THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

Effect of multiple firings on surface roughness and flexural strength of CAD-CAM ceramics

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ABSTRACT

Statement of problem. Knowledge on the effect of multiple firings on surface roughness and the flexural strength of different types of monolithic computer-aided design and computer-aided manufacturing (CAD-CAM) ceramics is limited.

Purpose. The purpose of this in vitro study was to evaluate the effect of multiple firings on the surface roughness and flexural strength of 4 different CAD-CAM ceramics after thermocycling.

Material and methods. Four different CAD-CAM ceramics (Lava All Zirconia 3-mol yttria tetragonal zirconia polycrystal [3-YTZP] [Z], VITA SUPRINITY [S], IPS e.max CAD [EX], IPS Empress CAD [E]) (n=33) were wet-sectioned to form rectangular 18×4×1.2-mm specimens. After glaze application, the specimens were divided into 3 subgroups according to the number of firings (1, 2, and 4) (n=11). The specimens were thermocycled (10 000 cycles), and surface roughness and flexural strength values were measured (n=10). One additional specimen from each group was analyzed by using scanning electron microscopy (SEM). Data were analyzed by 2-way analysis of variance (ANOVA) and the Tukey honestly significance difference (HSD) test (α=.05).

Results. According to the 2-way ANOVA, the material, number of firings, and the interaction between the material and number of firings affected the surface roughness (P<.001). For flexural strength, material (P<.001) and number of firings (P<.039) were found significant. Multiple firings (2 or 4 firings) affected the surface roughness of E (P<.001). Regardless of the number of firings, the Z material had the highest flexural strength (P<.001). Four firings affected the flexural strength values only for the Z material ($P\leq$.005).

Conclusions. CAD-CAM ceramic type affected the surface roughness and flexural strength values. The surface roughness of E was lower when fired 2 or 4 times than when fired once. The flexural strength of Z was lower when fired 4 times than when fired once. (J Prosthet Dent 2022;■:■-■)

Computer-aided design and computer-aided manufacturing (CAD-CAM) techniques have progressed, facilitating the rapid fabrication of dental restorations.[1-3] Patient expectations for life-like appearance have led to the development and use of CAD-CAM systems[2,4-11] for different monolithic ceramics to fabricate indirect restorations, which are glazed or polished before cementation.[12,13] The manufacturers typically recommend a single extrinsic characterization firing cycle to finalize the restorations; however, additional firings may be required before delivery to correct the contour, color,[14-19] or occlusion [20] or for glaze application.[3,17,21] Hence, ceramic restorations may undergo at least 2 or more firing cycles before cementation.[16,22]

Multiple firings may affect the structural integrity of ceramics.[23,24] Structural porosities and the pigments in the ceramic may become unstable, and the degree and organization of the crystal structure may change with firing, factors that can affect the clinical success of CAD-CAM ceramic restorations.[5,25-27]

Multiple firings may also affect the mechanical properties of ceramics,[24,28,29] including flexural strength,

Presented as an oral presentation at the TDB 24th International Dental Congress, 27-30 September 2018; Ankara.
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Clinical Implications

Even though 3-YTZP had the highest flexural strength among the tested CAD-CAM ceramics, clinicians should keep in mind that firing 4 times may decrease its flexural strength.

which is crucial for long-term success.[23,30] The effect of multiple firings on the color and roughness,[31] mechanical properties,[16,32-34] and optical and mechanical properties[6] of dental ceramics has been investigated. However, knowledge regarding the effect of multiple firings on the surface roughness and flexural strength of different types of monolithic CAD-CAM ceramics is limited. Therefore, the purpose of this in vitro study was to investigate the effect of the number of firings on the surface roughness and flexural strength of 4 different monolithic CAD-CAM ceramics (3-mol yttria tetragonal zirconia polycrystal [3-YTZP], zirconia-reinforced lithium silicate glass-ceramic, lithium disilicate glass-ceramic, and leucite-reinforced glass-ceramic) after thermocycling. The null hypothesis was that the surface roughness or flexural strength of CAD-CAM ceramics would not be affected by multiple firings.

