

RESEARCH AND EDUCATION

Alteration of the intaglio surface of lithium disilicate glass-ceramic



Catherine M. Gorman, MSc, PhD,^a Daithi de Faoite, BE, PhD,^b David Flannery, BDentTech,^c Marta Ratajczak, BDentTech,^d Tomás Kelly, BE,^e and Kenneth T. Stanton, BSc, MSc, PhD^f

Lithium disilicate glass-ceramic (LDGC) has exceptional strength for a material in the glass-ceramic category. This, coupled with excellent optical properties¹ and clinical survival rates,^{2,3} renders it among the most popular contemporary ceramic dental restorative materials,^{1,4-6} and it has been advocated for an extensive range of restorations, including inlays, onlays, veneers, crowns, and short-span fixed partial dentures.⁷

LDGC is composed of 70% lithium disilicate ($\text{Li}_2\text{Si}_2\text{O}_5$) crystals embedded in a glass matrix. The lithium disilicate crystals have a needle-like morphology, with length typically in the range 3 μm to 6 μm , and form a self-reinforced interlocking microstructure.⁸ The microstructure confers LDGCs with a high flexural strength (approximately 400 MPa), approximately 2.5 times higher than the flexural strengths of the traditional silica-based glass-

ABSTRACT

Statement of problem. Clinical adjustment of a lithium disilicate glass-ceramic (LDGC) restoration may necessitate its return to the laboratory for additional firing. Evidence of how the intaglio surface should be re-etched after internal adjustment, or after refiring, is lacking.

Purpose. The purpose of this in vitro study was to investigate the effects of different sequences of etching, refiring, diamond rotary instrument adjustment, airborne-particle abrasion, and re-etching on the microstructure and surface roughness of the intaglio surface of heat-pressed LDGCs.

Material and methods. Heat-pressed LDGC specimens were ground with abrasive paper to produce a uniformly flat surface. The groups (n=3) were subjected to different combinations of etching, refiring, diamond rotary instrument adjustment, airborne-particle abrasion, and re-etching. X-ray diffraction was used to characterize the crystalline phases. Scanning electron microscopy and surface profilometry were used to characterize the microstructure and surface roughness.

Results. Qualitative differences were observed in the surface texture of specimens etched for different periods. Excessive etching revealed more of the underlying lithium disilicate crystallites and caused surface pitting for the longest etching period studied. Refiring altered the surface condition but did not completely remove the texture created by the original etching. Diamond rotary instrument adjustment resulted in appreciable surface damage and a higher mean value of measured surface roughness (with or without re-etching) than the other groups. Airborne-particle abrasion caused embedding of particles in the specimen surface, likely corresponding to the abrasion media, although this process resulted in qualitatively less surface damage than diamond rotary instrument adjustment.

Conclusions. Excessive etching, refiring, and adjustment by airborne-particle abrasion or diamond rotary instrument result in qualitative changes in surface condition. Adjustment by diamond rotary instrument results in appreciable surface damage. (*J Prosthet Dent* 2019;122:411.e1-e10)

ceramics.⁸ Similarly, it exhibits a higher fracture toughness (2.5 $\text{MPa}\cdot\text{m}^{1/2}$ to 3.0 $\text{MPa}\cdot\text{m}^{1/2}$) than that of feldspathic or leucite glass-ceramics.⁸

^aAssistant Professor, Dental Technology, Division of Restorative Dentistry and Periodontology, Dublin Dental University Hospital, Dublin, Ireland.

^bPostdoctoral Fellow, School of Mechanical and Materials Engineering, University College Dublin, Dublin, Ireland.

^cGraduate student, School of Dental Science, Trinity College Dublin, Dublin, Ireland.

^dGraduate student, School of Dental Science, Trinity College Dublin, Dublin, Ireland.

^eGraduate student, School of Mechanical and Materials Engineering, University College Dublin, Dublin, Ireland.

^fProfessor, School of Mechanical and Materials Engineering, University College Dublin, Dublin, Ireland.

Clinical Implications

At the clinical evaluation stage, dental crowns often require additional adjustment to the intaglio surface to improve fit. The practice of clinical adjustment of the etched intaglio surface of LDGCs by using a diamond rotary instrument, with or without re-etching, may reduce the long-term clinical success of a lithium disilicate restoration.

Subsequent to adjustment by using a diamond rotary instrument, the etch period may need to be prolonged to reestablish a surface topography suitable for bonding. After airborne-particle abrasion, re-etching may not be required; however, cleaning with an air jet may be necessary to remove abrasive particles from the surface completely.

Glass-ceramic restorations benefit from resin bonding to tooth structure, with long-term clinical success being related to the quality of the resin bond.⁹ Hydrofluoric acid (HF) etching is required before bonding to increase the surface area (micromechanical retention) and the reactivity of the intaglio surface.¹⁰ Longer etching periods are associated with an increased depth of glass matrix dissolution. This increases the degree of mechanical interlocking through composite resin infiltration and has been correlated with increased microshear bond strength.¹¹ A longer etch time results in increased surface roughness (R_a), which is associated with decreased biaxial flexural strength.^{12,13} The application of a composite resin to a glass-ceramic creates a resin-ceramic layer that has been correlated with an increase in flexural strength.^{4,14-17}

Contamination of the fragile etched intaglio surface may occur at the clinical evaluation stage, where saliva, disclosing media,¹⁸ or evaluation paste¹⁹ may reduce the quality of the bond. Some dentists may airborne-particle abrade to remove contaminants. However, the quality of the resin bond relies on the surface condition of the glass-ceramic to which it adheres. Additionally, the strength of ceramic materials is controlled by their surface condition. At the clinical evaluation stage, a restoration may require adjustment to the intaglio surface to improve fit or to the external contours to improve esthetics or occlusal contacts.²⁰ Adjustments by using a diamond rotary instrument or by airborne-particle abrasion may detrimentally affect the surface characteristics of the intaglio surface of a previously etched glass-ceramic restoration and thereby result in a decrease in the bond strength.

