Research Square

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Effect of heat re-pressing and thermocycling on the mechanical properties of high-density micronized heat-pressed ceramics: an in vitro study

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Abstract

Background

Heat re-pressing discarded high-density micronized (HDM) glass ceramic could reduce waste and cost, but whether re-pressed material retains equivalent properties is unclear. We assessed the effects of heat re-pressing and thermocycling on the mechanical properties of HDM glass ceramic.

Methods

Forty-two HDM glass ceramic discs (GC Initial LiSi Press) were single (control), double, or triple heat-pressed (n = 14/group), with each group subdivided before and after thermocycling (5000 cycles, 5–55°C; n = 7/group). Elemental content, microstructure, biaxial flexural strength, and surface hardness were assessed. A separate sample set was prepared for μtensile testing (n = 12/group). Data were analyzed by two-way ANOVA and Tukey's post hoc test.

Results

Re-pressing altered the microstructure, changing lithium disilicate crystal shape, decreasing crystal size, and increasing crystal density. Re-pressing had no effect on biaxial flexural strength ($p = 0.074$) but increased surface hardness ($p < 0.001$) and reduced μtensile strength ($p < 0.001$). Thermocycling decreased crystal size and density and, although it did not affect biaxial flexural strength ($p = 0.384$), reduced both surface hardness and μtensile strength ($p < 0.001$).

Conclusions

Heat re-pressing altered the microstructure and mechanical properties of HDM glass ceramic. Future studies should confirm whether these alterations are clinically beneficial or detrimental.

BACKGROUND

Dental ceramic restorations are a popular choice in dentistry due to their favorable esthetics and durability. While all-ceramic restorations deliver long-term clinical success, they are also inherently susceptible to microcrack development during processing, compromising their physical and mechanical properties [1, 2]. Glass ceramic restorations can be classified according to their microstructural composition or processing technique, where the former includes the amount and type of glass and the crystalline structure ratio, while the latter includes mixing powder and liquid, heat pressing, and milling [3].

Glass ceramics can be fabricated by heat pressing to improve crystallization and thus marginal adaptation [4]. Heat pressing involves pressing ingots into molds in furnaces under high heat and pressure; then, after cooling, excess material is discarded. Recycling leftover sprues and buttons could be an approach to reducing waste and cost. However, there are few data on the effect of heat re-pressing on the mechanical and physical properties of the materials. Repeated heat cycling and processing could affect the ceramic microstructure, thus altering its characteristics and, potentially, its clinical performance [5, 6].

There have been a few analyses of changes in the physical and mechanical properties of dental ceramics after heat re-pressing. Where it does exist, evidence on the effects of heat re-pressing ceramics on their microstructure is conflicting. Chung et al. [7] showed that a second heat press cycle had no effect on the microstructure of lithium disilicate ceramics (IPS Empress 2, Ivoclar, Schaan, Liechtenstein), but several other studies have shown that a second heat press cycle modified the lithium disilicate microstructure (IPS e.max Press and Empress 2, Ivoclar, Schaan, Liechtenstein), with crystals embedded in the glass matrix becoming less packed (IPS e.max Press), more uni-directional, and larger and wider. Additionally, porosity and cracks increased (IPS e.max Press) [5, 6]. There are similar conflicting results on the effects of heat re-pressing ceramics on their biaxial flexural strength. One study reported that a second heat-pressing cycle reduced the biaxial flexural strength of lithium disilicate ceramic (IPS e.max Press) [6], whereas other investigations found no significant effect of an additional pressing cycle on the flexural strength of lithium disilicate systems (Empress I and Empress II) [5, 8]. Yet another study showed that a second heat pressing cycle significantly increased the biaxial flexural strength of lithium disilicate (Empress 2) [7]. Mirroring these findings, the effects of heat re-pressing ceramics on hardness are similarly ambiguous, with one study demonstrating that a second heat press cycle reduced the surface hardness of lithium disilicate ceramics (IPS e.max Press) [6], and another showing that surface hardness did not significantly change after two cycles of heat pressing lithium disilicate ceramics (GC Initial LiSi Press, GC America, Alsip, IL, USA and IPS e.max Press) [8, 9].