MATERIAL AND METHODS

The CAD-CAM ceramics tested in this study are displayed in Table 1. Thirty-three rectangular bar-shaped specimens were cut from each CAD-CAM block—3-YTZP zirconia (Lava All Zirconia; 3M ESPE) (Z); zirconia-reinforced lithium silicate glass-ceramic (VITA SUPRINITY; VITA Zahnfabrik) (S); lithium disilicate glass-ceramic (IPS e.max CAD; Ivoclar AG) (EX); leucite-reinforced glass-ceramic (IPS Empress CAD; Ivoclar AG) (E)—with a slow-speed sectioning machine (Secotom 10; Struers A/S) under water cooling. The specimen size of Z (22.5×5×1.5 mm) was determined because of the 20% sintering shrinkage, and other materials were cut to a final size of 18×4×1.2 mm from ceramic blocks. All sectioned specimens were ultrasonically cleaned in a bath (Eltrosonic Ultracleaner 07-08; Eltrosonic GmbH) of distilled water for 5 minutes.

For each ceramic material, the corresponding glaze material was applied on 1 surface of the specimens according to the manufacturers' instructions. Then, the specimens were divided into 3 subgroups according to the number of firings (1, 2, or 4) (n=11). The manufacturer's recommended firing procedures for each ceramic firing group are displayed in Table 2. Before sintering, sectioned Z specimens were stored in a plastic cup containing zirconia coloring liquid (A3) (Lava Plus High Translucency Zirconia Dyeing Liquid; 3M ESPE) for 2 minutes. The specimens were removed from the liquid

Table 1. Ceramic materials tested

Material	Composition	Code	Manufacturer	Lot Number
Lava All Zirconia (3-mol yttria tetragonal zirconia polycrystal [3-YTZP])	ZrO_2	Z	3M ESPE	547433
VITA SUPRINITY (zirconia-reinforced lithium silicate glass-ceramic)	ZrO_2, SiO_2, Li_2O, pigments	S	VITA Zahnfabrik	35540
IPS e.max CAD (lithium disilicate glass-ceramic)	SiO_2, Li_2O, K_2O, MgO, Al_2O_3, P_2O_5, other oxides	EX	Ivoclar AG	U17559
IPS Empress CAD (leucite-reinforced glass-ceramic)	SiO_2, Al_2O_3, K_2O, Na_2O, CaO, other oxides, pigments	E	Ivoclar AG	U22653

Al_{203}, aluminum oxide; CaO, calcium oxide; K_{20}, potassium oxide; Li_{20}, lithium oxide; MgO, magnesium oxide; Na_{20}, sodium oxide; P_{205}, phosphorus pentaoxide; SiO_2, silicon dioxide; ZrO_2, zirconium dioxide.

and dried for 2 hours before sintering (inFire HTC Speed; Dentsply Sirona) at 1500 °C for 160 minutes. After sintering, glaze (VITA AKZENT Plus Glaze; VITA Zahnfabrik) was applied on the Z material and fired according to the manufacturer's recommendations. The specimens were not polished after glaze firing. For glass-ceramic specimens (S, EX, and E), glaze (S: Vita Akzent Plus Glaze LT Spray; VITA Zahnfabrik, EX: IPS e.max CAD Crystall/Glaze Spray; Ivoclar AG, E: IPS Empress Universal Glaze Spray; Ivoclar AG) was sprayed on the specimens from a 10-cm distance to produce a uniform, dry, and whitish glaze layer on all specimens. The specimens were crystallized and glazed in 1 step in a ceramic oven (Programat P300; Ivoclar AG) by following the manufacturers' recommendations.