Adjustment by using a diamond rotary instrument has been shown to reduce the flexural strength of LDGC

by surface damage.²¹ Clinical adjustments may also necessitate return of the restoration to the laboratory for refiring. However, it is unknown to what extent a previously etched intaglio surface is affected as a result of additional high-temperature firing or clinical adjustment. Evidence for the retreatment of the intaglio surface with acid etch after internal adjustment or after it has been refired at high temperatures is lacking. Therefore, the purpose of this *in vitro* study was to address this gap in knowledge by characterizing the phase composition, microstructure, and surface roughness of a heat-pressed LDGC material that has been subjected to different surface treatments. The null hypothesis was that no change occurs to the surface condition of the intaglio surface of etched LDGCs after selected clinical adjustments or additional firing at a high temperature.

MATERIAL AND METHODS

Commercially supplied LDGC ingots (IPS e.max Press; Ivoclar Vivadent AG) were used in this study. Three as-received (AR) specimens, which did not undergo any surface treatment, were characterized to provide baseline results for the material. Thirty cylindrical glass-ceramic specimens (13.2 mm in diameter) were sourced from the excess material recovered from previously heat-pressed restorations.⁷ To remove any surface artifacts and homogenize the surface condition before further processing, these specimens were ground flat on 1 surface by using successively finer grades of silicon carbide abrasive paper (P240, P600, P1200, and P2500) (CarbiMet; Buehler) under running water.

Three specimens were assigned to group HP (Table 1). The remaining specimens were placed in a ceramic furnace (Programat P310; Ivoclar Vivadent AG), and 2 stain and glaze-simulated firing cycles were performed. The furnace was heated under vacuum at 60 °C/min from 400 °C to 770 °C and held at this temperature for 90 seconds. The specimens were removed from the furnace when the temperature had cooled to 400 °C and then allowed to cool to room temperature in air. Three specimens were assigned to each of the other 9 groups (groups E20s to APA-E) and subjected to different surface treatments as outlined in Table 1. Specimens from groups RF and RF-E underwent a refiring cycle to simulate the reglazing process after etching.

An etching gel, 5% HF (IPS Ceramic Etching Gel; Ivoclar Vivadent AG), was applied to the surface of the specimens (groups E20s to APA-E) according to the etch period assigned to the group (Table 1). The gel was rinsed thoroughly from the specimen surface with running water and then allowed to air dry. Additional re-etching was carried out after further surface treatment (groups RF-E, DB-E, APA-E) according to the processing parameters outlined (Table 1).

Table 1. Details of processing steps used for test groups

Group Code	Description	Surface Preparation by Using SiC Paper	Stain Firing Cycle	Glaze Firing Cycle	Etch Time (s)	Glaze Refiring Cycle	Diamond Rotary Instrument Adjustment	Airborne-Particle Abrasion	Re-etch Time (s)	n
AR	As received	-	-	-	-	-	-	-	-	3
HP	Heat pressed, surface ground	✓	-	-	-	-	-	-	-	3
E20s	Heat pressed, surface ground, 2 firing cycles, etch 20 s	✓	✓	✓	20	-	-	-	-	3
E40s	Heat pressed, surface ground, 2 firing cycles, etch 40 s	✓	✓	✓	40	-	-	-	-	3
E60s	Heat pressed, surface ground, 2 firing cycles, etch 60 s	✓	✓	✓	60	-	-	-	-	3
RF	Heat pressed, surface ground, 2 firing cycles, etch 20 s, plus refiring, but not re-etched	✓	✓	✓	20	✓	-	-	-	3
RF-E	Heat pressed, surface ground, 2 firing cycles, etch 20 s, plus refiring and re-etch 20 s	✓	✓	✓	20	✓	-	-	20	3
DB	Heat pressed, surface ground, 2 firing cycles, etch 20 s, plus diamond rotary instrument adjustment, but not re-etched	✓	✓	✓	20	-	✓	-	-	3
DB-E	Heat pressed, surface ground, 2 firing cycles, etch 20 s, plus diamond rotary instrument adjustment and re-etch 20 s	✓	✓	✓	20	-	✓	-	20	3
APA	Heat pressed, surface ground, 2 firing cycles, etch 20 s, plus airborne-particle abrasion, but not re-etched	✓	✓	✓	20	-	-	✓	-	3
APA-E	Heat pressed, surface ground, 2 firing cycles, etch 20 s, plus airborne-particle abrasion, and re-etched for 20 s	✓	✓	✓	20	-	-	✓	20	3

✓ Indicates that processing step performed and ‘-’ indicates that step not performed. Etching steps performed denoted with number of seconds for which acid etchant applied.

Diamond rotary instrument adjustment was performed by using a fine-grit (27 μm to 76 μm) diamond rotary instrument (D.850.016.FG; Frank Dental GmbH). This was performed by placing the length of the instrument rotating at 30 000 rpm on the flat surface of the specimens (groups DB and DB-E) and maintaining this contact with firm finger pressure as it was moved once across the surface in a single direction.

Alumina (110 μm) was used to airborne-particle abrade the glass-ceramic surface at 100-kPa pressure at a uniform distance of 10 mm across the surface of the specimens (groups APA and APA-E) for a 5-second period by using a dental air abrasion unit (Basic quattro; Renfert GmbH).

X-ray diffraction (XRD) was performed on 1 specimen from each group to identify the crystalline phases present. Analysis was carried out with a diffractometer (Siemens D500; Siemens AG) with CuKα X-rays. A step size of 0.02 degrees and a dwell time per step of 10 seconds were used over 10 to 90 degrees 2θ. The specimens were rotated during analysis to enhance averaging of crystallite orientation. Peak locations were compared with entries from the ICDD-JCPDS Powder Diffraction File database and Crystallography Open Database.

