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SEM evaluation of surface roughness variations in milled ceramic laminate veneers exposed to glazing and thermocycling

Abstract. CAD/CAM technologies represent one of the most rapidly advancing areas in digital restorative dentistry. This in vitro study aimed to investigate the effects of glazing and thermocycling on the surface roughness characteristics of four distinct milled CAD/CAM ceramic materials. **Aim** —to assess the effects of glazing and thermocycling on the surface roughness of four different types of CAD/CAM ceramic laminate veneers. The null hypothesis was that a statistically significant difference would exist in surface roughness between glazed and unglazed specimens across all four ceramic materials tested. **Materials and methods.** As part of the investigation, 80 CAD/CAM ceramic veneer samples were milled using CAD/CAM system. The processing occurred after scanning of the first right typodont incisor of the upper jaw model prepared with the palatal chamfer preparation design without approximal involvement (KaVo, Germany) via an Omnicam scanner. Four different CAD/CAM ceramic materials were evaluated in this study: lithium disilicate IPS E.max CAD (Ivoclar, Germany), leucite-reinforced ceramic IPS Empress CAD (Ivoclar), feldspathic ceramic Cerec (Dentsply Sirona, Germany), and hybrid ceramic Cerasmart (GC, Japan). The 80 samples were categorized into four groups (20 in each); each group was further subdivided into glazed and non-glazed subgroups, with 10 samples in each subgroup. All specimens underwent 10,000 thermal cycles. The surface roughness values were evaluated at three stages: post-milling, post-glazing, and post-thermocycling. Scanning electron microscope images (magnifications of 100x, 250x, 500x, and 1000x) were captured for each material before glazing and after thermocycling. **Results.** Significant differences in surface roughness values were observed among materials after glazing and thermocycling. Surface roughness notably decreased following glazing. Significantly higher surface roughness values were observed in the Cerec group compared to Cerasmart, Empress, and E.max groups ($p<0.05$). Analysis of the glazed surfaces after thermocycling also revealed significant differences among the groups ($p<0.05$). Tamhane's T2 post-hoc test revealed that the Cerec group exhibited significantly higher surface roughness values compared to Cerasmart, Empress, and E.max after thermocycling ($p<0.05$). For non-glazed samples, thermocycling similarly led to higher surface roughness values in the Cerec group compared to the other three groups ($p<0.05$). These findings highlight the effects of glazing and thermocycling on the surface roughness of CAD/CAM ceramic materials, reflecting their clinical behavior. **Conclusion.** There were statistically significant differences in surface roughness between glazed and non-glazed CAD/CAM materials. Among the tested materials, the Cerec group consistently showed higher roughness values compared to Cerasmart, Empress, and E.max ($p<0.05$). Glazing and thermocycling significantly influenced the surface roughness of all groups.

Key words: CAD/CAM ceramic blocks, restorative and digital dentistry, feldspathic ceramics, Cerec, leucite reinforced ceramic, Empress, hybrid ceramic, Cerasmart, lithium disilicate, E.max

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СЭМ-оценка изменений шероховатости поверхности фрезерованных керамических виниров после глазурования и термоциклирования

