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SURFACE ROUGHNESS AND HARDNESS OF TRANSLUCENT ZIRCONIA WITH DIFFERENT POST-SINTERING INTERVENTIONS

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ABSTRACT

Objective: This study was conducted to examine the effect of various post-sintering interventions on the surface characteristics and hardness of translucent zirconia. **Materials and methods:** Thirty-eight zirconia discs were cut from InCoris-TZI blocks. Discs were sintered, cleaned, and divided into four groups according to post-sintering intervention (n=8 in each): Group P: Polished only, Group PG: Polished and Glazed, Group PHT: Polished and Heat treated for one glaze firing cycle, and Group PGHT: Polished, Glazed and Heat treated as the previous group. Each treatment was carried out according to the manufacturer's guidelines. One disc from each group was examined using a Scanning Electron microscope to explore the surface morphology. Surface roughness was assessed using an optical noncontact method. Micro-hardness of the specimens was evaluated using a Vickers diamond indenter. All data were calculated, and statistical analysis was performed. **Results**: There was a significant difference between groups in surface roughness. The PHT samples had the greatest value (0.26±0.01) and the PG samples had the lowest one (0.23±0.03) while there was no significant difference between groups for microhardness. **Conclusions:** Glazing following polishing Translucent zirconia exhibited the best surface smoothness. All post-sintering interventions had no effect on translucent zirconia surface hardness.

KEYWORDS: Glazing, Polishing, Firing, Microhardness, Translucent Zirconia.

INTRODUCTION

All-ceramic materials are increasingly used in dentistry for fixed dental prostheses (FDP) due to the patients' rising aesthetic demands. When compared to other oxide ceramics, zirconia offers the best mechanical qualities among the alternatives. There are three allotropes of the polymorphic zirconia. At room temperature, pure zirconia is monoclinic, and this phase is stable up to 1170 °C. It changes into the tetragonal phase above this point, which is stable up to 2370 °C. On the other hand, the cubic phase is stable up to 2680 °C. Zirconia has a high initial strength and fracture toughness due to its transformation toughening feature, which enhances the resistance to crack propagation. The metastable tetragonal grains undergo a transition into monoclinic ones, their volume increases, and consequently, compressive stresses develop on the zirconia surface [1, 2].

By adding chemical stabilizing oxides, such as 3% mol yttrium-oxide (Y_2O_3) particles, it is necessary to stabilize the strong t-phase at room temperature, resulting in a 3-Yttrium partly stabilized Tetragonal Zirconia Polycrystal (3Y-TZP). To overcome the opaqueness of traditional zirconia and the drawbacks of veneer chipping, the translucent monolithic 3Y-TZP was introduced. Zirconia can become translucent by raising the sintering temperature, lowering alumina, or adding more Y_2O_3 [3, 4].

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Zirconia's hardness and friction resistance have a significant impact on wear behavior. Full contour zirconia restorations offer better hardness, fracture resistance, and structural stability than veneering porcelain [5]. The surface roughness of dental zirconia restorations is one of the factors that affect their long-term clinical success. Surface roughness affects the initial bacterial adhesion and microorganism colonization that can adversely affect the esthetics and had a detrimental effect on antagonist wear [6]. Therefore, before delivering zirconia restorations to patients, dentists must carry out some post-sintering procedures on the restorations. These procedures may involve grinding, adjusting, finishing, polishing, and glazing [7,8].

Multistep polishing processes are advised and frequently utilized because they can provide high-gloss zirconia surfaces that are equivalent to glazed surfaces [9]. However, in locations requiring high esthetics, glazing must be added to the zirconia because the polishing process might reduce its brightness and produce a shade that is dissimilar to the natural teeth [10].

The manufacturers normally recommend only one extrinsic characterization firing cycle. However, additional firings may be necessary before delivery to adjust the shape, color, [11] occlusion, [12] or for glaze application [13]. Therefore, before cementation, ceramic restorations may go through at least two firing cycles. Dental zirconia's mechanical characteristics, such as its hardness, fracture toughness, and microstructure, as well as its crystal organization, have all been documented to be impacted by different grinding and heat treatments [14, 15,16].

However, it is still controversial how various post-sintering procedures affect the surface characterization and hardness of monolithic translucent 3Y-TZP restorations. Therefore, this study was conducted to examine the influence of different post-sintering interventions (Polishing only, Polishing & Glazing, Polishing & Heat Treatment, Polishing, Glazing & Heat Treatment) on the surface morphology, surface roughness, and hardness of translucent zirconia. The first null hypothesis of this study postulated that the post-sintering interventions would not affect the surface roughness and the second one claimed that the same interventions would not affect the hardness of translucent zirconia.

