10.21608/adjc.2023.225887.1372 Advanced Dental Journal
Volume 6 (2024) | Issue 2| Pages 216 - 225

**Original Article**

# Effect of Thermocycling on Optical Properties and Surface Roughness of Different Heat-Pressed Glass Ceramics

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**Submitted:** 29-7-2023
**Accepted:** 17-8-2023

## Abstract

**Aim:** The aim of this study was to evaluate the impact of thermocycling on the optical properties and surface roughness of conventional lithium disilicate and zirconia reinforced lithium disilicate heat-pressed glass ceramics.
**Subjects and methods:** Eighteen samples (a diameter of 12 mm and 1.5 mm thickness) were fabricated from three types of heat-pressed ceramic: a conventional lithium disilicate heat-pressed glass-ceramic (IPS e.max Press, Ivoclar Vivadent, Germany) (n=6), and 2 heat-pressed zirconia reinforced lithium silicate press glass-ceramic, Celtra Press(Dentsply, Germany) (n=6) and Ambria Press (Vita-Zahnfabrik, Germany) (n=6). Specimen were finished and polished carefully and then glazed according to the manufacturers' instructions. The discs were assessed for their translucency, color change and surface roughness before and after thermocycling using distilled water with baths of 5 and 55 ◦C, for 10,000 cycles. Results were analyzed using one-way ANOVA ($\alpha$=0.05).
**Results:** For all pressed ceramics, thermocycling resulted in significant decrease in the translucency parameter ($p<0.001$). Vita Ambria and IPS e.max Press showed the highest significant $\Delta L$ compared to Celtra Press ($p<0.001$). For the $\Delta E$, Vita Ambria showed the highest $\Delta E$ values compared to Celtra Press and IPS e.max Press. For all pressed ceramics, a significant increase in surface roughness resulted after thermocycling at $p<0.001$.
**Conclusion:** Vita Ambria Pressed ceramic is significantly more translucent than all the other tested groups before and after thermocycling. The color and translucency of tested ceramics were affected by thermocycling but still considered clinically accepted. Thermocycling significantly increased the surface roughness of all the tested ceramic materials.

**Keywords:** pressable ceramics, thermal aging, translucency, color change.

## I. INTRODUCTION

Lithium disilicate-based glass ceramics (LDC) have been one the essential materials in the field of esthetic and restorative dentistry over the past 20 years. Thanks to their aesthetics, high strength, chemical, and processing properties, making them very well-liked by dental technicians and dentists. These advantages allow them to be used for a wide range of indications. The applications of LDC can range from single tooth restorations up to complete mouth rehabilitations employing inlays, onlays, veneers, and anterior/posterior crowns, as well as tooth replacement with fixed partial dentures up to the second premolars.(Höland and Beall, 2020)

In LDC, the high strength (400–610 MPa) and toughness (2.3–2.9 MPa) of the material are based on the high content of crystalline phase (60–70 vol.%) which makes crystal interlocking within the microstructure and a, generated by the lath-like Li2Si2O5 crystals.(Silva et al., 2017)

Glass-ceramics were further developed into glass-ceramics reinforced with polycrystalline ceramics. The predominant crystalline phase of these new glass-ceramics is the lithium silicate in a vitreous matrix reinforced with zirconium dioxide crystals (~10%). The lithium silicate crystals are up to six times smaller than the lithium disilicate crystals found in lithium disilicate glass-ceramics, ranging in size from 500 to 1000 nm. As an additive, zirconia particles affect crystallization by hindering crystal growth. The inner structure containing smaller crystals leads to excellent mechanical and optical properties and induces good surface finishing.(Rinke et al., 2016; Traini et al., 2016)

Compared to CAD/CAM, pressed ceramics can result in higher precision, which is challenging to achieve with other methods. Pressing has also developed into a future technology with potential by creating new, advanced materials and manufacturing techniques.(Höland and Beall, 2020)

The optical properties can be affected by a number of factors, including the ceramic thickness, manufacturing method, composition, inner structure, crystalline content, grain size, pores, additives, surface roughness, and topography.(Akar et al., 2014; Sen and Us, 2018) Translucency, in addition to color, is an essential consideration during the functional phase of restorations since it influences the aesthetic result.(Della Bona, Nogueira and Pecho, 2014) It is challenging to quantify this parameter and correlate it with intrinsic and external factors to follow the evolution of optical properties and the opportunities to take action for their preservation. The translucency parameter (TP) can be used to determine the relative translucency of ceramics.(Kim et al., 2016)