Ten specimens in each group were thermocycled (5-55 °C, transfer time 5 seconds, duration time 30 seconds, 10 000 cycles) (MTE-101; MOD Dental). The surface roughness (R_a in μm) of all specimens was measured with a contact profilometer (Mitutoyo Surftest SV-2100; Mitutoyo Corp) after thermocycling (5.5 mm transverse length, 1 mm/s stylus speed, and 0.8 mm cutoff value). Three measurements were made from the center of each specimen at least 0.5 mm away from each other, and the mean R_a value was calculated.

After surface roughness measurements, the 3-point flexural strength test was performed by using a universal testing machine (MIN 100; Esetron) according to International Organization for Standardization (ISO) 6872:2015 (Dentistry-Ceramic materials)[35] with a 1-mm/min loading rate. The load was applied at the center of the specimen. Maximum load at fracture was recorded for each specimen (N), and flexural strength values (MPa) were obtained by using the following formula[36]: σ=3Fd/2wh2, where F is the maximum load at fracture (N), d is the distance between the supports (mm), w is the width of the specimen at the center (mm), and h is the height of the specimen at the center (mm).

Table 2. Firing parameters for 3-mol yttria tetragonal zirconia polycrystal (3-YTZP) (Z), zirconia-reinforced lithium silicate glass-ceramic (S), lithium disilicate glass-ceramic (EX), leucite reinforced glass-ceramic (E), and experimental groups according to ceramic type and number of firings

	Z	S		EX		E
Firing Parameters	**Glaze**	**Crystallization+Glaze**	**Glaze**	**Crystallization+Glaze**	**Glaze**	**Glaze**
Stand by temperature (°C)	500	400	400	403	403	403
Closing time (min)	4:00	4:00	4:00	6:00	6:00	6:00
Heating rate (°C/min)	80	55	80	t1 90 t2 30	60	100
Firing temperature (°C)	900	840	800	T1 820 T2 840	770	790
Holding time (min)	1:00	8:00	1:00	H1 0:10 H2 7:00	1:00-2:00	1:00-2:00
Vacuum (°C)	–	V1 (°C) 410 V2 (°C) 840	–	V1 (°C) 1_1 550 2_1 820 V2 (°C) 1_1 820 2_1 840	V1 (°C) 450 V2 (°C) 769	V1 (°C) - V2 (°C) -
Long term cooling (°C)	–	680	–	700	–	–
Cooling rate (°C/min)	–	0	–	0	–	–
Experimental groups	Control (1 firing): Z (sintering, glaze firing), S (crystallization+glaze firing), EX (crystallization+glaze firing), E (glaze firing) 2 Firings: Z (sintering, 2 glaze firings), S (crystallization+glaze, 1 glaze firing), EX (crystallization+glaze, 1 glaze firing), E (2 glaze firings) 4 Firings: Z (sintering, 4 glaze firings), S (crystallization+glaze, 3 glaze firings), EX (crystallization+glaze, 3 glaze firings), E (4 glaze firings)					

CAD-CAM, computer-aided design and computer-aided manufacturing All firing procedures for each CAD-CAM ceramic material performed according to manufacturer's instructions.

For the effect of multiple firings on ceramic surfaces, 1 additional specimen from each group was examined with scanning electron microscopy (SEM) (Leo 440; Zeiss) at ×700 magnification. Specimens used in this analysis were not used in the surface roughness and flexural strength measurements. Surface roughness and flexural strength values were analyzed by 2-way analysis of variance (ANOVA) and the Tukey honestly significance difference (HSD) test (α=.05).

RESULTS

According to 2-way ANOVA (Table 3), the material, number of firings, and the interactions between the material and number of firings significantly affected the surface roughness of materials (*P*<.001). Material (*P*<.001) and number of firings (*P*=.039) significantly affected the flexural strength.

Except E, surface roughness values of materials did not change after multiple firings (2 or 4 firings) when compared with their control (1 firing) groups (*P*>.05) (Table 4, Fig. 1). The control (1 firing) group of E had the highest surface roughness compared with that of other materials (*P*≤.003) and firing groups of E (*P*<.001).