Surface profiles were recorded for each specimen by using a chromatic confocal optical profilometer (Nanovea PS50; Nanovea Inc). Surface roughness parameters R_a (arithmetic mean deviation of the profile) and R_z (maximum peak-to-valley height) were calculated according to ISO 4287:1997.²² Three different line scans of 5 mm in length and 0.50-μm step size were performed on each specimen, with the specimen positioned and rotated before each measurement to achieve a representative average of surface roughness.

Scanning electron microscopy (SEM) analysis was performed to examine the microstructure of the specimens (Hitachi TM-1000; Hitachi High Technologies Europe GmbH) by using a backscatter electron detector. Specimens were not additionally etched for SEM analysis as this would alter the surface condition created by the processing parameters. Specimens were gold coated by using a sputter coater (Emitech K575x; Quorum Technologies Ltd) to prevent electrostatic charging.

Using the Mead resource equation,²³ a sample size of 3 was determined for each treatment group in this qualitative pilot study. This sample size was too small to apply standard statistical analysis techniques because the statistical power achievable would be poor and the sample size would be too small to validly assess the normality of the measured data. Instead, the R_a and R_z surface roughness metrics for groups HP to APA-E were compared only in a qualitative sense based on mean values, without drawing inferences about the statistical significance of pairwise comparisons of means for different treatment groups.

RESULTS

The crystalline phases identified in the XRD pattern for a heat-pressed LDGC specimen from group HP are given in Figure 1. Orthorhombic lithium disilicate ($Li_2Si_2O_5$) was the major crystalline phase (COD file 2003027). Additional lower intensity peaks were matched to orthorhombic lithium metasilicate (Li_2SiO_3) (PDF 00-030-0766) and to orthorhombic lithium phosphate (Li_3PO_4) (PDF 00-015-0760). A small amorphous halo was also present between 20 and 30 degrees 2θ, indicative of a residual glassy phase. XRD patterns for all

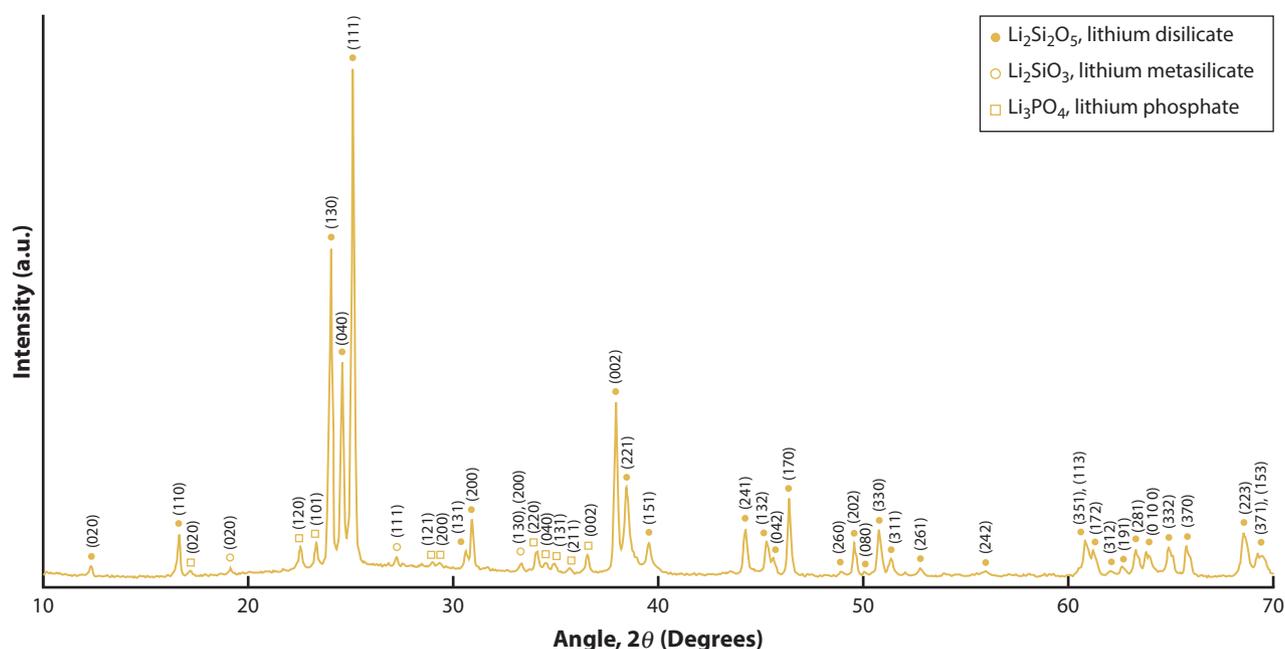


Figure 1. X-ray diffraction pattern for specimen from group HP, with crystalline phases identified. Diffraction peaks exhibited corresponding to lithium disilicate ($\text{Li}_2\text{Si}_2\text{O}_5$), lithium metasilicate (Li_2SiO_3), and lithium phosphate (Li_3PO_4).

groups are given in Figure 2. No difference was observed in crystal phase assemblage or amorphous halo intensity for the different groups, indicating that the additional processing steps did not change the crystalline composition of the glass-ceramics. Some small variations in relative peak intensity were observed for the lithium disilicate phase. Most noticeably, for group RF-E, the (130) peak was reduced in intensity, while the (002) peak was increased in intensity, indicating that some preferential orientation was present.

The arithmetic means of R_a and R_z surface roughness measurements for each of the groups are plotted in Figure 3, and mean values for R_a and R_z are given in Table 2. Representative surface profiles from each group are given in Figure 4.