Nevertheless, the value of reducing waste and cost by heat re-pressing ceramics mandates further investigation and clarification of any adverse impacts. Therefore, the aim of this study was to test whether repeat heat re-pressing GC Initial LiSi Press glass ceramic alters its mechanical and physical properties with respect to elemental content, microstructure, biaxial flexural strength, surface hardness, and μtensile strength. The null hypothesis was that there would be no effect on the microstructure and mechanical properties of GC Initial LiSi Press ceramics from heat re-pressing.

METHODS

Sample preparation

The experimental details of specimen preparation can be found in our previous publication [10]. Briefly, forty-two 1-mm thick, 15-mm diameter monolithic lithium disilicate glass ceramic discs (GC Initial LiSi Press, GC, Tokyo, Japan) were prepared (ISO 6872 specifications) and divided into three groups: (1)

single heat pressing (control); (2) double heat pressing; and (3) triple heat pressing (n = 14 per group). Each group was further randomly subdivided into two groups (n = 7 per group): before and after thermocycling. Discs were prepared using custom-made Teflon molds and heat pressing [stand-by temperature 700°C, press temperature 920°C, 25 min holding time (Programat EP3000, Ivoclar Vivadent, Schaan, Liechtenstein)]. Samples were then devested (IPS PressVEST, Ivoclar Vivadent, Schaan, Liechtenstein) by sand blasting (50 μm Alu-Oxid, 3 bar). After devesting, specimens undergoing one or two additional pressing cycles were prepared by separating the residual sprues and buttons from the initial press using a diamond wheel saw. The discs were then ultrasonically cleaned for 15 minutes and refined by grinding to ensure proper adaptation within the refractory mold. All specimens were subsequently sandblasted under low pressure (50 μm aluminum oxide, 1.5 bar). For artificial aging, discs were subjected to thermocycling for 5,000 cycles between 5°C and 55°C according to ISO 10477, with dwell and transfer times of 15 seconds (SD Mechatronik, Feldkirchen-Westerham, Germany), corresponding to approximately five years of clinical service [11].

Energy dispersive X-ray analysis (EDAX)

EDAX was used to assess the effects of heat re-pressing and thermocycling on the elemental composition of the GC Initial LiSi Press ceramic. Discs were placed on a holder of the diffractometer (X'Pert^3 MRD; Malvern Panalytical, Malvern, UK; PW 3040/60) and scanned using a Cu Kα X-ray angle from 20–40° and a 5s step interval.

Scanning electron microscopy (SEM)

Sample microstructure was assessed by field-emission scanning microscopy (FESEM; Zeiss Sigma300P, Oberkochen, Germany). All discs were cleaned and etched with 9.5% buffered hydrofluoric acid gel for 90 seconds following the manufacturer's instructions (Bisco Inc., Anaheim, CA, USA). Then, the etchant was washed off with distilled water and cleaned with ultrasonic bath cleaner. Later, the samples were dried and sputter coated with gold. The assessment was performed at 5000x magnification.

Biaxial flexural strength

The same samples were used for biaxial flexural strength testing using a piston-on-three-ball test. Specimens were centrally positioned on a custom-made metallic platform (10 mm diameter) supported by three equidistant steel balls (3.2 mm diameter each). Then, a load was applied with a universal testing machine (Instron Co., Canton, MA, USA) through a flat punch tip diameter of 1.4 mm at a crosshead speed of 0.5 mm/min. Finally, the fracture load for each disc was recorded, and the corresponding biaxial flexural strength was calculated using the following equation:

$$S= [-0.2387P(X-Y)]/d2$$

where S is maximum tensile stress (MPa), P is fracture load (N), and d is specimen disk thickness at fracture origin (mm).

X and Y were determined as follows:

X: $(1 + n) \ln(r2 /r3)2 + [(1 – n) / 2] (r2 /r3)2$

Y: $(1 + n) [1 + \ln(r1 /r3)2] + (1 – n) (r1 /r3)2$

where n is Poisson's ratio, r1 is the radius of the support circle (mm), r2 is the radius of the tip of the piston (mm), and r3 is the radius of the specimen (mm). Poisson's ratio is 0.24 for lithium disilicate ceramic.