Glazing is typically used to smooth the surface of ceramic materials, although for new types of micro- and nanostructured ceramics, mechanical polishing without a firing process can also be recommended. After finishing, the surface becomes smoother, which helps the ceramic's biocompatibility, reduces the accumulation of plaque, excessive tooth wear, and stain susceptibility, and improves the restoration's long-term aesthetics by making it appear more natural.(Kilinc and Turgut, 2018) It is well-established that surface roughness significantly affects how restorations perform clinically. The surface area for a material-environment interaction is increased by roughness.(Porojan et al., 2017)

The clinical success of dental restoration is determined by its longevity, which is greatly impacted by the oral environment. In vitro research can mimic the oral environment to study how restorations behave, to produce trustworthy restorations. Thermal cycling and artificial aging tests that imitate the oral environment should be implemented to appropriately estimate the long-term durability of dental restorative materials in the clinical environment.(Hampe et al., 2018)

Therefore, the aim of this study was to evaluate the optical properties and surface roughness of two zirconia-reinforced glass ceramics before and after thermos-cyclic aging. The null hypotheses were, 1. Optical properties of different pressed ceramics will not be affected by thermocycling. 2. Surface roughness of different pressed ceramics will not be affected by thermocycling.

## II. SUBJECTS AND METHODS

### Samples preparation

A disc shape, with a diameter of 12 mm and thickness of 1.5mm was designed with the aid of 3D Builder software version 20.0.3.0 (Microsoft Corporation, USA) and was saved as an STL file. The STL file was imported to a computer assisted designing software version 2017. (Exocad GmbH, Darmstadt, Germany) and a total of 18 standardized discs were milled from ProArt CAD Wax (Ivoclar Vivadent, Ellwangen, Germany) using a VHF CAM 5-S1 IMPRESSION 5-axis milling machine (VHF International, Ammerbuch, Germany). The dimensions of all discs were verified using a digital caliper and any defective discs were discarded.

Three different types of heat-pressed glass-ceramic (n=18) were obtained by heat-pressing. One lithium disilicate heat-pressed glass-ceramic (IPS e.max Press, Ivoclar Vivadent, Ellwangen, Germany) (n=6), and two heat-pressed zirconia reinforced lithium disilicate press; Celtra Press, Dentsply, Hanau, Germany (n=6) and Ambria Press, Vita-Zahnfabrik, Postfach, Germany (n=6). The compositions of the tested materials are presented in Table 1.

Each specimen was heat-pressed following the manufacturer instructions for each used material. The parameters for pressing the different types of ceramics are mentioned in Table 2. The pressed discs were retrieved from their corresponding invested rings by divesting the investment by marking the length of the pressing plunger by a pencil on the invested rings using a disc mounted on a straight hand piece. The investment around the discs were removed using sandblasting with alumina oxide particles Al2O3 (50 µm) at a pressure of 4 bars carefully to avoid damage to the retrieved discs. The retrieved discs were visually inspected and were measured with a digital caliper once again to ensure that there was no deviation from the designed STL file. All discs were finished and polished carefully and then glazed according to the manufacturers' instructions and each group was stored in a separate labeled vial containing distilled water until further usage.

**Table 1.** Materials compositions

| Material | Composition | Manufacturer | Translucent/Shade |
|---|---|---|---|
| IPS e.max Press (heat-presse lithium disilicate ceramic) | lithium disilicate crystals (approx. 70%), $Li_2Si_2O_5$ crystals measure 3 to 6 µm ir length. | Ivoclar Vivadent Ellwangen, Germany | HT/A2 |
| Celtra Press (zirconia reinforced lithium silicate glass-ceramic) | a glass matrix and lithium disilicate crystals 1.5 µm + nanoscale zirconia oxide, 10% $ZrO_2$ | Dentsply, Hanau, Germany | MT/A2 |
| Vita Ambria (zirconia reinforced lithium silicate glass-ceramic) | a glass matrix and lithium disilicate crystals + zirconia oxide, 8-12% $ZrO_2$ | Vita-Zahnfabrik, Postfach, German | HT/A2 |