MATERIALS AND METHODS

Ethics approval:

All techniques in this in vitro have been granted permission by the research ethics committee of the faculty of dentistry at Ain Shams University in compliance with its rules and licensing requirements. FDASU-RecPC072337 is the acceptable exemption code by the ethics committee.

Sample size calculation:

A power analysis was designed to have enough power to perform a statistical test of the null hypothesis. The estimated total sample size (n), which corresponds to 8 samples in each group, has been identified to be (32) samples after adopting an alpha (α) level of 0.05 (5%), a beta (β) level of (0.2) (i.e., power=80%), and an effect size (f) of (0.561). Using G Power version 3.1.9.7, sample size analyses were made [17,18].

Zirconia specimens' preparation:

By cutting Translucent Yttrium partially stabilized zirconia blocks inCoris TZI, Sirona Bensheim, Germany) with a diamond disc installed on a Computer Numerical Control (CNC) milling machine, 32 disc-shaped zirconia specimens (12 mm diameter x 2.4 mm thickness) were created. All sectioned specimens were ultrasonically cleaned in a bath of distilled water for 5 minutes.

All specimens were placed into the sintering bowel of the inFire HTC speed furnace (Sirona

Bensheim, Germany) after being finished with a silicon carbide paper of 400 grit. After that, the software began to run automatically. The program duration was 90 minutes. At a sintering temperature of 1540°C, the specimens were sintered. The specimens' dimensions changed after sintering due to a 20% sintering shrinkage (10 mm diameter x 2 mm thickness).

Post-sintering interventions:

Under water coolant, the specimens were finished with ultrafine sandpaper. According to the post-sintering method used, the specimens were allocated into 4 groups (n=8 in each).

Group P: Polished only.

Group PG: Polished and Glazed.

Group PHT: Polished and Heat treated.

Group PGHT: Polished, Glazed, and Heat treated.

Group P (Polished only): The three-step diamond-impregnated silicone polishing system was used to polish one surface of each specimen (Jazz Lab polishing kit, SS White, New Jersey). Polishing was performed by the same and experienced operator using a low-speed straight handpiece and the strokes were done in one direction. Group PG (Polished and Glazed): a Glaze paste (Realism, UPCERA, China) was applied and fired on one surface of each specimen after polishing according to the manufacturer's instructions. One coat of the glaze was applied by the same operator in one direction. Glaze firing parameters are shown in table (1). Group PHT (Polished and Heat-Treated): specimens were polished as previously mentioned and fired for one more glaze firing cycle in a porcelain firing furnace (VITA V60 i-Line, VITA Zahnfabrik, Germany) at 900°C for 1 minute without application of glaze material. Group PGHT: Polished, Glazed then Heat Treated for one glaze firing cycle, without additional glaze application, following the same guidelines previously applied.

TABLE (1) The firing parameters of the glaze cycle.

Stand by temperature °C	Closing time (min)	Heating rate (°C/min)	Firing temperature °C	Holding time (min)
500	4:00	80	900	1:00

Measurements

Surface morphology examination:

Using an environmental scanning electron microscope (Inspect S, FEI company, USA), the morphology of one representative sample from each subgroup was inspected prior to the hardness test. 2000 X magnification images were obtained and explored.

Surface roughness measurement:

The demand for quantitative, non-contact surface topography characterization is typically satisfied by optical approaches. The photos were captured using the following image acquisition system: a digital camera (U500x Digital Microscope, Guangdong, China) mounted vertically at a distance of 2.5 cm from the samples, with a resolution of 3 Mega Pixels. The axis of the lens makes an approximately 90^{o} angle with the sources of illumination.

Eight LED lamps (adjustable by the control wheel) were used to create the illumination, and they had a color index of about 95%. At a fixed magnification of 90X, the images were saved at maximum resolution and connected to a suitable personal computer. Each image was captured with a resolution of 1280 x 1024 pixels. To define and standardize the area of roughness measurement, digital microscope images were resized to 350 x 400 pixels using Microsoft Office Picture Manager. Utilizing WSxM software (Version 5 Develop 4.1, Nanotec, Electronica, SL), the adjusted images were examined [19] .