When the same firing groups of different materials were compared, regardless of the number of firings (Table 4, Fig. 2), the Z material had the highest flexural strength (*P*<.001). When different firing groups of the same materials were compared, for Z, the control (1 firing) group had a higher flexural strength than the 4-firing group (*P*=.005). Except Z, multiple firings did not affect the flexural strength values of ceramic materials (*P*>.05).

Table 3. Summary of analysis of variance of surface roughness and flexural strength *df*, numerator degrees of freedom

Test	Effect	*df*	F	*P*
Surface roughness	Material	3	13.936	<.001
	Number of firings	2	22.966	<.001
	Material×number of firings	6	8.313	<.001
Flexural strength	Material	3	80	<.001
	Number of firings	2	3	.039
	Material×number of firings	6	2	.067

SEM images of each ceramic system submitted to a different number of firings are given in Figure 3. For Z, small lines were present in the control group. After multiple firings, these lines disappeared, and, especially after 4 firings, small pores were present on Z specimen surfaces. For S, multiple firings had no visible effect on the material surface. For EX, after 2 firings, complex multiple lines were seen, and, after 4 firings, these complex lines disappeared. For E, no difference was seen between the control (1 firing) and 2-firing groups. However, after 4 firings, small lines and pores were seen on the material surface.

DISCUSSION

The null hypothesis was rejected because the surface roughness and flexural strength were affected by multiple firings. After multiple firings (2 or 4 firings), the surface roughness values of the E material were significantly smaller than those in the control group (1 firing). However, the surface roughness values of the other materials after multiple firings (2 or 4 firings) were not significantly

Table 4. Comparison of mean ±SD of surface roughness (μm) and flexural strength (MPa) of different CAD-CAM ceramics and firing groups (n=10 per group)

Property	Ceramic	Mean ±SD	Firing Groups	Mean ±SD
Surface roughness (μm)	Z	1.20 ±0.39	Control (1 firing)	1.39 ±0.29[c]
			2 Firings	1.21 ±0.37[bc]
			4 Firings	1.0 ±0.44[abc]
	S	0.53 ±0.38	Control (1 firing)	0.60 ±0.36[ab]
			2 Firings	0.42 ±0.33[a]
			4 Firings	0.58 ±0.46[ab]
	EX	0.84 ±0.43	Control (1 firing)	1.09 ±0.52[abc]
			2 Firings	0.73 ±0.39[abc]
			4 Firings	0.69 ±0.29[ab]
	E	1.16 ±1.00	Control (1 firing)	2.26 ±1.0[d]
			2 Firings	0.53 ±0.28[ab]
			4 Firings	0.70 ±0.32[ab]
Flexural strength (MPa)	Z	886.64 ±429.27	Control (1 firing)	1091.6 ±513.06[a,A]
			2 Firings	881.56 ±470.20[ab,C]
			4 Firings	686.76 ±135.48[b,E]
	S	157.92 ±131.16	Control (1 firing)	189.30 ±182.36[c,B]
			2 Firings	176.72 ±84.67[c,D]
			4 Firings	107.74 ±103.55[c,F]
	EX	157.98 ±60.79	Control (1 firing)	145.04 ±42.58[d,B]
			2 Firings	139.92 ±49.54[d,D]
			4 Firings	188.99 ±77.62[d,F]
	E	172.79 ±87.20	Control (1 firing)	231.86 ±85.27[e,B]
			2 Firings	125.43 ±67.94[e,D]
			4 Firings	166.99 ±80.98[e,F]

CAD-CAM, computer-aided design and computer-aided manufacturing; SD, standard deviation. For surface roughness, different lowercase letters in same column show significant differences among material-firing group pairs. For flexural strength, different lowercase letters in same column show significant differences among firing groups within same material, while different uppercase letters in same column show significant differences among same firing groups of different CAD-CAM materials. SD, standard deviation.

different from those of the control groups (1 firing). The control (1 firing) group of Z had a higher flexural strength than the 4-firing group.