**Table 2.** Pressing parameters

| | IPS e.max Press | Celtra Press | Ambria |
|---|---|---|---|
| Starting Temperature | 700 °C | 700 °C | 700 °C |
| Hold Time | 29 min | 30 min | 30 min |
| Press Time | 1 min | 3 min | 3 min |
| Heat Rate | 60 °C/min | 40 °C/min | 50 °C/min |
| Press Temperature | 915 °C | 860 °C | 890 °C |
| Press Pressure | 3 bar | 3 bar | 3 bar |
| Vacuum | On | On | On |

**Optical properties measurements**

The color parameters were measured (baseline) using a reflective spectrophotometer (UV- Shimadzu 3101 PC, Japan). The aperture size was set to 4 mm and the discs were positioned in the center of the measuring port. A white background (Commission internationale de l'eclairage (CIE) L* = 88.81, a* = −4.98, b* = 6.09) was used and the measurements were made according to the CIE L*a*b* color space relative to the CIE standard illuminant D65, where L* refers to the degree of lightness (0–100), a* to the color coordinate on the red/green axis and b* to the color coordinate on the yellow/blue axis. A total of three measurements were taken for each disc and an average reading was recorded.

For translucency, the same spectrophotometer was used and the discs were measured against white (CIE L* = 88.81, a* = −4.98, b* = 6.09) and black (CIE L* = 7.61, a* = 0.45, b* = 2.42) backgrounds relative to the CIE standard illuminant D65.The translucency parameter (TP) values were obtained by calculating the difference in the color of the specimens against black and white backgrounds using the following formula:

TP= [(L*b − L*w)2 + (a*b − a*w)2 + (b*b − b*w)2]1/2

where:

TP – translucency parameter;

L* – degree of lightness;

a* – color coordinate on the red/green axis;

b* – color coordinate on the yellow/blue axis;

the subscripts b and w refer to the color coordinates against black and white backgrounds, respectively.

All discs were subjected to thermocycling (SD Mechatronik Thermocycler, Germany) to induce thermal stresses within the ceramic materials. The discs were immersed in distilled water bath of 5 °C and 55 °C with a dwell time of 20 seconds and a lag time of 10 seconds for 10,000 cycles.

The process of color parameters and translucency measurement were repeated, and the measurements were recorded after thermocycling. The color change (ΔE) of each specimen was calculated using the following formula:

ΔE = [(ΔL*)2 + (Δa*)2 + (Δb*)2]1/2

where:

ΔE – change in color;

ΔL* = L*after thermocycling − L*baseline;

Δa* = a*after thermocycling − a*baseline;

Δb* = b*after thermocycling − b*baseline.

The ΔE values greater than 1.2 were considered perceptible, whereas values greater than 2.7 were considered clinically unacceptable, according to the 50:50% threshold.(Paravina et al., 2015)

### Surface roughness measurements

Surface roughness was measured on the glazed side of the discs using a contact profilometer (SJ-210 surface roughness tester, Mitutoyo, Japan) with a diamond stylus tip of a 2 µm. Seven random measurements were taken. Values Parameters Ra (µm) average surface roughness was recorded at baseline. The measuring distance was 4 mm and speed of 0.5 mm/s with 0.75 mN force. The measurements were repeated after thermocycling.

### Scanning electron microscope (SEM)

An environmental Scanning Electron Microscope (FESEM QUANTA FEG 250, Netherland) was used to demonstrate the microstructure of each tested ceramic material before and after thermocycling. A magnification of 1,000x was utilized in this study.

### Statistical analysis

Sample size was estimated based on the data extracted from a previous study.(Hallmann et al., 2019) For ΔE evaluation; a large effect size (F=1.1) was detected and a total of 18 specimen (n=6 for each group) will be sufficient to have a power of 95% with alpha=0.05 (R foundation of statistics (version 4.0)). Data checked for normality using Kolmogorov-Smirnov test and showed normal distribution. A dependent t-test was used to compare between before and after thermocycling for translucency and surface roughness. One Way ANOVA used to compare between tested groups for the change in color parameters after thermocycling followed by Tukey's HSD test for pairwise comparisons. A significant level was set at 0.05 (IBM SPSS, Ver. 23, Armonk, NY, USA).