Реферат. CAD/CAM-технологии представляют собой одну из наиболее динамично развивающихся областей цифровой ортопедической стоматологии. Данное in vitro исследование было направлено на изучение влияния глазурования и термоциклирования на характеристики шероховатости поверхности четырех различных фрезерованных керамических материалов CAD/CAM. **Цель работы** — оценить воздействие глазурования и термоциклирования на шероховатость поверхности четырех типов керамических виниров CAD/CAM. Нулевая гипотеза заключалась в предположении, что статистически значимые различия в шероховатости поверхности будут выявлены между глазурованными и неглазурованными образцами во всех четырех исследуемых материалах. **Материалы и методы.** В рамках исследования было изготовлено 80 образцов керамических виниров CAD/CAM методом фрезерования. Подготовка осуществлялась после сканирования первого правого резца верхней челюсти на типодонте, препарированного по методике нёбного шамферного уступа без апроксимального вовлечения (KaVo, Германия), с использованием сканера Omnicam. В исследовании были протестированы четыре различных CAD/CAM-керамики: литий-дисиликат IPS E.max CAD (Ivoclar Vivadent, Германия), лейцит-усиленная керамика IPS Empress CAD (Ivoclar Vivadent), полевошпатная керамика Cerec (Dentsply Sirona, Германия) и гибридная керамика Cerasmart (GC, Япония). Все 80 образцов были разделены на четыре группы (по 20 в каждой), каждая группа была дополнительно подразделена на глазурованные и неглазурованные подгруппы по 10 образцов. Все образцы подвергались 10 000 циклам термоциклирования. Шероховатость поверхности оценивали на трех стадиях: после фрезерования, после глазурования и после термоциклирования. Для каждого материала были получены изображения с использованием сканирующего электронного микроскопа при увеличениях 100, 250, 500 и 1000 до глазурования и после термоциклирования. **Результаты.** После глазурования и термоциклирования выявлены статистически значимые различия в показателях шероховатости поверхности между материалами. Шероховатость поверхности значительно снижалась после глазурования. Наиболее высокие показатели шероховатости были зарегистрированы в группе Cerec по сравнению с группами Cerasmart, Empress и E.max (p<0,05). Анализ глазурованных поверхностей после термоциклирования также показал достоверные различия между группами (p<0,05). Пост-хок тест Tamhane T2 выявил, что группа Cerec имела статистически более высокие показатели шероховатости по сравнению с Cerasmart, Empress и E.max после термоциклирования (p<0,05). Для неглазурованных образцов термоциклирование также привело к повышению шероховатости поверхности в группе Cerec по сравнению с тремя другими группами (p<0,05). Эти результаты подчеркивают влияние глазурования и термоциклирования на показатели шероховатости поверхности CAD/CAM-керамики, что отражает их клиническое поведение. **Заключение.** Между глазурованными и неглазурованными образцами CAD/CAM-материалов были выявлены статистически значимые различия в шероховатости поверхности. Среди протестированных материалов группа Cerec стабильно демонстрировала более высокие значения шероховатости по сравнению с Cerasmart, Empress и E.max (p<0,05). Глазурование и термоциклирование оказывали существенное влияние на показатели шероховатости всех исследуемых групп.

Ключевые слова: CAD/CAM-керамические блоки, реставрационная и цифровая стоматология, полевошпатная керамика, Cerec, лейцит-усиленная керамика, Empress, гибридная керамика, Cerasmart, литий-дисиликат, E.max

ДЛЯ ЦИТИРОВАНИЯ:

Ahmedbeyli F.R., Ahmedbeyli R.R., Ersoy M. SEM evaluation of surface roughness variations in milled ceramic laminate veneers exposed to glazing and thermocycling. — *Клиническая стоматология*. — 2025; 28 (3): 84—89. DOI: 10.37988/1811-153X_2025_3_84

INTRODUCTION

A wide variety of materials is now available for digital milling, expanding the range of options in CAD/CAM treatment planning. However, selecting the most appropriate material for a specific clinical indication remains a challenge. Accordingly, this study aims to provide an overview of the evolution of ceramic CAD/CAM materials, evaluate their individual characteristics, and critically assess recent clinical evidence [1].

In dentistry, the CAD/CAM procedure refers to the fabrication of indirect restorations using computer-aided design (CAD) in combination with computer-aided manufacturing (CAM) through a digital milling process [2]. The first system was developed in 1971 by Duret and colleagues, but due in large part to insufficient digitizing accuracy, insufficient processing power, and other considerations, it was not widely utilized [3]. The development of CAD/CAM technology was driven by the desire to improve the esthetics of restorations, give them a more uniform appearance, ensure the strength of restorations, and simplify, speed up and improve dental procedures [4].