The difference between E and other materials in terms of the effect of multiple firings on the surface roughness may be because of the difference in composition of these materials and the different glaze materials used. As reported previously,[37,38] further melting and fusion of the porcelain surface with the glaze material may have filled small irregularities and led to smoother surfaces after multiple firings compared with the control groups. The surface roughness findings were consistent with those of previous studies.[31,32] Goudas et al[31] evaluated the surface roughness of a dental ceramic (IPS Classic) after multiple firings (1, 3, 5, 7 firings), reporting that lower surface roughness values were observed when the number of firings increased. Meng et al[32] reported that up to 4 firings (1, 2, 3, 4 firings) had no effect on the surface roughness of lithium disilicate glass-ceramic (IPS e.max CAD).

In the present study, multiple firings did not affect the flexural strength of S, EX, and E materials. Only the flexural strength of Z was significantly affected by multiple firings. After 4 firings, the Z material had lower flexural strength than the control (1 firing) group, whereas the flexural strength of 2- and 4-firing groups was similar. Previous studies,[6,16,33,34] which investigated the effect of the flexural strength of ceramics, used different materials and different firing cycles. Miranda et al[33] evaluated the effect of multiple firings (1, 3, 5 firings) on the biaxial flexural strength of lithium disilicate glass-ceramic (IPS e.max CAD), and Ozdogan and Ozdemir[34] evaluated the effects of multiple firings (1, 2, 3, 4 firings) on the flexural strength of lithium disilicate ceramics (press and CAD-CAM). Similar to the present study, the flexural strength of lithium disilicate glass-ceramic was reported to be unaffected by multiple firings. Campanelli de Morais et al[6] reported that multiple firings (control, 2, 5, 7 firings) did not affect the flexural strength of zirconia-reinforced lithium silicate glass-ceramic; however, for lithium disilicate glass-ceramic, the flexural strength decreased significantly after the seventh firing when compared with the second-firing group. Gozneli et al[16] evaluated the biaxial flexural strength of pressable ceramic materials (Empress 2; Ivoclar AG, Finesse; Ceramco, Cergo; Degussa Dental, Evopress; Wegold Edelmetalle) after multiple firings (1, 3, 5, 7 firings), reporting that up to 7 firings had no significant effect on the flexural strength of pressable lithium disilicate ceramics; however, the flexural strength of leucite-reinforced ceramic (Cergo) decreased after the seventh firing when compared with that after the first