The mean surface roughness (R_a) of the as-received specimens (group AR) was $\bar{x}=0.179 \mu\text{m}$, $s=0.024 \mu\text{m}$, where \bar{x} is the mean and s is the sample standard deviation. Specimens from group HP (heat-pressed only) had a lower mean R_a surface roughness ($\bar{x}=0.052 \mu\text{m}$, $s=0.044 \mu\text{m}$) than all other groups. Achieving a low surface roughness was the intended effect of the surface-grinding process performed after heat pressing to homogenize the surface condition before further processing. Specimens etched with 5% HF (20 seconds) according to the manufacturer's instructions (group E20s) exhibited a higher mean R_a ($\bar{x}=0.143 \mu\text{m}$, $s=0.057 \mu\text{m}$) and R_z ($\bar{x}=1.23 \mu\text{m}$, $s=0.43 \mu\text{m}$) relative to group HP. Specimens etched for 40 seconds (group E40s) exhibited a slightly lower mean R_a ($\bar{x}=0.111 \mu\text{m}$, $s=0.012 \mu\text{m}$) and R_z ($\bar{x}=1.05 \mu\text{m}$,

$s=0.25 \mu\text{m}$) relative to specimens etched for 20 seconds (group E20s). Specimens etched for 60 seconds (group E60s) exhibited a mean R_a ($\bar{x}=0.131 \mu\text{m}$, $s=0.010 \mu\text{m}$) close to that of specimens etched for 20 seconds (group E20s). The scatter in R_a and R_z for specimens etched for 40 seconds or 60 seconds were lower than those for specimens etched for 20 seconds.

Specimens treated by simulated refiring at high temperature after etching (group RF) exhibited a slightly higher mean R_a ($\bar{x}=0.161 \mu\text{m}$, $s=0.043 \mu\text{m}$) and R_z ($\bar{x}=1.38 \mu\text{m}$, $s=0.54 \mu\text{m}$) values relative to specimens prepared according to the manufacturer's instructions (group E20s). Specimens refired and then re-etched for 20 seconds (group RF-E) exhibited appreciably higher mean R_a ($\bar{x}=0.243 \mu\text{m}$, $s=0.063 \mu\text{m}$) and R_z ($\bar{x}=2.02 \mu\text{m}$, $s=0.73 \mu\text{m}$) values relative to group E20s and relative to specimens refired but not re-etched (group RF). Figure 4 illustrates the increase in the peak-to-trough height of a typical surface profile from group RF-E relative to group E20s, resulting from refiring and re-etching.

Specimens adjusted by using a diamond rotary instrument after etching (group DB) exhibited order-of-magnitude higher mean R_a ($\bar{x}=2.65 \mu\text{m}$, $s=1.10 \mu\text{m}$) and R_z ($\bar{x}=19.4 \mu\text{m}$, $s=6.9 \mu\text{m}$) values relative to group E20s, and these were the highest R_a and R_z values among the groups analyzed. Specimens re-etched after diamond rotary instrument adjustment (group DB-E) similarly exhibited order-of-magnitude higher mean R_a ($\bar{x}=1.66 \mu\text{m}$, $s=0.76 \mu\text{m}$) and R_z ($\bar{x}=9.24 \mu\text{m}$, $s=4.77 \mu\text{m}$) values relative to group E20s, although the mean R_a and R_z

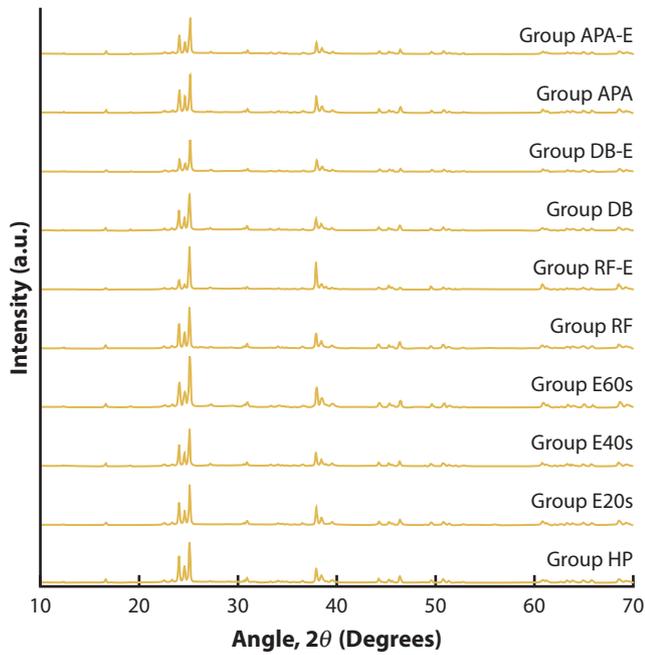


Figure 2. X-ray diffraction patterns for specimens from group HP to group APA-E, with diffraction peaks indicating presence of lithium disilicate ($\text{Li}_2\text{Si}_2\text{O}_5$), lithium metasilicate (Li_2SiO_3), and lithium phosphate (Li_3PO_4). Group HP (pressed once and not etched), group E20s (pressed, simulated stain and glaze firing, etched for 20 seconds), group E40s (pressed, simulated stain and glaze firing, etched for 40 seconds), group E60s (pressed, simulated stain, and glaze firing, etched for 60 seconds), group RF (pressed, simulated stain and glaze firing, etched for 20 seconds, simulated addition and glaze re-firing, not re-etched), group RF-E (pressed, simulated stain and glaze firing, etched for 20 seconds, simulated addition and glaze re-firing, re-etched for 20 seconds), group DB (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by using a diamond rotary instrument, not re-etched), group DB-E (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by using a diamond rotary instrument, re-etched for 20 seconds), group APA (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by airborne-particle abrasion, not re-etched), and group APA-E (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by airborne-particle abrasion, re-etched for 20 seconds).

values for the re-etched specimens were appreciably lower than those from group DB. A decrease in surface roughness was consistent with the SEM analysis, which revealed that the striations caused by the rotary instrument were reduced by re-etching.