## *III. RESULTS*

### Optical properties

Results of translucency are presented in Table 3. For all pressed ceramics, thermocycling resulted in significant decrease in the translucency parameter ($p<0.001$).

**Table 3.** Translucency parameter for different tested pressed ceramics

| Translucency | Before thermocycling | | After thermocycling | | p-value |
|---|---|---|---|---|---|
| | Mean | SD | Mean | SD | |
| Vita Ambria | 19.00[a] | 0.18 | 17.67[a] | 0.18 | <0.001* |
| Celtra Press | 14.83[c] | 0.16 | 13.76[c] | 0.15 | <0.001* |
| IPS e.max Press | 16.53[b] | 0.21 | 15.70[b] | 0.16 | <0.001* |
| P-value | <0.001* | | <0.001* | | |

Different letters within each column indicate significant difference.

Color parameters are presented in Table 4. Ambria and IPS e.max Press showed the highest significant ΔL compared to Celtra Press (p<0.001). For Δa, significant difference between the tested groups at p<0.001. Ambria showed the highest significant Δa followed by IPS e.max Press followed by Celtra Press. For Δb, Ambria and Celtra Press showed insignificant difference between each other, and both showed significantly higher Δb compared to IPS e.max Press (Fig. 1). For the ΔE, Ambria showed the highest ΔE values compared to Celtra Press and IPS e.max Press.

**Table 4.** Color parameters for different tested pressed ceramics

| | ΔL | | Δa | | Δb | | ΔE | |
|---|---|---|---|---|---|---|---|---|
| | Mean | SD | Mean | SD | Mean | SD | Mean | SD |
| Vita Ambria | -1.67[a] | 0.41 | 1.07[a] | 0.09 | 2.88[a] | 0.15 | 3.52[a] | 0.25 |
| Celtra Press | -0.77[b] | 0.08 | 0.37[c] | 0.02 | 2.77[a] | 0.19 | 2.9[b] | 0.17 |
| IPS e.max Press | -1.3[a] | 0.21 | 0.68[b] | 0.06 | -2.07[b] | 0.26 | 2.54[b] | 0.27 |
| p-value | <0.001* | | <0.001* | | <0.001* | | <0.001* | |

Different letters within each column indicate significant difference.

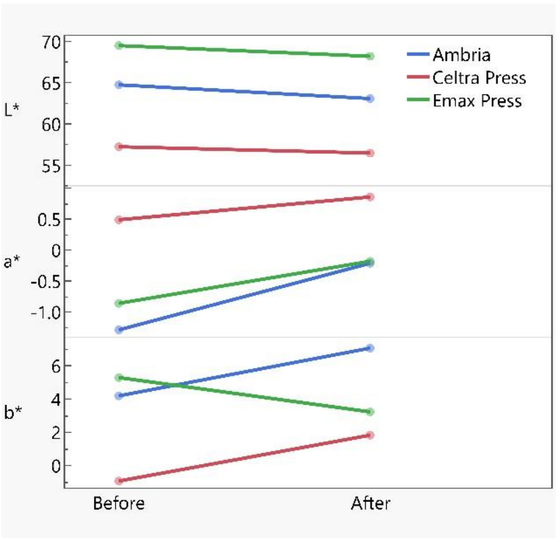


**Figure 1.** Color parameters for different tested pressed ceramics before and after thermocycling

## Surface roughness

Surface roughness results presented inTable 5. For all pressed ceramics, a significant increase in surface roughness resulted after thermocycling for all tested groups at p<0.001.

**Table 5.** Surface roughness (Ra) for different tested pressed ceramics

| Ra | Before thermocycling | | After thermocycling | | p-value |
|---|---|---|---|---|---|
| | Mean | SD | Mean | SD | |
| Vita Ambria | $0.32^{a}$ | 0.06 | $0.81^{a}$ | 0.06 | <0.001* |
| Celtra Press | $0.21^{b}$ | 0.05 | $0.62^{b}$ | 0.05 | <0.001* |
| IPS e.max Press | $0.22^{b}$ | 0.07 | $0.6^{b}$ | 0.11 | <0.001* |
| p-value | 0.018 | | 0.002 | | |

Different letters within each column indicate significant difference

**SEM**

Results of SEM observations before and after thermocycling were presented in figure 2. All samples from all the tested materials displayed defects following thermocycling.