Vickers surface hardness

A randomly chosen fractured segment from each specimen was chosen to test Vickers surface hardness. The hardness was measured using micro-hardness tester (Tukon 1102 Wilson, Buehler, Germany) with a load of 1000 g applied without impact while forcing the indenter into the specimen. The indenter was held in place for 15 seconds. Following removal of the load, the indentation was observed with a magnifying microscope, and the two impression diagonals were measured to the nearest 0.1 μm with a micrometer and averaged. Vickers hardness (HV) was calculated using the following equation:

$HV = 1854.4L/d2$

where L is the load applied and d is the average diagonal.

μTensile strength

μtensile testing was performed as previously [12]. Briefly, samples were prepared by cutting the blocks cross-sectionally using a low-speed diamond saw (Smart Cut 6009, UKAM Industrial Superhard Tools, Valencia, CA, USA) to produce four cubes (10 x 10 x 6 mm) from each press. All bonding surfaces (10 x 10 mm) were abraded (600-grit silicon carbide paper, 15 seconds) and washed with water for 60 seconds. The bonding surfaces were etched with 9.5% hydrofluoric acid for 90 seconds and then rinsed with water and dried. After that, a thin layer of silane treatment (Monobond N, Ivoclar, Schaan, Liechtenstein) was applied, air dried for 1 minute, and the adhesive system (Tetric N-Collection, Ivoclar, Schaan, Liechtenstein) applied, air dried, and light cured (3M Elipar DeepCure-S LED Curing Light, St. Paul, MN, USA) for 20 s. Subsequently, nanohybrid composite (Tetric N-Collection, Ivoclar, Schaan, Liechtenstein) was applied in 2 mm increments and then light-cured for 40 seconds to create a 6 mm thickness forming ceramic/composite blocks. These ceramic/composite blocks were longitudinally sectioned with a low-speed diamond saw at 1 mm increments. Twenty-four ceramic/composite beams (1 x 1 x 12 mm) per press were prepared. Six groups of 12 specimens were prepared, following which the μtensile bond strength was tested in a μtensile universal tester (MultiTest 2.5-I, Mecmesin, UK) at a 2

mm/min crosshead speed. The μtensile bond strength (MPa) was measured by dividing the failure load (N) by the cross-sectional area (mm2) of the specimen.

Statistical analyses

G*Power v3.1.9.4 [13] was used for power calculations. An effect size of 0.811 was calculated based on previous research [8] by adopting an alpha (α) level of 5%, and a beta (β) level of 20% (power of 80%). The expected sample size (n) was 18 (n = 6 per group). The normality of the data distribution was assessed with the Shapiro-Wilk test. Descriptive data are presented as mean and standard deviation (SD). The effects of number of heat pressings and thermocycling on surface hardness, biaxial strength, and μtensile strength were analyzed using two-way ANOVA. Tukey's post hoc test was used to test differences between groups. The significance level was set at $p < 0.05$ for all tests. Data were analyzed using IBM SPSS Statistics v26 (IBM Corp, Armonk, NY, USA).

RESULTS

Effect of heat re-pressing and thermocycling on ceramic composition and microstructure

To assess the effect of heat re-pressing and thermocycling on GC Initial LiSi Press ceramic composition and microstructure, samples were analyzed with EDAX and SEM. The phase separation droplets or peak corresponding to silicon (Si) was present in all samples. While there was a minimal increase in the peak corresponding to the oxide layer (O) in the second press, this disappeared after thermocycling. In general, the EDAX elemental distribution map suggested that there was no change in composition between the first and the second presses. However, in the third press, the phosphorus (P) content had increased (Fig. 1).

SEM images of the first press showed needle-like, interlocked lithium disilicate crystals evenly distributed within the glassy matrix (Fig. 2). The lithium disilicate crystals appeared densely packed with no specific orientation. However, thermocycling resulted in crystals that seemed to be more irregular and appeared fractured. After the second press, the appearance of crystals changed, with the surface changing to a spongy, multi-layered morphology and appearing more compact and smaller in dimension compared with controls. After the third press, crystals in the control group appeared more cracked and broken, and, after thermocycling, crystals rearranged but seemed smaller and less packed (Fig. 2). Measuring grain size (length, width and aspect ratio, in μm) revealed a slight change in size with each press, with the aspect ratios mainly altering based on grain width changes (Table 1).

Table 1
Effect of heat re-pressing and thermocycling on grain microstructure (SEM) dimensions.