Specimens subjected to airborne-particle abrasion after etching (group APA) exhibited mean R_a ($\bar{x}=0.131 \mu\text{m}$, $s=0.039 \mu\text{m}$) and R_z ($\bar{x}=1.83 \mu\text{m}$, $s=1.06 \mu\text{m}$) values close to those of group E20s, while specimens additionally subjected to re-etching (group APA-E) exhibited slightly higher mean R_a ($\bar{x}=0.164 \mu\text{m}$, $s=0.025 \mu\text{m}$) and R_z ($\bar{x}=2.10 \mu\text{m}$, $s=0.61 \mu\text{m}$).

Numerous thin scratches were evident in an SEM image (Fig. 5A) for group HP (heat-pressed only). The

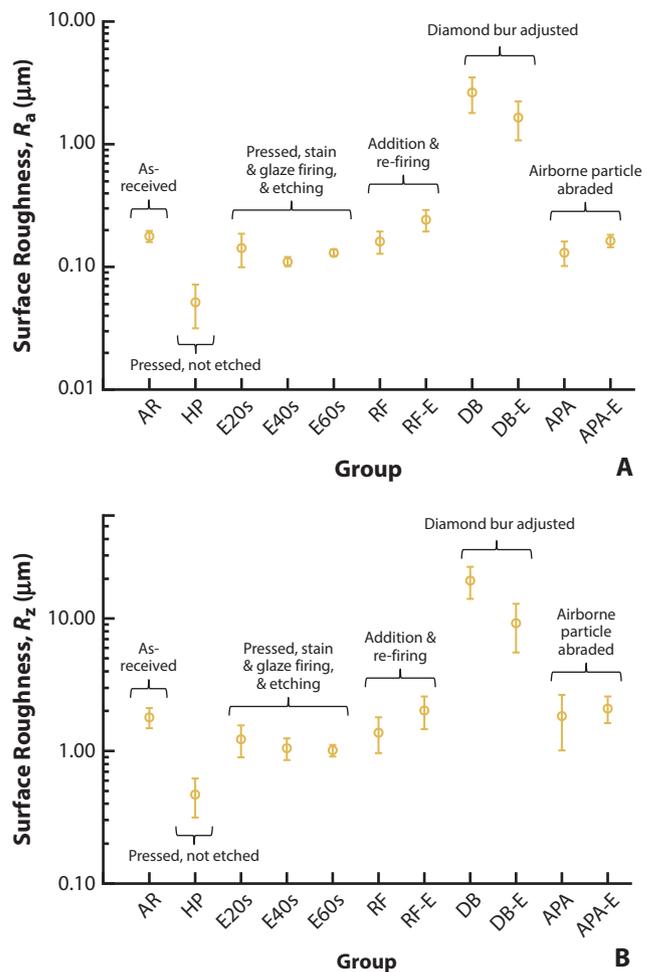


Figure 3. Arithmetic mean of surface roughness values for each group. A, R_a . B, R_z . Vertical bars indicate 95% confidence intervals for mean values. Note that vertical axes have logarithmic scales. Group AR (as received from manufacturer), group HP (pressed once and not etched), group E20s (pressed, simulated stain and glaze firing, etched for 20 seconds), group E40s (pressed, simulated stain and glaze firing, etched for 40 seconds), group E60s (pressed, simulated stain, and glaze firing, etched for 60 seconds), group RF (pressed, simulated stain and glaze firing, etched for 20 seconds, simulated addition and glaze re-firing, not re-etched), group RF-E (pressed, simulated stain and glaze firing, etched for 20 seconds, simulated addition and glaze re-firing, re-etched for 20 seconds), group DB (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by using a diamond rotary instrument, not re-etched), group DB-E (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by using a diamond rotary instrument, re-etched for 20 seconds), group APA (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by airborne-particle abrasion, not re-etched), and group APA-E (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by airborne-particle abrasion, re-etched for 20 seconds).

extent of scratches varied among specimens, likely caused by the manual polishing regimen.

The characteristic appearance of etched LDGCs as previously reported²⁴ was evident in a SEM image for

Table 2. Arithmetic mean values \pm standard deviations of surface roughness measurements (R_a and R_z) for specimens from each group

Group Code	R_a (μm)	R_z (μm)
AR	0.179 \pm 0.023	1.80 \pm 0.40
HP	0.052 \pm 0.044	0.468 \pm 0.227
E20s	0.143 \pm 0.057	1.23 \pm 0.43
E40s	0.111 \pm 0.012	1.05 \pm 0.25
E60s	0.131 \pm 0.010	1.01 \pm 0.13
RF	0.161 \pm 0.043	1.38 \pm 0.54
RF-E	0.243 \pm 0.063	2.02 \pm 0.73
DB	2.65 \pm 1.10	19.4 \pm 6.9
DB-E	1.66 \pm 0.76	9.24 \pm 4.77
APA	0.131 \pm 0.039	1.83 \pm 1.06
APA-E	0.164 \pm 0.025	2.10 \pm 0.61

Group AR (as received from manufacturer), group HP (pressed once and not etched), group E20s (pressed, simulated stain and glaze firing, etched for 20 seconds), group E40s (pressed, simulated stain and glaze firing, etched for 40 seconds), group E60s (pressed, simulated stain, and glaze firing, etched for 60 seconds), group RF (pressed, simulated stain and glaze firing, etched for 20 seconds, simulated addition and glaze refiring, not re-etched), group RF-E (pressed, simulated stain and glaze firing, etched for 20 seconds, simulated addition and glaze refiring, re-etched for 20 seconds), group DB (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by using a diamond rotary instrument, not re-etched), group DB-E (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by using a diamond rotary instrument, re-etched for 20 seconds), group APA (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by airborne-particle abrasion, not re-etched), and group APA-E (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by airborne-particle abrasion, re-etched for 20 seconds)

group E20s (Fig. 5B). The HF acid etch had the effect of selectively removing the less chemically resistant glassy phase, creating a surface suitable for resin bonding. However, the effect of the etchant on the specimen surfaces was found to be spatially nonuniform. Some regions of a specimen appeared unetched, while some other regions appeared only partially etched. This variation is likely caused by nonuniform application of the HF gel to the specimen surfaces during the short 20-second etching period. Specimens etched for 40 seconds or 60 seconds appeared more uniformly etched (Fig. 5C, 5D). Additionally, the needle-like crystallites appeared more clearly, likely caused by removal of the additional glassy phase. For the specimens etched for 60 seconds (group E60s), some regions of larger surface pitting were also observed (Fig. 5D).