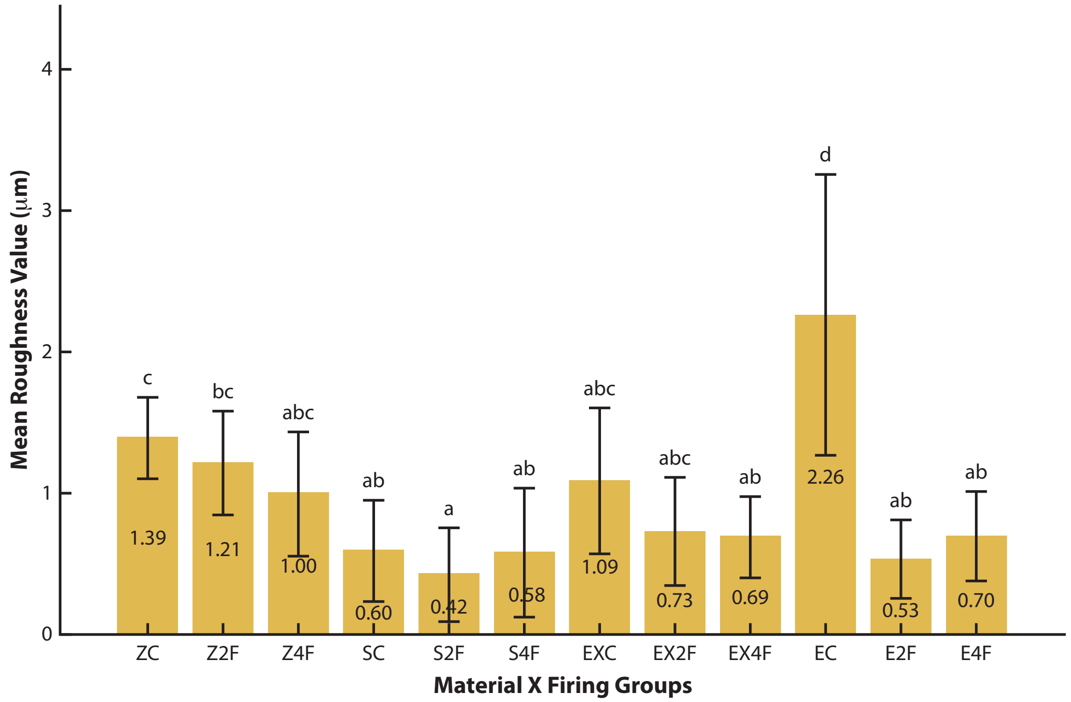

Figure 1. Mean values and 95% confidence limits for surface roughness (R_a in μm) of CAD-CAM ceramic-firing group combinations after thermocycling. Different lowercase letters show significant differences among material-firing group pairs (*P*<.05). CAD-CAM, computer-aided design and computer-aided manufacturing; EC, leucite-reinforced control (1 firing); E2F, leucite-reinforced 2 firings; E4F, leucite-reinforced 4 firings; EXC, lithium disilicate control (1 firing); EX2F, lithium disilicate 2 firings; EX4F, lithium disilicate 4 firings; SC, zirconia-reinforced lithium silicate control (1 firing); S2F, zirconia-reinforced lithium silicate 2 firings; S4F, zirconia-reinforced lithium silicate 4 firings; ZC, zirconia control (1 firing); Z2F, zirconia 2 firings; Z4F, zirconia 4 firings.