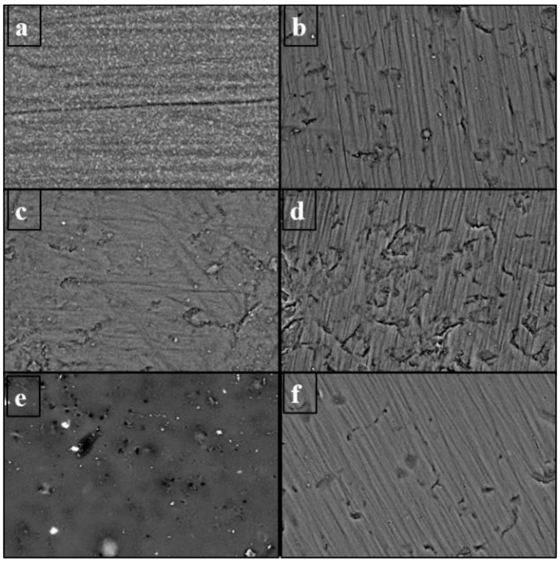


**Figure 2.** SEM micrographs (a) IPS e.max Press before and (b) IPS e.max Press after thermocycling, (c) Vita Ambria before and (d) Vita Ambria after thermocycling, (e) Celtra Press before and (f) Celtra Press After thermocycling, 1,000x

## *IV. DISCUSSION*

This in-vitro study was designed to compare the optical properties and surface roughness of conventional lithium disilicate heat-pressed glass-ceramic and two zirconia reinforced lithium disilicate heat-pressed glass ceramics before and after thermocycling. Both null hypotheses were rejected as thermocycling affected both optical properties and surface roughness.

Lithium disilicate (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) is a particle-filled glass ceramic system that has shown to have improved flexural strength as well as good optical properties. These mechanical properties have made it possible to create monolithic posterior restorations as well as bi-layered anterior crowns.(Guess et al., 2011)

IPS e.max Press is composed of 3 to 6 µm-long needle-like lithium disilicate crystals in a volume fraction of about 70%. Thanks to these elongated crystals which give the pressable ceramic its high flexural strength of 400 MPa.(Silva et al., 2017)

Zirconia-reinforced lithium disilicates, Celtra Press (Dentsply Sirona) and Vita Ambria Press (Vita-Zahnfabrik) have recently been developed, that claimed to combine the superior mechanical properties of yttria-stabilized zirconia (Y-TZP) with the translucency of lithium disilicate.(Höland and Beall, 2020)

In dentistry, optical properties are crucial to both patient pleasure and treatment success. Translucency of restorative materials, in addition to color, is an important component in aesthetic restoration.(Ilie and Stawarczyk, 2015)Aesthetic restorative materials can be evaluated for their translucency and opacity using one of two methods: absolute translucency, which is determined by the direct transmission of light, or relative translucency, which is determined by the translucency parameter (TP).(Della Bona, Nogueira and Pecho, 2014)

The TP is defined as the color difference determined from the L*a*b* values, between a uniform thickness of a material over a white and black backing. If the material is completely opaque, the TP value is zero. As the TP value increases, the translucency of the material also increases.(Brainard and Maloney, 2011; Hamza et al., 2017)

A 1.5 mm thick samples were chosen because most ceramics require a 1.5 mm reduction for tooth structure restoration. Values for the translucency for the human structures range between 15 and 19.(Yu, Ahn and Lee, 2009) The translucency of restorative materials ranges from 9 to 19, at a thickness of 1 mm after other studies can reach 25.(Wang, Takahashi and Iwasaki, 2013; Awad et al., 2015; Wang, Yu and Chen, 2020)

The ratio of crystalline to glass phases, the difference in refractive indices between these phases, the morphology of crystals, grain boundaries, pores, second-phase components, additives, and light scattering from the surface are just a few of the variables that affect the translucency of dental materials.(Kim, Paravina and Chen, 2007; Kim et al., 2016)

Thermocycling is a helpful method for accelerating the artificial aging as it simulates the temperature in the oral environment.(Morresi et al., 2014; Kelly et al., 2017) Based on the assumption that dental restorations experience 20 temperature variations each day, 10,000 cycles are roughly similar to a year in the oral environment.(Morresi et al., 2015; Alp et al., 2018)