The effect of returning the LDGC restoration to the furnace for additional refiring at high temperatures after previous etching (group RF) can be seen in Figure 6A. Relative to group E20s, the crystallites appear less clearly, likely resulting from viscous flow of the residual glassy phase at the surface of the specimens. However, refiring did not have the effect of completely removing the original etched surface texture. Re-etching after refiring (group RF-E) reestablished the morphology of the needle-like crystals (Fig. 6B), although pitting like that observed in group E60s was noted. No observable change in crystal size occurred from the extra processing step with a simulated glaze-refiring cycle.

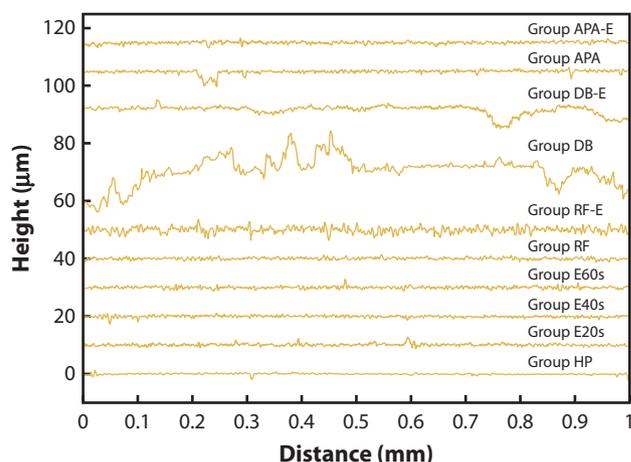


Figure 4. Representative surface profiles according to surface treatment. Profiles vertically offset for visual clarity. Group HP (pressed once and not etched), group E20s (pressed, simulated stain and glaze firing, etched for 20 seconds), group E40s (pressed, simulated stain and glaze firing, etched for 40 seconds), group E60s (pressed, simulated stain, and glaze firing, etched for 60 seconds), group RF (pressed, simulated stain and glaze firing, etched for 20 seconds, simulated addition and glaze refiring, not re-etched), group RF-E (pressed, simulated stain and glaze firing, etched for 20 seconds, simulated addition and glaze refiring, re-etched for 20 seconds), group DB (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by using a diamond rotary instrument, not re-etched), group DB-E (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by using a diamond rotary instrument, re-etched for 20 seconds), group APA (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by airborne-particle abrasion, not re-etched), and group APA-E (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by airborne-particle abrasion, re-etched for 20 seconds).

Adjustment of the LDGC surface by using a diamond rotary instrument (group DB) caused appreciable surface damage and the formation of surface striations (Fig. 6C). However, the extent of surface modification varied among specimens. In some regions, the etched microstructure remained intact, while in other regions, the etched appearance was removed by the action of the rotary instrument. In Figure 6C, the etched microstructure remained on the left side of the image, while the surface region on the right was affected by the diamond rotary instrument. This was likely caused by operator-induced variable pressure of the rotary instrument on the specimen surface. Re-etching the surface after adjustment (group DB-E) resulted in the surface striations becoming somewhat less pronounced, but they were still clearly visible on the surface, indicating that the re-etching process did not completely remove the damage caused by the rotary instrument (Fig. 6D). The needle-like crystals were revealed in some regions, although appreciably less so than for specimens from group E20s.