All confines, sizes, frames, and measured characteristics are expressed in pixels within the WSxM software. System calibration was therefore performed in order to translate the pixels into precise real-world units. To calibrate, a known-size object (in the current case, a ruler) was compared to a scale calculated by the software. A 3D picture of the specimens' surface profile was subsequently produced. For each specimen, three 3D photos were taken in the middle and on either side of the specimen in an area measuring 10 μm x 10 μm. The average of heights (Ra), expressed in μm, which has been suggested to be a credible indicator of surface roughness, was calculated using WSxM software.

Hardness test:

Surface Micro-hardness of the specimens was assessed using a Vickers diamond indenter and a 20X objective lens on a digital display Vickers Micro-hardness Tester (Model HVS-50, Laizhou Huayin Testing Instrument Co., Ltd., China). The specimens' treated surfaces were subjected to a 300g load for 15 seconds. On the surface of each specimen, three indentations were made, evenly spaced around a circle and not more than 0.5 mm apart from one another. Using the capabilities of the built-in scaled microscope, the lengths of the indentations' diagonals were measured, and Vickers values were then transformed into micro-hardness values.

Micro-hardness calculation.

Micro-hardness was calculated using the following equation:

$$HV=1.854\ P/d^2$$

where, **HV** is Vickers hardness in Kgf/mm^2, **P** is the load in Kgf and **d** is the length of the diagonals in mm

RESULTS

1- Statistical analysis:

The mean and standard deviation (SD) data have been utilized for illustrating numerical data. To check for normalcy, the Shapiro-Wilk test was carried out. Using Levene's test, the homogeneity of variances was assessed. The homogeneity condition had not been violated and the data were normally distributed. One-way ANOVA was used for analyzing intergroup comparisons, afterwards, Tukey's post hoc analysis was performed. Spearman's rank order correlation coefficient was used to analyze correlation. For all tests, the significance level was set at $p<0.05$. R statistical analysis software for Windows (20), version 4.3.1, was used to conduct the statistical analysis.

a. Surface roughness:

Statistical results indicated that the difference of surface roughness was statistically significant (p=0.003). The PHT samples had the greatest value (0.26±0.01), followed by the P samples (0.25±0.01), the PGHT samples (0.24±0.03), and the PG samples, which had the lowest value (0.23±0.03) Fig (1).

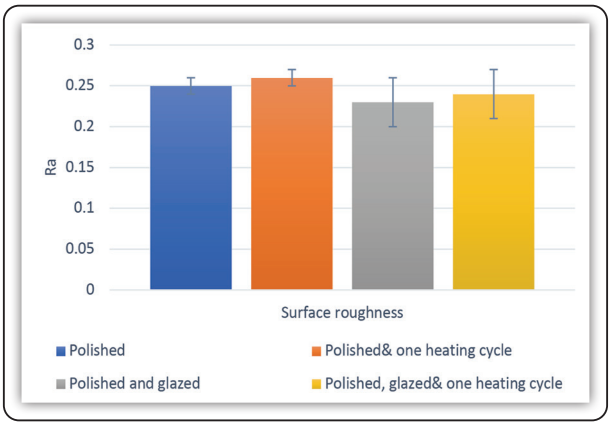

FIG (1) Bar chart showing mean and standard deviation values (error bars) of surface roughness (Ra)

b- Microhardness:

Statistical analysis showed that there was no significant difference in micro-hardness across the groups (p=0.200). The greatest value was discovered in PG samples (808.21±1.09), followed by PHT samples (806.97±2.36), then P samples (806.79±2.41), while the lowest value was discovered in PGHT samples (806.72±2.43). Fig (2) Micro-hardness and roughness values were not significantly correlated (rs=-0.053, p=0.689). Figure (3) shows a scatter plot of the correlation between the two examined variables.

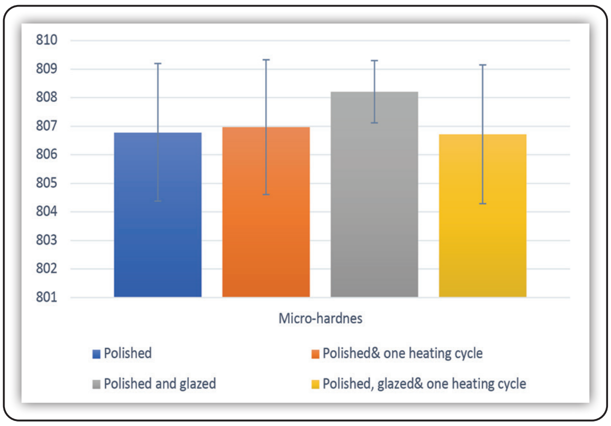

FIG (2) Bar chart showing mean and standard deviation values (error bars) of micro-hardness