		Number of heat presses					
		One		Two		Three	
		L x W (μm)	**AR**	**L x W** (μm)	**AR**	**L x W** (μm)	**AR**
Number of thermocycles	Control	2.73 x 0.8	3.37	2.51 x 0.93	2.69	2.58 x 0.72	3.58
	5000	3.45 x 0.602	5.73	2.9 x 1.2	2.41	2.73 x 0.881	3.09

L: length, W: width, AR: aspect ratio

The effect of heat re-pressing and thermocycling on flexural strength

Neither heat re-pressing nor thermocycling affected the biaxial strength of the tested ceramic. Heat re-pressing two (398.36 ± 44.51) and three (363.79 ± 43.34) times did not affect the biaxial flexural strength of the ceramic compared with the single press (358.70 ± 32.29; $p = 0.074$) (Table 2, Fig. 3A). Additionally, thermocycling (382.06 ± 47.15) had no effect on the biaxial flexural strength of the ceramic compared with the control group (368.79 ± 40.91; $p = 0.384$) (Table 2, Fig. 3B).

Table 2
Effect of heat re-pressing and thermocycling on mechanical properties of the ceramic.

Mechanical Properties	**Number of Heat Presses**			
Mean (SD)	**One**	**Two**	**Three**	***p-value***
Surface Hardness (kgf/mm^2)	565.86 (27.51)^a	612.02 (45.67)^b	597.34 (49.67)^b	< 0.001
Biaxial Flexural Strength (MPa)	358.70 (32.29)	398.36 (44.51)	363.79 (43.34)	0.074
μ**Tensile Strength (MPa)**	16.89 (6.80)	9.19 (4.29)	6.33 (4.39)	< 0.001

Values are mean (SD); p-values from two-way ANOVA. Different superscript letters indicate significant differences between groups (Tukey's post hoc test). SD: standard deviation.

The effect of heat re-pressing and thermocycling on surface hardness

Vickers hardness (HV) was measured (kgf/mm^2) to assess the effect of heat re-pressing and thermocycling on the GC Initial LiSi Press ceramic's surface hardness. Both heat re-pressing and thermocycling had a significant effect on HV, with heat re-pressing two (612.02 ± 45.67) and three

(597.34 ± 49.67) times significantly increasing the surface hardness of the ceramic compared with the single press (565.86 ± 27.51; $p < 0.001$) (Table 2, Fig. 4A). Furthermore, thermocycling (558.17 ± 45.97) significantly decreased the hardness of the ceramic compared with the control group (625.30 ± 34.49; $p < 0.001$) (Table 2, Fig. 4B).

The effect of heat re-pressing and thermocycling on μtensile strength

Both heat re-pressing and thermocycling had a significant effect on the μtensile strength of the ceramic. Heat re-pressing two (9.19 ± 4.29) and three (6.33 ± 4.39) times significantly reduced the μtensile strength of the ceramic compared with the single press (16.89 ± 6.80; $p < 0.001$) (Table 2, Fig. 5A). In addition, thermocycling (8.13 ± 5.48) significantly reduced the μtensile strength of the ceramic compared with the control (13.48 ± 7.16; $p < 0.001$) (Table 2, Fig. 5B).

DISCUSSION

The reuse of residual ceramic buttons following heat re-pressing represents a potentially cost- and material-efficient approach. However, the impact of repeated pressing cycles on the mechanical performance of such ceramics remains insufficiently explored. In the present study, subjecting GC Initial LiSi Press ceramic to two and three pressing cycles resulted in measurable alterations in both microstructure and mechanical properties. Although the magnitude of these changes was modest, they reached statistical significance, leading to rejection of the null hypothesis.

Chemical composition analysis using EDAX revealed minimal changes, with only phosphorus (P) content increasing by a small amount after the third press. Heat re-pressing did not induce additional phases. This increase in phosphorus might have been due to contamination from investing material (IPS PressVEST) onto the surface of the tested ceramic specimen, so only present at the surface and not affecting the ceramic content [14]. However, secondary crystals did appear on EDAX, and their number increased with increasing number of heat presses, consistent with a report of minimal, non-significant changes in EDAX readings [5, 7, 15].

We observed a change in crystal morphology after heat re-pressing in terms of crystal packing and size. Crystals were more densely packed and smaller after heat pressing and were less densely packed after thermocycling. This is different to previous studies, which reported that crystals were less densely packed and larger after heat re-pressing and thermocycling [16, 17]. These differences might be because these two studies tested the same ceramic, IPS e.max Press [16, 17], while we used GC Initial LiSi Press. GC Initial LiSi Press was developed using high-density micronization technology, which enables packing and allows the diffuse dispersion of micro-crystals within the glass matrix, thereby improving the optical and mechanical properties of the material [18].