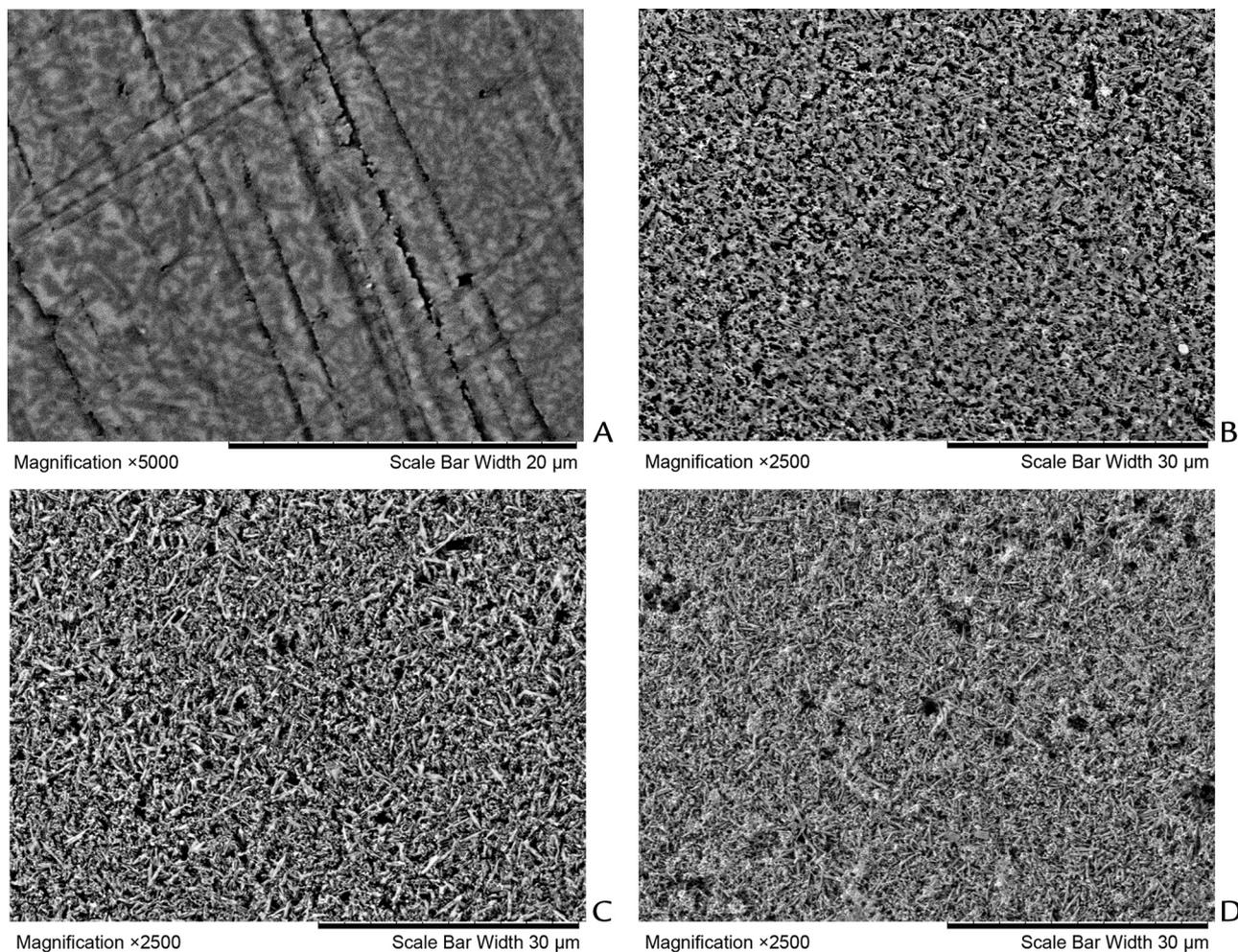


Figure 5. Scanning electron microscopy images of specimens from group HP to group E60s. A, Group HP (pressed once and not etched) original magnification $\times 5000$. B, Group E20s (pressed, simulated stain and glaze firing, etched for 20 seconds) original magnification $\times 2500$. C, Group E40s (pressed, simulated stain and glaze firing, etched for 40 seconds) original magnification $\times 2500$. D, Group E60s (pressed, simulated stain, and glaze firing, etched for 60 seconds) original magnification $\times 2500$.

Airborne-particle abrasion of the surface after etching (group APA) resulted in sporadic surface damage (Fig. 6E). Plate-shaped particles, which were likely the abrasion media, were embedded in the surface of 1 specimen. The surface damage, however, was appreciably less extensive than that caused by diamond rotary instrument adjustment, and the etched character of the surfaces did not appear to be detrimentally affected (Fig. 6E). The surface damage was not removed by re-etching (group APA-E), although the needle-like crystallites appeared slightly more revealed (Fig. 6F) than those of group APA.

DISCUSSION

Based on the qualitative analysis of the SEM images, the null hypothesis that the intaglio surface of a lithium disilicate glass-ceramic can be adjusted

by using any of the investigated surface treatments without affecting the surface topography was rejected.

Spatially nonuniform etching was noted for specimens etched for 20 seconds according to the manufacturer's instructions (group E20s), which was attributed to the possibly uneven application of the etchant. SEM analysis revealed that excessive etching resulted in more extensive removal of the glassy phase and increased needle-like microstructure, while etching for 60 seconds led to a surface-pitting effect. Pitting was also observed in specimens subjected to simulated glaze refiring and re-etching (group RF-E). This phenomenon may originate from formation of regions of slightly higher than average residual glass phase during initial material synthesis because of slightly inhomogeneous crystallization, with preferential etching of these less chemically resistant regions then leading to pitting.