In the present study, the results indicated that there was significant difference among the tested materials in terms of translucency before and after thermocycling. Vita Ambria showed the highest translucency before and after thermocycling followed by IPS e.max press. meanwhile, Celtra Press showed the lowest translucency before and after thermocycling. The effect of thermal cycling on the optical properties could be attributed to the increase of the crystal size, the orientation of the crystals, and may be to the change of the glass matrix.(Harianawala et al., 2014; Elsaka and Elnaghy, 2016)

This was in accordance with other studies, who reported the significant decrease in the translucency parameter of ceramics materials with artificial aging.(Porojan et al., 2020; Vasiliu et al., 2020)

Porojan L. et al (Porojan et al., 2020), evaluated artificial aging behavior on six tested monolithic restoration materials (milled lithium disilicate glass-ceramic MT, hot-pressed lithium disilicate glass-ceramic MT and HT, milled zirconia-reinforced lithium silicate ceramic MT and hot-pressed zirconia-reinforced lithium silicate ceramic MT and HT) and found that artificial aging decreased translucency significantly for various lithium silicates ceramics.

Similarly, Vasiliu R. et al (Vasiliu et al., 2020), assessed the effect of thermocycling on translucency of heat-pressed and milled glass-ceramic. They found significant differences between the translucency parameter before and after thermocycling.

On the other hand, the results of the current study were in disagreement with Aljanobi and Al-Sowygh (Aljanobi and Al-Sowygh, 2020), who investigated the effect of thermocycling on two CAD/CAM glass ceramics; IPS e.max CAD HT and Vita Suprinity HT (a zirconia reinforced glass-ceramics) and found that there is a statistically significant increase in their TP at different intervals.

Regarding the Color parameters in the present study, Vita Ambria showed the highest ΔE values when compared to Celtra Press and IPS e.max Press.

The values of color change found in this research vary from 2.54 to 3.52. It is suggested that a color difference is perceptible when it can be detected by the human eye and acceptable when it is tolerable.(Vichi et al., 2011) Considering a threshold of perceptibility of ΔE=1 and a threshold of acceptability of ΔE=3.7 as concluded in a review by Khashayar et al (Khashayar et al., 2014), the color change values following thermocycling in the current study is regarded perceptible but clinically acceptable for all specimens.

In general, color change may vary with aging and may be attributed to either the ceramic material itself and/or the underlying luting agent. Since no cement was used in the current study, it may be as a result of loss of glaze uniformity, increase in surface roughness, occurrence of surface cracks and change in translucency parameters.(Ashy et al., 2021) This was in agreement with the outcomes of translucency parameters and SEM analysis of the current study.

Ceramic glazes are formed of a powder and liquid mixture that are burned onto the ceramic surfaces, giving them a glossy finish. The pores on the surface of the fired ceramic are sealed in the lab by glazing the ceramic restoration.(al-Wahadni and Martin, 1998) By closing the pores that emerge on the surface of ceramics, a glazed ceramic surface minimizes roughness and the potential for abrasiveness and is believed to increase the strength of ceramic (Wright et al., 2004). This might have an impact on the optical properties in the current study in addition to the induced stresses from thermocycling, which might increase the pores and defect size.

Vita Ambria showed the highest and significant surface roughness values before and after thermocycling. The outcomes of the current study exhibited that there was insignificant difference in the surface roughness among the Celtra Press and IPS e.max Press either before or after thermocycling.

Thermocycling resulted in an increase in surface roughness for all tested groups as confirmed by the SEM micrographs. The glaze layer did not maintain the uniform aspect and several deep and well-defined defects appeared after thermocycling. These findings were in harmony with previous study (Vasiliu et al., 2020) that showed the remarkable effect of thermocycling. They found that 10,000 thermocycles had a substantial impact on the surface roughness of the e.max Press lithium disilicate material.

## *V. CONCLUSION*

Within the limitations of the current study and based on the findings and behavior of materials with thermocycling, the following can be concluded:

1.Vita Amberia Pressed ceramic is significantly more translucent than all the other tested groups before and after thermocycling.

2.Thermocycling affected color and translucency of tested ceramics but still considered clinically accepted.

3.Thermocycling significantly increases the surface roughness of all the tested ceramic materials.

**Conflict of Interest:**

The authors declare no conflict of interest.

**Funding:**

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

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