The reliability of CAD/CAM blocks is boosted since they show fewer defects and porosity than hand-built materials do [5]. Dental CAD/CAM technologies are widely recommended for the fabrication of indirect restorations using ceramic or hybrid materials. Recently, ceramic-containing composite materials have been significantly enhanced, offering superior physical and mechanical properties compared to traditional ceramics [6, 7]. The data acquisition phase of a dental scan varies depending on the CAD/CAM system employed. The scanner interfaces with CAD software, which is typically compatible with multiple components within the CAD/CAM workflow [8, 9]. The digital scanning method makes the process more comfortable for the patient, reducing the time and resources required for classical impression procedures and the preparation of plaster models [10—12]. Users can either select a predefined template provided by the CAD software or modify it to create a customized design. Once the restoration design is finalized, the CAD software converts the virtual model into a format compatible with the CAM system, initiating the milling process [13—15].

This study provides new data characterizing the physical properties of various CAD/CAM materials, which will enable a more rational approach to selecting CAD/CAM blocks for different clinical situations.

The purpose of the study — to evaluate the effect of glazing and thermal cycling on the surface roughness values of four different types of ceramic milled CAD/CAM veneers.

MATERIALS AND METHODS

In this in vitro study, typodont models (Kerr, Germany) were used, each featuring a maxillary central incisor prepared with minimal incisal edge reduction and no proximal intervention, in accordance with standardized guidelines.

Four types of ceramic materials in CAD/CAM block form were employed:

I — lithium disilicate-based glass ceramics IPS E.max CAD (Ivoclar Vivadent, Germany);
II — leucite-reinforced glass ceramics Empress CAD (Ivoclar Vivadent);
III — feldspathic ceramics CEREC (Dentsply Sirona, Germany);
IV — hybrid nanoceramics Cerasmart (GC, Japan).

To design the restoration, three-dimensional modeling was conducted using CEREC SW 4.6.1 software. The margins of the prepared tooth were initially identified automatically and then manually adjusted (fig. 1). The software then automatically generated the digital shape of the restoration (fig. 2).

Fig. 1. Drawing the margins of the prepared tooth

Fig. 2. Designing and editing the restoration

Twenty laminate-shaped specimens were milled from each material (n=20), each measuring 10 mm in length, 0.8 mm in thickness at the middle and incisal thirds, and 0.7 mm in the cervical region. All specimens were divided into four main groups (n=20 per material), and each group was further subdivided into two subgroups: glazed (n=10; fig. 3A) and unglazed (n=10; fig. 3B).

Milling of the samples was carried out using a milling device CEREC MC XL (Dentsply Sirona). After the appropriate block was fitted into the milling device, it was fixed using a special diameter screwdriver. Three-dimensional modeling for the restoration design was carried out using the CEREC software. Cylindrical burs and step burs were used for the milling process (see table 1).

Before milling, separate burs were dedicated for each group of materials. After milling, the samples were carefully taken and placed into a designated box with identification numbers to prevent mixing. Due to the higher hardness of the E.max block, the milling process took longer compared to the Empress, CEREC, and Cerasmart groups. To achieve final strength, the partially crystallized samples from the IPS E.max CAD group were fully crystallized in oven at 850°C, with an initial temperature of 550°C for 20 minutes.

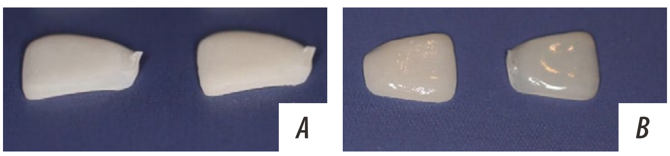

Fig. 3. Samples without (A) and with (B) the application of the glaze layer (B)

Ten samples each from the IPS Empress CAD and IPS E.max CAD groups were glazed using IPS Ivocolor Glaze Paste (refer to Table 1). These glazed samples were fired for 20 minutes at 840°C, with an initial temperature of 550°C. CEREC samples underwent the glazing process at a temperature of 820°C, with an initial temperature of 550°C.

Cerasmart samples were finished with Optiglaze Color glaze. The external surface of Cerasmart samples was treated with a steam cleaner for 5 seconds and subsequently sandblasted with aluminum oxide particles sized at 25 μm (0.15 MPa/2.5 bar). After cleaning and drying, Multi Primer was applied to the surface of 10 samples from the Cerasmart group. The samples were then stored in a closed box for 60 seconds. Glaze Opticolor was applied to the surface of the Cerasmart samples. Polymerization was carried out using a polymerization lamp for 40 seconds. Surface roughness measurements were conducted on both sides of each sample using a profilometer.