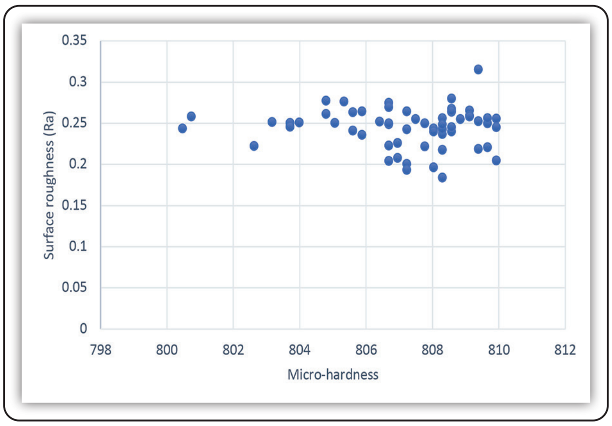

FIG (3) Scatter plot showing the correlation between surface roughness and micro-hardness

2- Surface characterization:

a) Scanning Electron microscope examination (SEM):

The SEM photographs of the polished specimen (P) showed numerous striations and grooves running in one direction. Also, many micropores were observed. The grooves became more prominent and some irregularities were noticed following heat treatment for one firing cycle (PHT) as seen in Fig (4P and 4PT). For Polished and Glazed (PG), the majority of the surface was covered with glaze material, making scratches less noticeable. Numerous locations revealed crystals with irregular forms. The polished and glazed specimen had a lot of inconsistencies after one firing cycle (PGHT), certain sections weren't coated with glaze, and the size of the crystals enlarged as shown in Fig (4PG and 4PGHT).

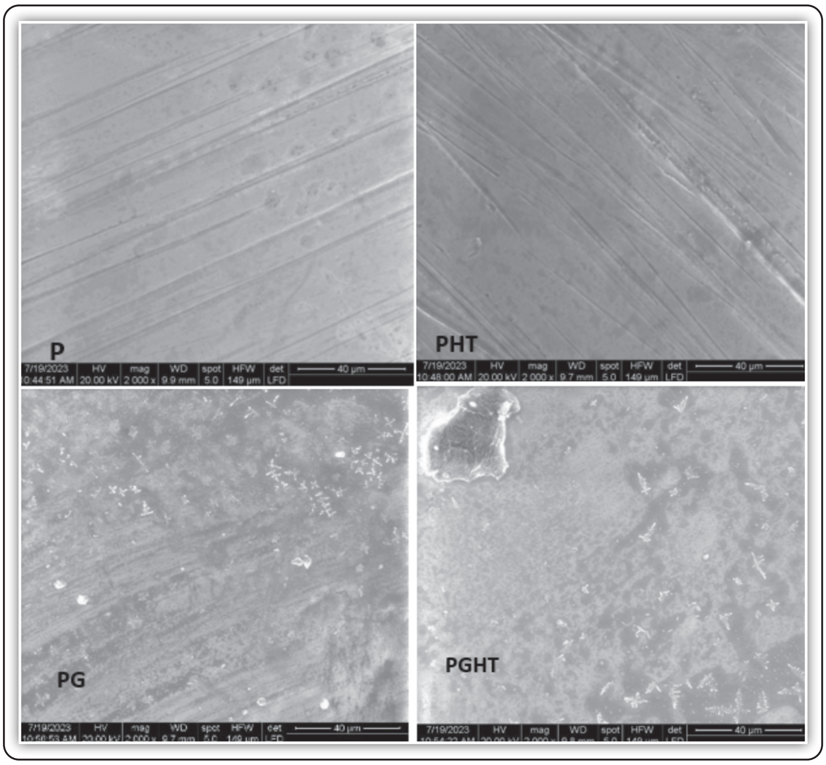

FIG (4) SEM photos of Polished (P), Polished and Heat Treated (PHT), Polished and Glazed (PG), and Polished, Glazed and Heat Treated (PGHT).

b) Surface roughness examination:

Regarding the topographical characteristics of the surface, it was observed in 3D images that the surfaces of both P and PHT specimens showed increased surface roughness as well as micro irregularities with deeper and narrower valleys and pointed peaks that were larger in number and pointed when compared to the surfaces of PG and PGHT as shown in Figs (5&6).

