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Analysis of Flexural Strength and Contact Pressure After Simulated Chairside Adjustment of Pressed Lithium Disilicate Glass-Ceramic

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Abstract

Statement of problem

Research evaluating load-to-failure of pressed lithium disilicate glass-ceramic (LDGC) with a clinically validated test after adjustment and repair procedures is scarce.

Purpose

The purpose of this in vitro study was to investigate the effect of the simulated chairside adjustment of the intaglio surface of monolithic pressed LDGC and procedures intended to repair damage.

Material and methods

A total of 423 IPS e.max Press (Ivoclar Vivadent AG) disks (15 mm diameter, 1 mm height) were used in the study. The material was tested by using an equibiaxial loading arrangement (n≥30/group) and a contact pressure test (n≥20/group). Specimens were assigned to 1 of 14 groups. One-half was assigned to the equibiaxial load test and the other half underwent contact pressure testing. Testing was performed in 2 parts, before glazing and after glazing. Before-glazing specimens were devested and entered in the test protocol, while after-glazing specimens were devested and glazed before entering the test protocol. Equibiaxial flexure test specimens were placed on a ring-on-ring apparatus and loaded until failure. Contact pressure specimens were cemented to epoxy resin blocks with a resin cement and loaded with a 50-mm diameter hemisphere until failure. Tests were performed on a universal testing machine with a crosshead speed of 0.5 mm/min. Weibull statistics and likelihood ratio contour plots determined intergroup differences (95% confidence bounds).

Results

Before glazing, the equibiaxial flexural strength test and the Weibull and likelihood ratio contour plots demonstrated a significantly higher failure strength for 1EC (188 MPa) than that of the damaged and/or repaired groups. Glazing following diamond-adjustment (1EGG) was the most beneficial post-damage procedure (176 MPa). Regarding the contact pressure test, the Weibull and likelihood ratio contour plots revealed no significant difference between the 1PC (98 MPa) and 1PGG (98 MPa) groups. Diamond-adjustment, without glazing (1EG and 1PG), resulted in the next-to-lowest equibiaxial flexure strength and the lowest contact pressure. After glazing, the strength of all the groups, when subjected to glazing following devesting, increased in comparison with corresponding groups in the before-glazing part of the study.

Conclusions

A glazing treatment improved the mechanical properties of diamond-adjusted IPS e.max Press disks when evaluated by equibiaxial flexure and contact pressure tests.

Clinical Implications

When adjustments are made on the intaglio surface of a pressed lithium disilicate glass-ceramic, a subsequent glazing treatment is recommended to improve strength.

Flaws have been determined to be the main cause of failure of ceramic restorations and may be present at an interface, on the cameo or intaglio surface, or within the bulk of the material.1, 2 In response to a load, stress will concentrate around these defects, and a crack may result.3, 4 Ceramic restorations are fabricated by different techniques, and each technique produces different flaw populations with respect to type, geometry, and distribution.[1] Formation of porosity is commonly observed after pressing different pressable ceramic materials. Guazzato et al[5] found that pressed lithium disilicate glass-ceramic (LDGC) exhibited 3% porosity, while pressed leucite-based ceramics exhibited 9% porosity. During fabrication, reaction with the phosphate-bonded investment material will result in the formation of a reactionary layer. Airborne-particle abrasion and grinding are necessary to remove the reactionary layer, and these procedures may create additional defects,6, 7 which could negatively impact long-term performance.

Abrasive grinding of dental ceramics has been observed to produce cracking and surface defects8, 9, 10 that reduce restoration strength.[6] In a clinical situation, chairside adjustments are frequently necessary to improve seating and the marginal fit of a prosthesis, while adjustment of the cameo surface is often performed to improve occlusion.[11] Transgranular and intergranular cracks and flaws will form in LDGC materials after adjustment with a diamond rotary cutting instrument.[12]