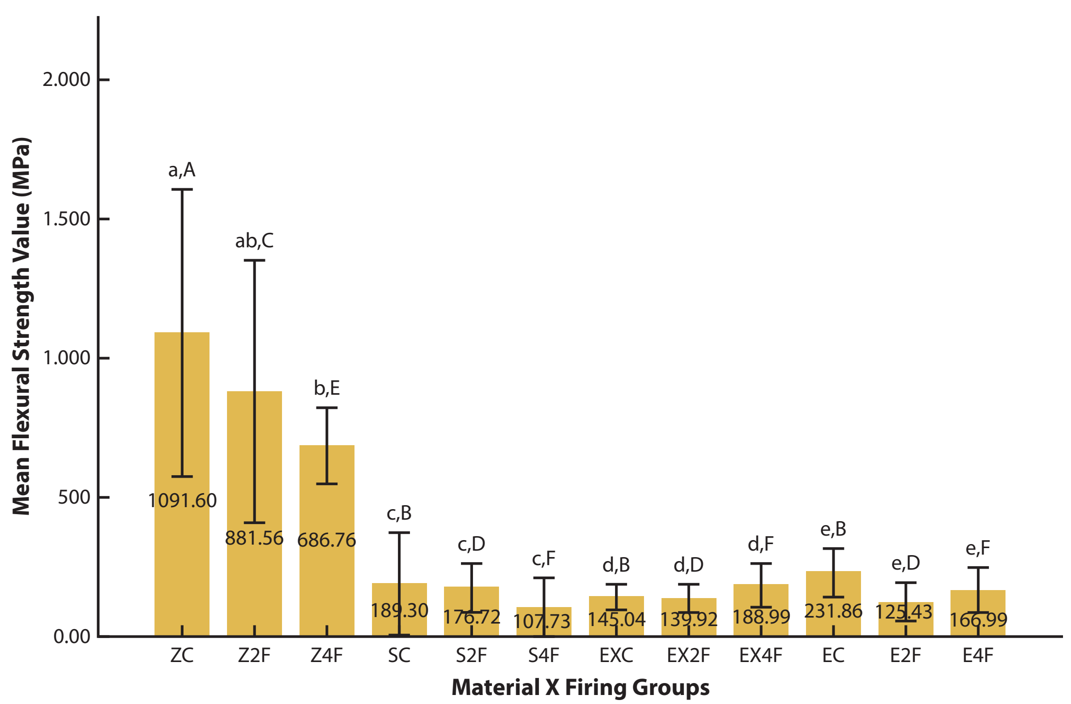

Figure 2. Mean values and 95% confidence limits for flexural strength (MPa) of CAD-CAM ceramic-firing group combinations after thermocycling. Abbreviations as in Figure 1. Different lowercase letters show significant differences among firing groups within same material. Different uppercase letters show significant differences among same firing groups of different CAD-CAM materials (*P*<.05).