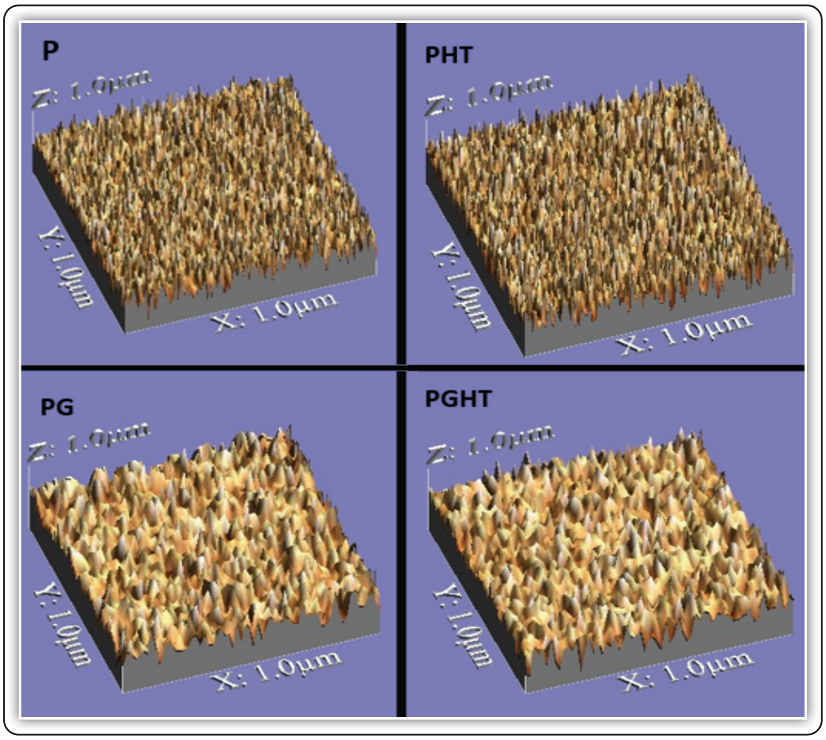

FIG (5) 3D images of surface topography.

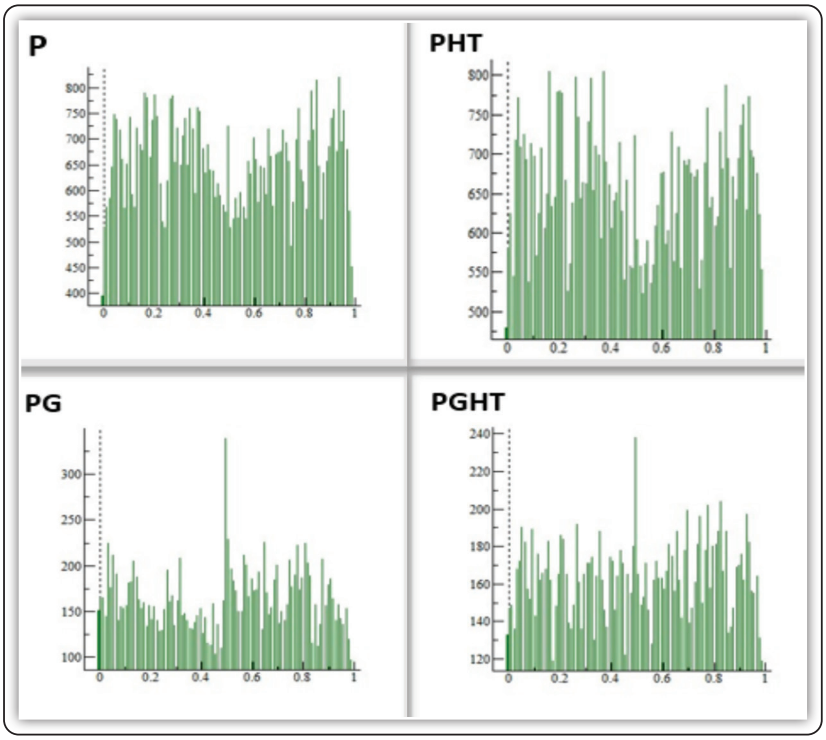

FIG (6) Histogram of the surface roughness.