The effect of simulated adjustments on LDGC have been evaluated; however, the testing protocols used did not replicate clinical failure features.6, 13, 14, 15 Ruschel et al[15] evaluated polishing procedures on adjusted LDGC specimens and found that polishing did not improve flexural strength. Adjustment depths were not reported, and a 3-point bend test was used in which strength was largely dependent upon specimen edge finish.16, 17 Hung et al[6] studied simulated clinical grinding of LDGC and subsequent heat treatment on microcrack healing. The result was that diamond rotary cutting tools may introduce flaws and cracks, and subsequent veneer firing or glazing are recommended.[6] A limitation of that study was that a piston-on-3-balls loading arrangement was used, which may lead to contact stresses and crack initiation at one of the balls.[18] Although several studies have considered the effect of etching or bonding on ceramic materials, none have looked at simulated laboratory or clinical adjustments in a clinically validated test.19, 20, 21, 22, 23

The authors are unaware of studies that have investigated the effect of adjustment size on the intaglio surface of LDGC and its effect on strength. Studies that have evaluated after-adjustment repair protocols and their effect on load-to-failure by using clinically validated methods are lacking.24, 25 Therefore, this study considered 4 null hypotheses: no difference will be found in the strength of diamond-adjusted and repaired IPS e.max Press (Ivoclar Vivadent AG) restorations compared with the nonadjusted IPS e.max Press when specimens are in the devested condition; no difference will be found in the contact pressure of diamond-adjusted and “repaired” IPS e.max Press restorations compared with the nonadjusted IPS e.max Press when specimens are in the devested condition; no difference will be found in the strength of diamond-adjusted and repaired” IPS e.max Press restorations compared with the nonadjusted IPS e.max Press when specimens are in the natural glaze condition; and no difference will be found in the contact pressure of diamond-adjusted and repaired IPS e.max Press restorations compared with the nonadjusted IPS e.max Press when specimens are in the natural glaze condition. For this investigation, “repaired” meant that a diamond-adjusted specimen received a natural glaze heat treatment, was acid-etched, or was cemented with a resin cement.

Material and Methods

Pressed LDGC (IPS e.max Press ingot HT, Shade A1; Ivoclar Vivadent AG) disk-shaped specimens were fabricated according to the manufacturer's instructions and subsequently modified (Fig. 1). Failure of ceramics is probabilistic in nature, and consequently a sufficient number of specimens, generally greater than 20, must be tested to reduce statistical uncertainty.26, 27, 28 For the equibiaxial flexural strength test, each group consisted of ≥30 specimens, and for the contact pressure test, each group consisted of ≥20 specimens. Testing was performed in 2 parts: before-glazing specimens were devested and entered the test protocol, while after-glazing specimens were devested and received a natural glaze according to the manufacturer's instructions before entering the test protocol.