The samples underwent the thermocycling process in distilled water at temperatures of 5°C and 55°C (±2°C) using 10,000 cycles. It has been noted that 10,000 cycles approximately correspond to one year [14]. The dwell time was set to 10 seconds, and the holding time at each temperature was set to 20 seconds.

Table 1. List of materials used in this study

Material	Materials type	Manufacturer	Part no.
Step Bur 12S	CAD/CAM bur	Dentsply, Sirona, Germany	E72579
Cylinder Pointed Bur 12S	CAD/CAM bur	Dentsply, Sirona, Germany	M71663
IPS Ivocolor Refill	Glaze material—liquid	Ivoclar Vivadent, Germany	Z005KX
IPS Ivocolor Refill	Glaze material—powder	Ivoclar Vivadent, Germany	Z003H6
Glaze mixture	Mixture for glaze from liquid and powder	Ivoclar Vivadent, Germany	Z005KX, Z003H6
Optiglaze Color	Glaze material	GC, Japan	1909141

The average surface roughness values from each sample were measured three times at three different points using a profilometer Perthometer M1 (Mahr, Germany). Measurements were taken at the incisal edge, in the middle, and cervical areas of the samples. These measurements were performed after milling, after glazing, and after thermocycling.

Surface roughness measurements were performed at three stages: after milling, after glazing, and after thermocycling. Scanning electron microscopy images (at magnifications of 100x, 250x, 500x, and 1000x) were obtained from each material both after milling and after thermocycling.

The Kolmogorov—Smirnov and Shapiro—Wilk tests were used to assess the normality of the data distributions and confirm their suitability. When group variances were homogeneous, the Tukey HDS and Tamhane's T2 tests were applied; otherwise, the oneway ANOVA test was used to compare parameters between material groups. Within-group comparisons before and after thermocycling were conducted using paired sample t-tests. For repeated measurements, ANOVA was employed, followed by post hoc Bonferroni tests to compare values across milling, glazing, and thermocycling periods. The significance level for all analyses was set at $p<0.05$.

RESULTS

A statistically significant difference in surface roughness values was observed among the different ceramic materials tested. The application of a glaze layer led to a notable reduction in surface roughness across all groups. However, the glazed CEREC group exhibited significantly higher surface roughness compared to the Cerasmart, IPS E.max, and IPS Empress CAD groups ($p<0.05$).

A statistically significant difference was observed in the surface roughness values of the glazed materials following thermocycling ($p<0.05$). Similarly, significant differences were noted between materials in terms of mean surface roughness values after milling and glazing ($p<0.05$). Among the groups, the E.max samples exhibited significantly higher surface roughness after milling compared to the Empress, Cerasmart, and CEREC groups ($p<0.05$; table 2).

In the unglazed subgroups, a statistically significant difference in surface roughness values was also found after milling ($p<0.05$). E.max again showed significantly higher roughness values compared to the other materials ($p<0.05$). Furthermore, in the absence of a glaze layer, thermocycling resulted in statistically significant differences in average surface roughness among the materials ($p<0.05$). Surface roughness values after thermocycling were highest in the E.max group, significantly exceeding those of the Cerasmart, CEREC, and Empress groups ($p<0.05$). Overall, surface roughness values after thermocycling were significantly lower than those observed after the milling process across all materials ($p<0.05$; table 3).

