant, ns = nonsignificant, Means with different superscript letters are statistically significant ($P < 0.05$).
C1: Celtra freshly pressed, **C2:** Celtra repressed, **E1:** E.max freshly pressed, **E2:** E.max repressed.

zirconium oxide and behave as a nucleating agent to stop fracture growth [4].

Pressable ceramic materials are accessible in variable ingot sizes. Large ones are helping to fabricate multiple restorations in the same time; thus, the ingot may be fully or incompletely used, so a valuable quantity of depleted material is usually discarded by lab staff [8]. Therefore, recycling this waste could change the microstructure, which could change the material's qualities including hardness and surface topography.

The effect of frequent pressing of E.max and Celtra on microstructure, hardness, and surface topography of the material before and after thermomechanical aging was investigated by this study.

In the current investigation, repressed samples' SEM pictures (Fig. 1a, c) showed low densely crammed lithium disilicate crystals with evident crystal expansion compared with fresh pressed samples' SEM images (Fig. 1b, d). The crystals are what undergoing redistribution and growth as a result of more pressure and temperature [20].

All SEM observations from earlier frequently pressed lithium disilicate ceramic research [2,6,20] have consistently shown the crystal expansion of frequently pressed lithium disilicate. The ripening of Ostwald, in which massive crystals form at the cost of the little ones [21].

To enhance clinical performance, surface hardness, a measure of how abrasive a material that opposes natural dentition, is a crucial component of an ideal restorative material. A material's resistance to piercing, scratching, or chronic indentation is determined by this surface property. It is typically connected with the mechanical strength of the material. Any ceramic material with a high hardness value has reportedly been shown to be able to withstand strong masticatory forces [10]. Therefore, surface hardness was evaluated in the present study to assess whether repressing will affect it or not.

The hardness values of both materials in the current investigation slightly decreased following repression, although there was no statistically significant difference between the groups (Table 3). This finding is consistent with a prior study's finding that the first pressed lithium disilicate group E.max and the repressed group had similar Vickers microhardness values [20].

Additionally, this outcome is the same as a different study that discovered that the hardness of lithium disilicate considerably decreases after repression, indicating that this material can be reused while preserving good mechanical capabilities and without damaging the environment radically changing the crystalline or chemical makeup [22].

However, in another study that concluded that repeated heat-pressing is bad for the hardness of IPS e.max Press, Vickers hardness dramatically decreased after two heat-pressing sessions [23].

In addition, specific factors and circumstances have an impact on the surface Ra of all ceramic restorative materials. It was reported that a clinically acceptable Ra (mean surface Ra) threshold for restorative material was 0.2 μm and when Ra is greater than 0.2 μm, excessive plaque development takes place [8]. Smooth restorations have been shown to improve oral hygiene and patient comfort [9].

An increased probability to color changing, microbial plaque, wear to opposing restorations or hard tooth component, lower translucency as well as decreased mechanical strength may be caused by poorly finished all ceramic surfaces. Features that ultimately cause the restoration to fail [24].

In the current study, the mean value of surface Ra of both materials was slightly decreased after repressing with no statistically significant difference while still in the clinically accepted range. This result agrees with the result of another study that found no statistically significant difference between pressed and repressed E.max groups [25].

Heat pressing is done under pressure from a pneumatic press furnace, which is what led to this outcome. Additionally, the enhanced density and homogeneity provided by the ingot delivery form avoid the production of faults within the ingot. Both produced samples that were less porous and devoid of defects [25].

Additionally, this finding was reached for the two tested materials, E.max and Celtra press; the mean Ra value (μm) of the press and repress states was not statistically significantly different.

Concluding that, it is possible to recycle both of the tested glass ceramic materials without appreciably changing the surface Ra [26].

Ageing of intraoral dental restorations is accelerated by mastication-related mechanical forces, chemicals, and saliva. For scientific study to be clinically applicable, oral environmental conditions must be simulated *in vitro*. A chewing simulator is an effective tool for examining variation in the surface characteristics of dental materials during use in a patient mouth [10].

In the current investigation, Tables 3 and 4 show that following thermo-mechanical ageing, hardness decreased and Ra increased. This can be attributed to the effect of thermo-cycling.

Thermal stresses at the crystal matrix interface could have resulted from an imbalance in the coefficient of thermal expansion between the crystal contents and the glassy matrix, and as a result, microcrack formation. Resulting in reducing hardness while increasing Ra.

However, the present study used thermo mechanical fatigue simulating only 6 months. The obtained results may differ if the longer simulation is applied. Although clinical simulation was followed as much as possible, still the present study has the limitations of *in vitro* studies.

Validation of obtained results through *in vivo* studies is therefore recommended.

4.1. Conclusions

(a) Repressing of both E.max and Celtra glass ceramics has no impact on their surface hardness and topography.
(b) Thermo-mechanical aging negatively affects the surface hardness and topography of E.max and Celtra glass ceramics in fresh and repressed states.

4.2. Recommendations

This study has a broad range of applications and could also push other investigators to evaluate other properties of both materials. Also, further investigation evaluating the hardness and surface topography with increased time is recommended.

Funding

This investigation got no funding.

Ethical approval

The Research Ethics Committee, Faculty of Dental Medicine for Girls, Al-Azhar University approved our study under code: (REC-CR-21-01).

Conflicts of interest

There are no conflicts of interest.

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