Furthermore, heat re-pressing and thermocycling had no significant effect on biaxial flexural strength, as reported consistently previously [9, 16, 17]. This observed sustained strength may be due to the densely

packed crystals after repeated heat pressing [19–21]. Alternatively, due to the narrower but more densely packed crystals after repeated heat pressing, this might prevent crack propagation through the residual glass matrix and thus maintain specimen strength [5]. Although heat re-pressing and thermocycling altered the microstructure of high-density micronized (HDM) glass ceramics, these changes might not translate into changes in flexural strength. However, further studies are needed to confirm this observation.

The surface hardness of the tested lithium disilicate ceramic was positively affected by heat re-pressing, in contrast to previous findings. For example, Tang et al. [6] demonstrated that heat re-pressing reduced surface hardness of lithium disilicate ceramics (IPS e.max Press), and AbuHaimed et al. [16] showed that re-pressing had no effect on the same ceramic. Nevertheless, both investigators showed that heat re-pressing reduced the packing and density of the lithium disilicate crystals [6, 16]. However, we report an increase in both crystal density and surface hardness, which may be directly related. Further studies are needed to confirm these observations.

We detected a decrease in μtensile strength of the ceramics after heat re-pressing and thermocycling, which might be due to compaction of the crystal particles following size reduction and a loss of intervening matrix, thereby reducing microporosities. This change might later affect the microporosities induced by hydrofluoric acid etchant, therefore leading to alterations and a reduction in bond strength. Indeed, at the microstructural level, when hydrofluoric acid is applied to glass ceramics, it creates tetrafluorosilane, which in turn reacts with the ceramic to form soluble hydrofluorosilicic acid. This matrix, when washed away, leaves a high-energy, porous, micro-retentive surface topology [22]. This surface becomes more reactive to silane-coupling agents forming chemical bonds, thus enhancing bond strength. When the glass matrix is scarce, this mechanism might be negatively affected, reducing bond strength [23].

However, the opposite was noticed after thermocycling, after which crystals were smaller and more dispersed within the silica matrix. These data suggest that moisture absorption after thermocycling may still have a negative effect on μtensile bond strength. This is consistent with another study indicating that thermocycling significantly reduced μtensile bond strength with either hydrofluoric acid etchant or air-abraded surface treatment of lithium disilicate glass ceramic (IPS e.max Press) [24]. Moreover, the current results are consistent with a study showing that repeated re-pressing of ceramics can minimize the μtensile strength, even after aging [15].

Limitations of this study include its *in vitro* design and restriction to one type of ceramic, and similar experiments with other ceramics could help to establish generalizability. Additionally, samples undergoing μtensile testing were prepared by directly packing and curing composite resin over ceramic samples following adhesive application. This approach does not accurately simulate clinical protocols, where resin cements are typically used, thus limiting the interpretation of these results. Further *in vitro* studies confirming these observations are needed before translation to clinical studies.

CONCLUSIONS

Both heat re-pressing and thermocycling altered the microstructure and mechanical properties of high-density micronized glass ceramics by changing lithium disilicate crystal shape, decreasing crystal size, and altering crystal density within the glass matrix without an effect on biaxial flexural strength. They also altered surface hardness, with heat re-pressing increasing it and thermocycling decreasing it. Further *in vitro* studies are needed to confirm whether the observed effects of heat re-pressing on the mechanical properties of high-density micronized glass ceramics are positive or negative prior to clinical translation.

Abbreviations

ANOVA
analysis of variance
AR
aspect ratio
DSR
Deanship of Scientific Research
EDAX
energy dispersive X-ray analysis
FESEM
field-emission scanning electron microscopy
HDM
high-density micronized
HV
Vickers hardness
ISO
International Organization for Standardization
SD
standard deviation
SEM
scanning electron microscopy

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Competing interests

The authors declare that they have no competing interests.