Scanning electron microscope (SEM) analysis revealed that glazed specimens exhibited smoother surface topography compared to their unglazed counterparts. In the Cerasmart group (fig. 4), glazed surfaces showed only minor irregularities. The E.max group presented a glazed

Table 2. Surface roughness values with glaze layer (in μm)

Materials	After milling	After glazing	After thermocycling
Cerasmart	1.06±0.07	0.31±0.02*	0.34±0.02
E.max	1.30±0.06	0.16±0.01*	0.16±0.01
Empress	1.14±0.04	0.19±0.01*	0.19±0.01
Cerec	0.98±0.04	0.39±0.01*	0.39±0.02
$p^{\#}$	<0.001	<0.001	<0.001

*Remark. * — Statistically significant differences between the milling and glazing stage (p<0.001); # — oneway ANOVA test.*

Table 3. Surface roughness values without a glaze layer (in μm)

Materials	After milling	After thermocycling	p*
Cerasmart	1.04±0.06	1.29±0.04	<0.001
E.max	1.30±0.03	1.39±0.02	<0.001
Empress	1.14±0.02	1.10±0.21	0.543
Cerec	0.96±0.06	1.24±0.04	<0.001
$p^{\#}$	<0.001	<0.001	

*Remark. * — paired samples t test; # — oneway ANOVA test.*

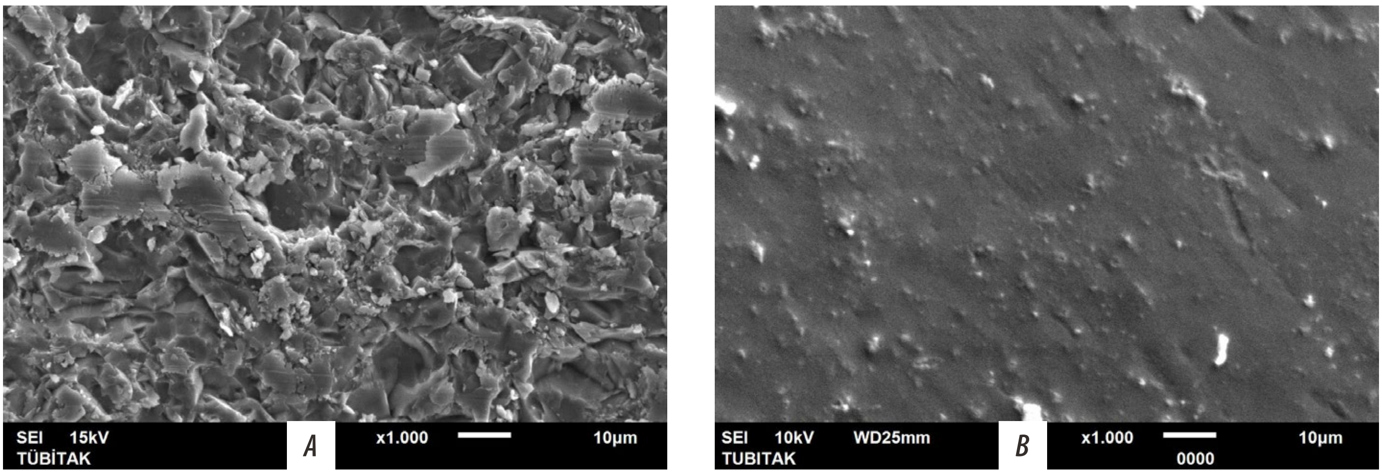

Fig. 4. Surface of Cerasmart sample: A — after milling, B — after glazing and thermocycling (SEM, mag. 1000×)

layer with uneven areas and localized surface irregularities (fig. 5). In contrast, the glazed surface of the CEREC group (fig. 6) appeared more homogeneous and uniform. The Empress group (fig. 7), however, displayed distinct surface defects, including fuzzy scratches and pronounced, deep irregularities on the glazed layer.