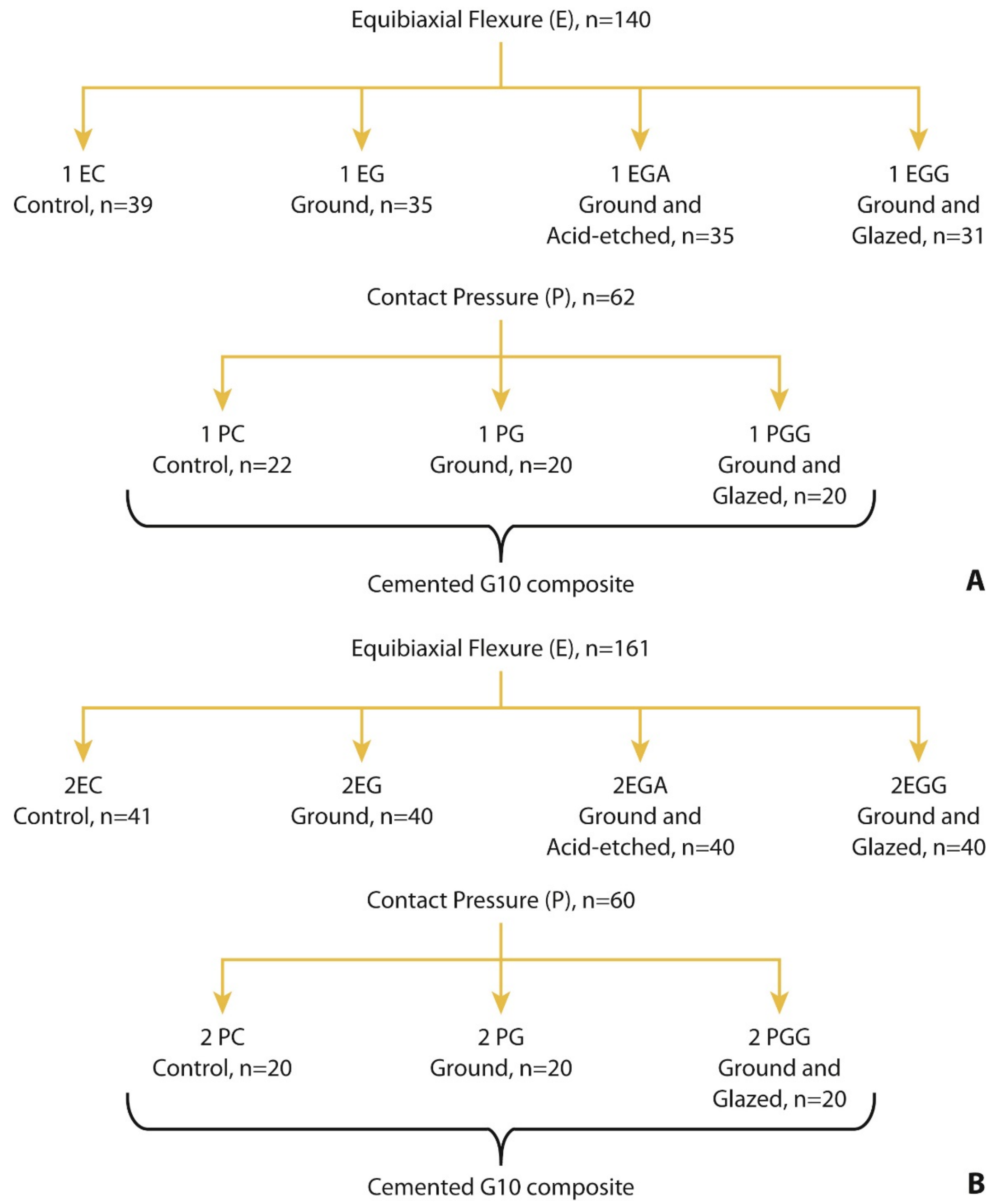

Figure 1. Schematic of experimental design. A, Before glazing. Groups entered test directly after devesting. B, After glazing. Groups received glazing cycle after devesting, and then entered test.

Wax (GEO Classic; Renfert) disks (15×1.0 mm height) were prepared in a metal mold. Upon retrieval, they were inspected under ×10 magnification for voids or other imperfections, and blemished specimens were rejected. Eight-gauge wax (5 mm length) was used to connect the wax patterns to the investment ring base (200g, IPS e.max Investment Ring System; Ivoclar Vivadent AG). Two-hundred grams of phosphate-bonded investment (IPS PressVEST Speed; Ivoclar Vivadent AG) with 32 mL of special liquid (IPS PressVEST Speed; Ivoclar Vivadent AG) and 22 mL of distilled water were mixed for 2.5 minutes in a vacuum mixer. The mixture was poured into the investment ring system to the reference point and allowed to set for 45 minutes. The wax elimination oven was preheated to 850°C, and the investment ring was placed in the preheated furnace (Vulcan Multi-Stage Programmable furnace, 3-130; Dentsply Sirona) facing down toward the rear wall. Upon removal from the oven, an LDGC ingot was inserted into the ring, followed by the Alox plunger. The assembly was positioned at the center of the hot press furnace and pressed.

After cooling to room temperature (60 minutes), the investment was removed by using glass beads at 0.4 MPa pressure followed by glass beads at 0.2 MPa pressure. Hydrofluoric acid (Invex Liquid; Ivoclar Vivadent AG) was used to remove the reactionary layer. Residual reactionary layer was removed with a fine diamond rotary cutting instrument, and sprues were sectioned with a fine diamond disk.