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Authors' **contributions**

AYI: Writing – original draft, Project administration, Methodology, Funding acquisition, Conceptualization. **NMM:** Writing – original draft. **LMA:** Writing – review & editing. **HYM:** Writing – review & editing. **RAA:** Writing – review & editing, Writing – original draft, Project administration, Funding acquisition, Formal analysis, Data curation, Conceptualization. All authors read and approved the final manuscript.

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Figures

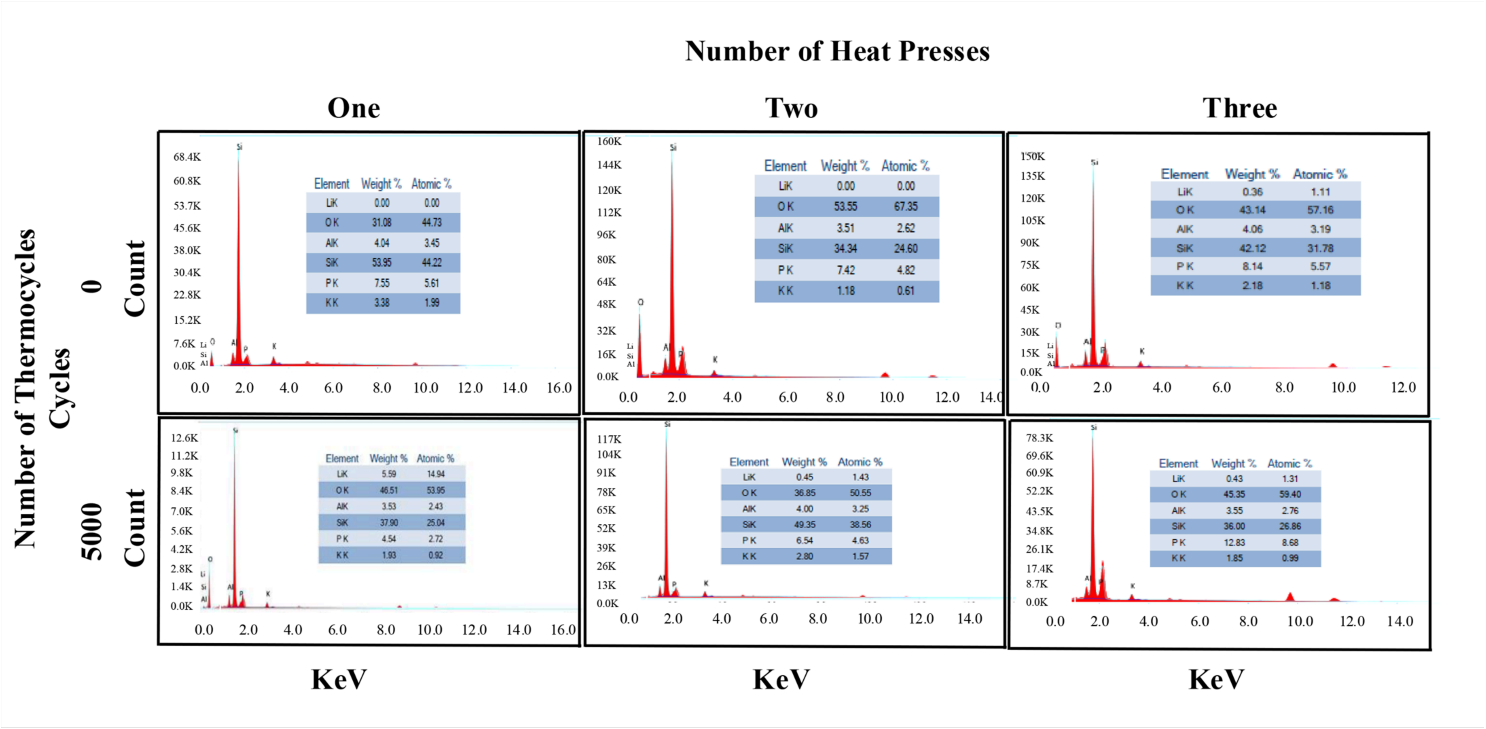


Figure 1

Effect of heat re-pressing and thermocycling on the ceramic crystalline structure (EDAX).