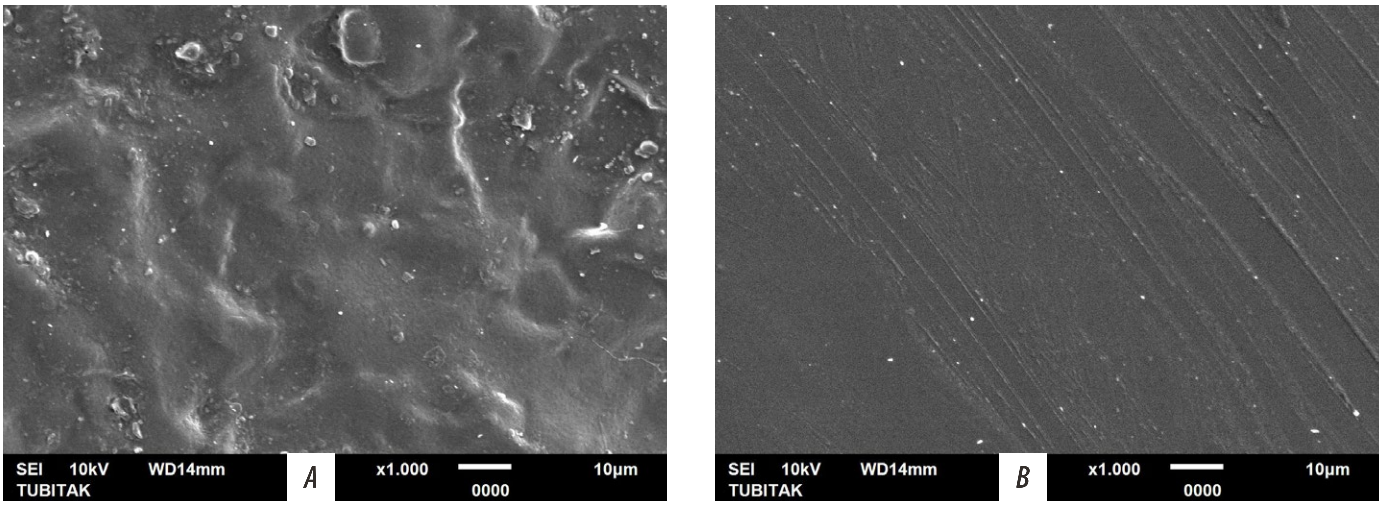

Fig. 5. Surface of E.max sample: A — after milling, B — after glazing and thermocycling (SEM, mag. 1000×)

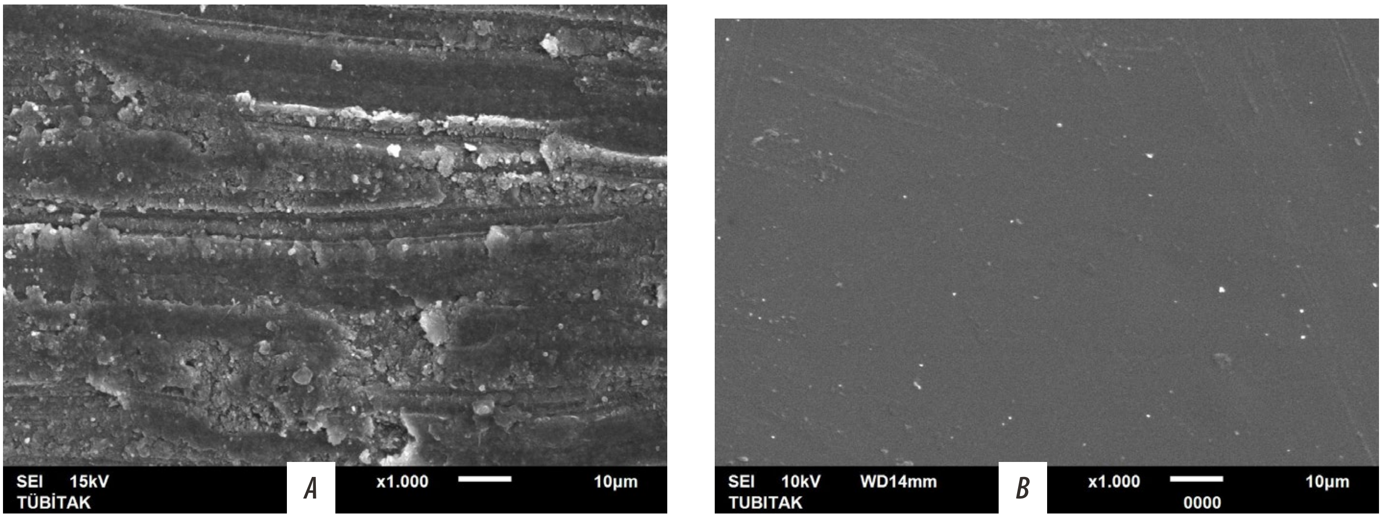

Fig. 6. Surface of Cerec sample: A — after milling, B — after glazing and thermocycling (SEM, mag. 1000×)