DISCUSSION

Due to their outstanding mechanical and biological characteristics, translucent Yttria-stabilized tetragonal zirconia polycrystals (Y-TZPs) have become preferred materials in dental fixed prostheses [1,2, 3]. To achieve a uniform smooth surface that minimizes the potential for bacterial plaque accumulation and wears off the opponent's natural teeth, post-sintering alterations of the restoration through finishing, and polishing procedures are crucial. Prior to cementation, clinical modifications to the restoration may be performed during the evaluation phase [9,21]. Therefore, this in vitro study was performed to assess the influence of several post-sintering treatments (polishing only, polishing & glazing, polishing & heat treatment, polishing, glazing & heat treatment) on the surface characteristics, and microhardness of translucent zirconia.

As introduced by Abouelatta, [22] optical non-contact techniques are very useful for the 3D assessment of sensitive and complicated surfaces and the surface roughness measurement in comparison to stylus instruments. This study indicated that post-sintering interventions influenced the surface roughness of translucent 3 YTZP discs and therefore, the first null hypothesis was rejected. The PHT samples had the greatest value (0.26±0.01), followed by the P samples (0.25±0.01). This can be attributed to the findings in our SEM images (Fig 1P) where Polishing created many striations, grooves, and micropores on the zirconia surface. This was consistent with the findings of Cakmak et al [14]. These surface defects hadn't been resolved by heat treatment as demonstrated in our SEM photo (Fig 1PHT): it was observed that grooves created by polishing became more evident, and more irregularities were noticed after one cycle of heat treatment creating more surface roughness. This can be accounted for the Low-Temperature Degradation (LTD) that takes place in zirconia under thermal and mechanical stress, where the tetragonal phase changes into a monoclinic one. The process of transformation begins to move quickly with the uplift of some grains, pushing them to the surface and causing micro-fissures that will open the property for water penetration on the long term beneath the surface of monolithic zirconia, spreading the t-m changing to the interior of the restoration, and ultimately, it will cause ceramic to deteriorate and produce bigger fissures [16, 23].

For Polished and Glazed zirconia samples (PG), surface roughness was the lowest and this may be due to the healing effect of the glaze material fused on the surface that obliterated all grooves and micropores as demonstrated in our SEM micrograph (Fig.1PG). After being exposed to an additional firing cycle the polished and Glazed zirconia (PGHT) demonstrated higher surface roughness (0.24±0.03) which can be explained by the evident large crystals and inconsistencies found on the surface which supported the findings of ElSherif et al [15] who found that LTD of aged zirconia samples resulted in clear crystalline structures on the surface and increased surface roughness. This may be caused by heat from the glaze firing [9].

The second null hypothesis was accepted because the microhardness data in our investigation showed that all post-sintering interventions had no effect. These findings aligned with those of De Souza et al. [24] who utilized artificial aging to explore the impact of aging on the surface morphology and hardness of yttria tetragonal zirconia polycrystal (Y-TZP). The results of our current study are supported by the conclusion that grain pull-out accompanied an increase in surface roughness. There was no difference in hardness, which might be explained by the fact that aging-related t-m phase conversion not only causes a main deformity but also causes the grain size to increase. This process creates a localized pressure stress on the surface that forces a potentially expanding split to close in order to maintain its mechanical balance. This may lead to an increase in mechanical features.

Another justification comes from Moqbel et al.[25] who examined the Vickers hardness of translucent dental zirconia after autoclave aging and discovered no impact on hardness. The cause of this could be a consequence of the t-m phase's transition percentage was insufficient to cause surface deterioration, leaving the hardness values unaltered from before aging [26].

However, ElSherif et al. [15] made it to the conclusion that various aging processes led to a decrease in surface microhardness and an increase in surface roughness. They attributed these to phase change and zirconia LTD. The development of the monoclinic stage in zirconia hydrothermal aging has two main effects: an increase in pressure stress, which elevates mechanical features, and the emergence of disorders which lowers these characteristics [27]. Therefore, depending on the effectiveness of each impact, a material's mechanical properties may increase, decrease, or remain unchanged [15].

Based on the findings of our study, it is recommended to add glaze layer after polishing on translucent zirconia for better surface finish, smoothness, and long-term durability. More investigations on the effect of more firing cycles and hydrothermal aging for glazed zirconia are suggested together with Xray diffraction to investigate more the existing phases.

CONCLUSIONS

1. Glazing following polishing Translucent zirconia exhibited the best surface smoothness.
2. All post sintering interventions carried out in this study had no effect on translucent zirconia surface hardness.

Clinical recommendations:

Even though full contour zirconia restorations that have been corrected require finishing and polishing, glazing provides superior cosmetic and biological results.

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