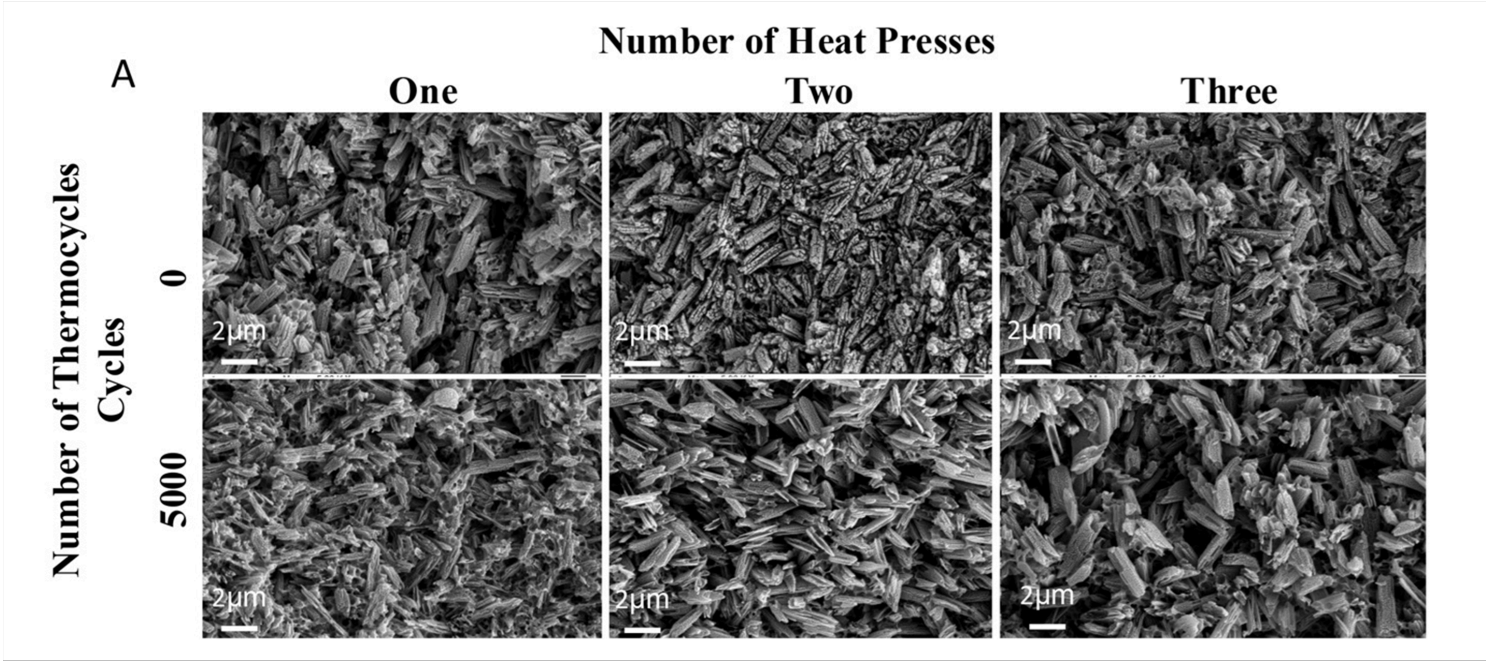


Figure 2

Effect of heat re-pressing and thermocycling on ceramic microstructure (SEM).

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Figure 3

Effect of heat re-pressing and thermocycling on ceramic biaxial flexural strength.