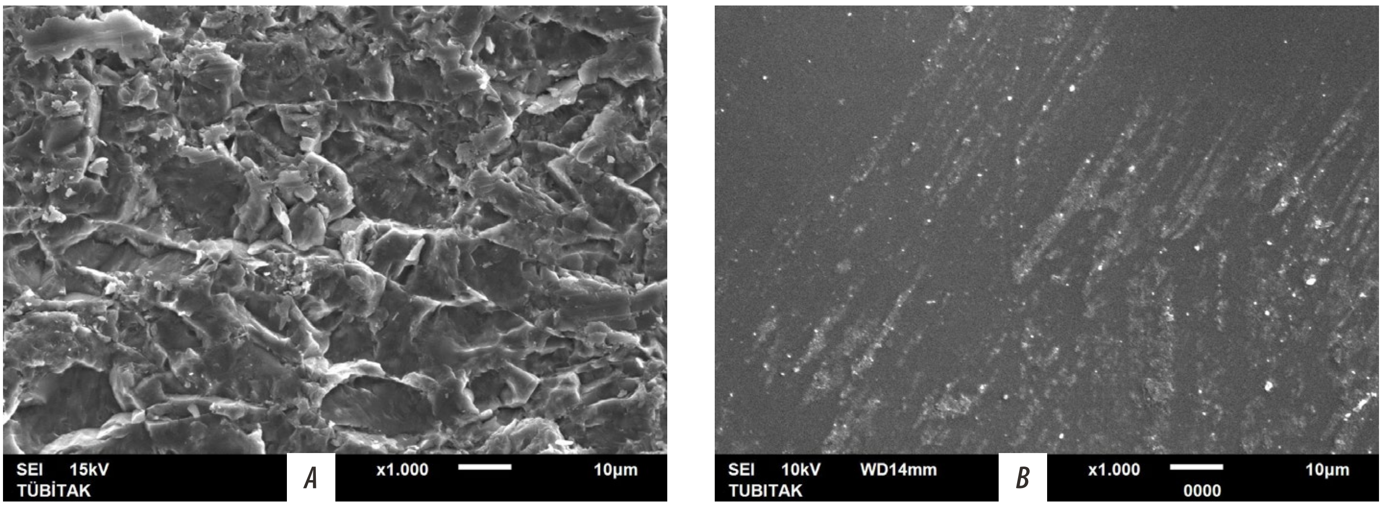

Fig. 7. Surface of Empress sample: A — after milling, B — after glazing and thermocycling (SEM, mag. 1000×)

DISCUSSION

In a study by Vasiliu et al. (2020) [16], the effect of thermocycling on the surface roughness of various CAD/CAM ceramics was investigated. The results showed that feldspathic ceramics (Vita Mark II) exhibited a higher mean surface roughness (R_a=0.09±0.06 μm) compared to lithium disilicate ceramics E.max CAD (R_a=0.03±0.07 μm) after thermocycling. These findings suggest that lithium disilicate is more resistant to surface degradation caused by thermal aging. Similarly, in the present study, the lithium disilicate group demonstrated lower R_a values than the feldspathic ceramic group following thermocycling. This supports previous research indicating the superior resistance of lithium disilicate ceramics to surface alterations under thermal stress. The consistency between the two studies reinforces the understanding that material composition significantly influences surface roughness changes due to aging procedures.

CONCLUSION

The findings of this in vitro study confirmed the research hypothesis, demonstrating statistically significant differences in surface roughness between glazed and non-glazed CAD/CAM ceramic samples. Notable variations in surface roughness values were also observed among the different material groups. In particular, the CEREC group exhibited significantly higher surface roughness compared to the Cerasmart, Empress, and IPS E.max groups (p<0.05). Both the glazing and thermocycling processes were found to have a significant impact on the surface roughness of the tested materials. Overall, the study concludes that the application of a glaze layer and exposure to thermocycling lead to measurable and statistically significant alterations in the surface characteristics of CAD/CAM ceramics.

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