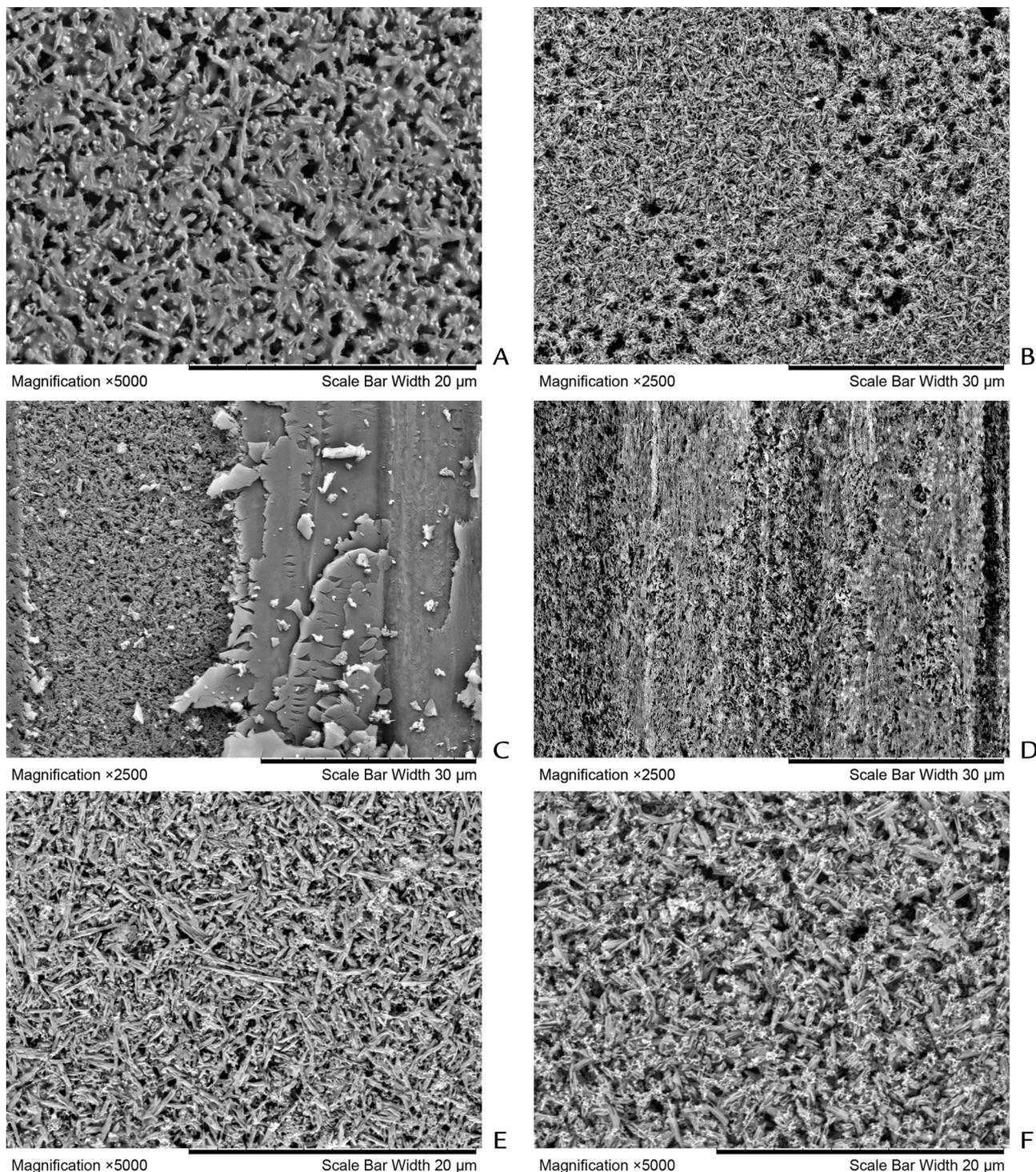


Figure 6. Scanning electron microscopy images for specimens from group RF to group APA-E. A, Group RF (pressed, simulated stain and glaze firing, etched for 20 seconds, simulated addition and glaze refiring, not re-etched) original magnification $\times 5000$. B, Group RF-E (pressed, simulated stain and glaze firing, etched for 20 seconds, simulated addition and glaze refiring, re-etched for 20 seconds) original magnification $\times 2500$. C, Group DB (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by using a diamond rotary instrument, not re-etched) original magnification $\times 2500$. D, Group DB-E (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by using a diamond rotary instrument, re-etched for 20 seconds) original magnification $\times 2500$. E, Group APA (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by airborne-particle abrasion, not re-etched) original magnification $\times 5000$. F, Group APA-E (pressed, simulated stain and glaze firing, etched for 20 seconds, adjusted by airborne-particle abrasion, re-etched for 20 seconds) original magnification $\times 5000$.

SEM analysis (Fig. 6A) revealed an apparent increase in the glassy phase at the specimen surface as a result of refiring (group RF). The apparent increase in peak-to-trough height (Fig. 4) for specimens subjected to refiring followed by re-etching (group RF-E) may have resulted from the flow of the glassy phase from the bulk of the specimen to the surface during refiring and subsequent removal of this phase by re-etching, resulting in an increased depth of etch. This qualitative change in surface topography may affect the resulting bond strength.

Adjustment, particularly with a diamond rotary instrument, has been shown to significantly affect the surface condition of LDGCs.²¹ Based on the results of the present study, the abrasion from the diamond rotary instrument obliterated the previously etched surface that had been prepared for bonding. Re-etching after diamond rotary instrument adjustment is recommended by the authors; however, the etch period (20 seconds) was too short to overcome the damage caused by the rotary instrument to reestablish a suitable surface topography for bonding, and hence, this etch period may therefore need to be extended.

Airborne-particle abrasion, while not recommended by the manufacturers, did not lead to any observed deterioration in the etched character of the specimen surface, although some abrasive media particles may have become embedded in the surface. After airborne-particle abrasion, re-etching may not be required; however, cleaning with an air jet may be necessary to completely remove abrasive media from the surface.

Differences were apparent in SEM images among specimens etched according to the manufacturer's instructions and among specimens which were excessively etched (groups E40s and E60s), refired (group RF), and re-etched (group RF-E); adjusted by using a diamond rotary instrument (group DB) and re-etched (group DB-E); and airborne-particle abraded (group APA) and further re-etched (group APA-E). Although the sample size used in this pilot study was too small to determine whether differences in surface roughness were statistically significant, differences in surface texture were apparent. The quality of the intaglio surface based on how it is prepared could affect the quality of the bond achieved with the composite resin luting agent and also the biaxial flexural strength and reliability of the LDGC restoration. Further in vitro studies could investigate these possibilities.

CONCLUSIONS

Within the limitations of this qualitative pilot study, the following conclusions were drawn.

1. Etching of an LDGC for longer than the period recommended by the manufacturer resulted in a

more revealed microstructure, while etching for 60 seconds led to surface pitting.

2. Refiring after etching deteriorated the as-etched character of the surface, while re-etching reestablished the etched surface topography but led to surface pitting.
3. Adjustment of the intaglio surface by using a diamond rotary instrument resulted in appreciable surface damage and, in some regions, removed the etched character of the surface. Surface damage was not entirely removed by re-etching for a further 20 seconds.
4. Airborne-particle abrasion did not remove the etched character of the surface.
5. Tentative support is given for re-etching an LDGC intaglio surface after refiring in a ceramic furnace at high temperatures and also after diamond rotary instrument adjustment.

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Corresponding author:

Dr Catherine M. Gorman
Division of Restorative Dentistry and Periodontology
Dublin Dental University Hospital, Dublin 2
IRELAND
Email: Catherine.Gorman@dental.tcd.ie

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