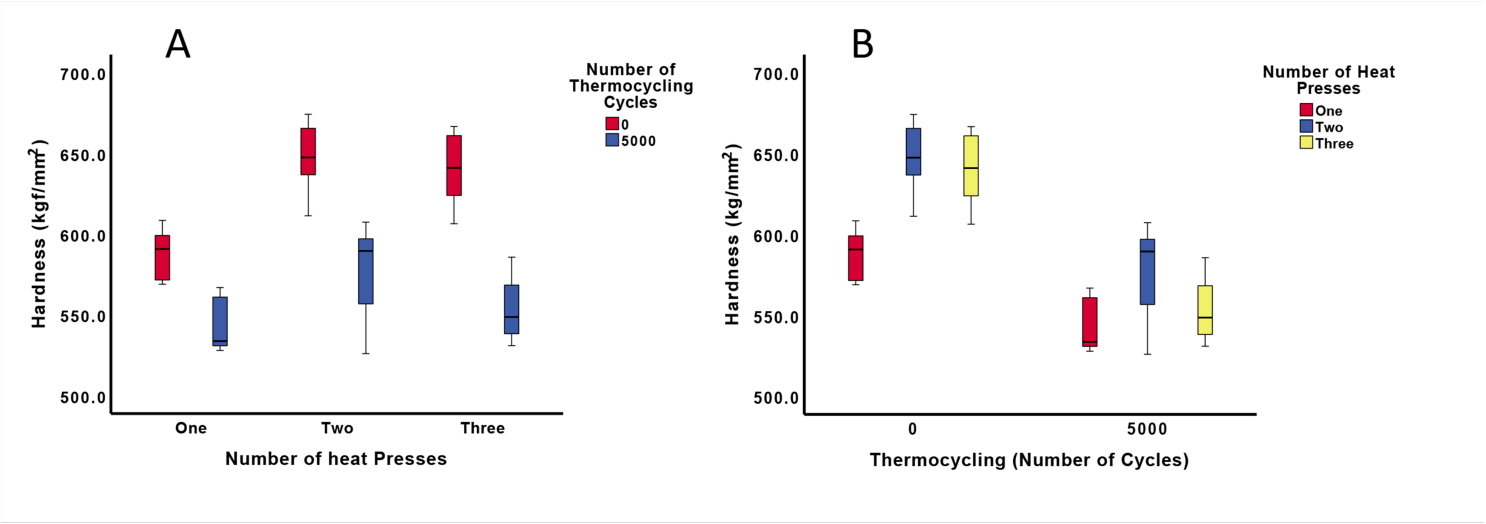


Figure 4

Effect of heat re-pressing and thermocycling on ceramic surface hardness.

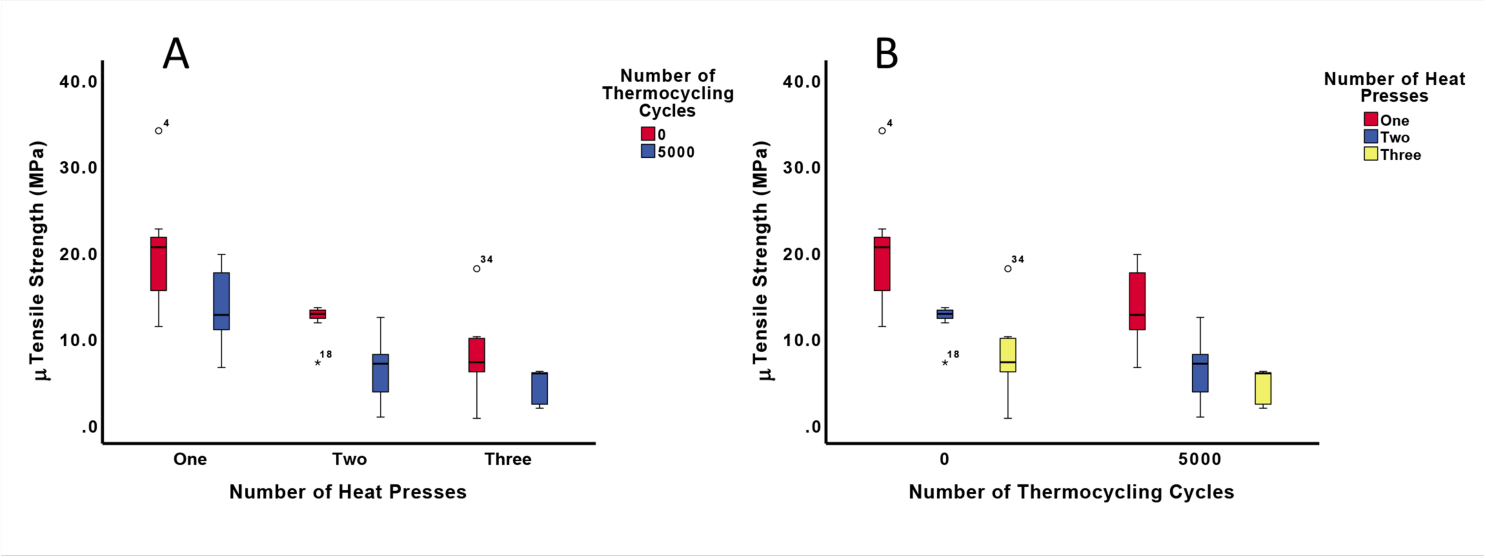


Figure 5

Effect of heat re-pressing and thermocycling on mtensile strength.