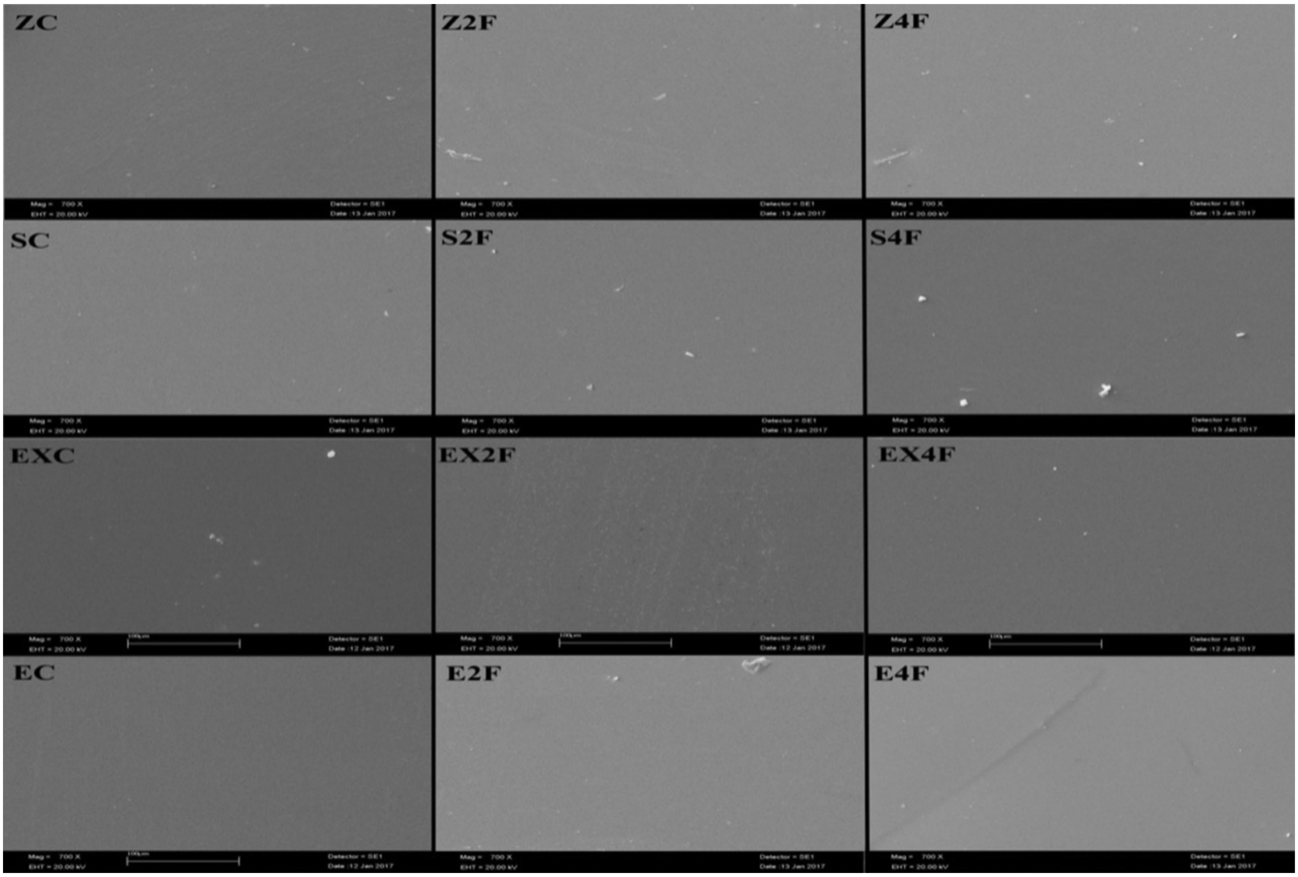

Figure 3. Scanning electron microscopy images (original magnification ×700) of CAD-CAM ceramic materials firing groups. Abbreviations as shown in Figure 1.

firing. Differences between the present study and other studies[6,16] may be because of the use of different materials and different numbers of firings.

In the present study, according to the SEM images, EX material had complex multiple lines on the surface in the 2-firing group, whereas in the 4-firing group, complex lines were not observed. This finding is consistent with that of Ozdogan and Ozdemir's study,[34] where SEM images of CAD-CAM-fabricated lithium disilicate glass-ceramic showed a complex mesh structure in the first- and second-firing groups. The mesh structures disappeared after multiple firings and were replaced by a porous surface. In contrast, Meng et al[32] did not observe alterations in the morphology of polished lithium disilicate glass-ceramic specimens in SEM images, which received a different number of firings (control and 4 firing). The difference between the study by Meng et al[32] and the present study may be because glaze was applied instead of polishing the EX material. In the Z material, a smoother surface was observed after multiple firings compared with the control (1 firing) group, which was consistent with the surface roughness results. In the S and E materials, a similar surface topography was observed in all firing groups. Based on the results of previous studies[32,34] and the present study, the effect of multiple firings on surface topography may depend on material type.

In the present study, commonly used monolithic CAD-CAM ceramics (Z, S, EX, E) were selected. Glazing was performed according to the manufacturer's recommendations for each ceramic material. Multiple firings were performed because, in clinical practice, restorations may need to be fired multiple times for color correction, glaze application, or after adjustment.[6,13,32,34] The 10 000 thermocycles used as the aging procedure has been reported to correspond to 1-year clinical aging.[39] In addition, for surface topography, a SEM analysis was performed to determine the surface changes of each ceramic material after multiple firings.

Limitations of the present study included the absence of a priori power analysis, and sample size was determined based on previous studies.[16,34,40] However, significant differences were observed. The present study was performed in laboratory conditions; thermocycling was performed only in distilled water, and both surfaces of the specimens were exposed to the thermocycling procedure, which does not completely simulate intraoral conditions. The wear of tested materials after multiple firings was not evaluated; the effects of multiple firings on wear, ion release, color, translucency, and microbial adhesion of monolithic CAD-CAM ceramics should be evaluated in future studies. To further interpret the increase or the decrease in the flexural strength values of the tested ceramics after multiple firings, their microstructures should be evaluated after each firing. Clinical studies should also be performed to investigate the long-term performance of the tested material-firing combinations.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. CAD-CAM ceramic type affected surface roughness and flexural strength values.
2. The surface roughness of the tested materials, except for E, was not affected by multiple firings. The surface roughness of E was lower after 2 and 4 firings than that in its control (1 firing) group.
3. The flexural strength of the tested materials, except for Z, was not affected by the number of firings. The Z material had a higher flexural strength than the other materials regardless of the number of firings. After 4 firings, Z had a lower flexural strength than its control (1 firing) group.

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Acknowledgments
The authors thank Ivoclar AG, VITA, and 3M ESPE for supplying the ceramic materials. The authors also thank İhsan Aksit for performing SEM analysis and Erman Toktay for the statistical analysis.

CRediT authorship contribution statement
Meryem Gülce Subaşı: Conceptualization, Investigation, Methodology, Data curation, Writing – original draft. **Gülce Çakmak:** Conceptualization, Data curation, Investigation, Methodology, Writing – review & editing. **Murat Sert:** Investigation, Data curation.

https://doi.org/10.1016/j.prosdent.2022.05.021