Specimens were assigned to 1 of 14 groups (Fig. 1). One-half was assigned to the equibiaxial load test, and the other half underwent contact pressure testing. The thickness of each specimen was measured at 3 different points near the center of the disk with a digital micrometer (IP65 series 342-27; Mitutoyo), and a mean was determined. Each specimen was overlaid with transparent tape with a 15-mm-diameter circle and center point printed on 1 side to standardize the location of the diamond rotary cutting tool adjustments made on the specimens and to position the specimen in the equibiaxial loading apparatus.

Before-glazing specimens simulated devesting and the fitting of LDGC restorations before glazing. All simulated chairside adjustments to ceramic specimens were performed with a positioning tool and milling machine (AF30; Nouvag). The handpiece was positioned perpendicular to the specimen surface, and depth (0.4 mm) was controlled with the milling machine micrometer. A pilot study determined that diamond-adjustment to a 0.4-mm depth would reliably produce a fracture that went through the milling defect. Shallower depths sometimes displayed fractures that did not originate at or include the damaged area. The adjustments were made at 10 000 rpm by using a diamond rotary cutting instrument (856DEF.016; Brasseler USA) and light pressure. A new diamond rotary instrument was used for each specimen.

1EGA and 1PG received an acid etching treatment (20 seconds, 9.5% hydrofluoric acid; Bisco) on the diamond-adjusted side as recommended for clinical practice. 1EGG and 1PGG were placed in a furnace (Vita Vacumat 500; Vita Zahnfabrik) for glazing after the simulated clinical adjustments. The glazing protocol followed the manufacturer’s recommendations.

Before glazing, equibiaxial flexure strength specimens were centered on a Delrin polymer supporting ring (11-mm diameter). The compressive surface of each specimen was covered with a clear template (0.05-mm thickness) to distribute the load equally and aid in centering the disk on the testing apparatus. The diamond-adjusted side was placed facing down, as it represents the intaglio surface. The specimens were loaded with a Delrin polymer loading ring (5-mm diameter) at 0.5 mm/min in a mechanical testing apparatus (Model 5500R; Instron) with a 5-kN load cell (Instron) until failure and the load was recorded.[14] Before glazing, contact pressure test specimens were cemented to epoxy resin blocks (G10; Ridout Plastics) possessing an elastic modulus similar to dentin. Before cementation, the cementation surface of the block was roughened with 25-µm aluminum oxide for 20 seconds at a distance of 15 mm and 0.28-MPa pressure.

Contact pressure specimens were cemented onto the resin blocks according to the manufacturer's instructions (Multilink; Ivoclar Vivadent AG). Each test piece was treated with 5% hydrofluoric acid for 20 seconds, and then cleaned with water and dried. The etched surface was treated with a universal primer (Monobond Plus; Ivoclar Vivadent AG) for 60 seconds and air-dried. The cementing surface was scrubbed with a 1:1 mixture of self-etching primer for 30 seconds and air-dried. Cement was dispensed onto the treated surface, a 49-N load was placed on the specimen, and the specimen was light-polymerized (Demi Ultra; Kerr Corp).

The compressive surface of the contact pressure specimens received a clear template to equalize contact stresses and assist with positioning the specimen. Specimens were loaded with a 6.5-mm-diameter piston with a 50-mm-tip radius at a cross-head speed of 0.5 mm/min by using a mechanical testing apparatus (Model 5500R; Instron) with a 5-kN load cell until failure.

Contact pressure between the spherical indenter and the surface of the tested material was determined by using the relationship described by Lawn et al,[29]

$$P = (\frac{3E_1}{4kr})^{2/3} \cdot \frac{L^{1/3}}{\pi}, and k = \frac{9}{16}[(1 - v_1^2) + (1 - v_2^2)] \cdot \frac{E_1}{E_2},$$

where P=contact pressure; E_1=elastic modulus of epoxy resin; E_2=elastic modulus of the spherical indenter material; v_1 and v_2 are the respective Poisson ratios; L=applied load; and r=radius of spherical indenter.

Peak loads were identified with a precision-measuring microphone (Model M53; LinearX Systems). An amplitude-versus-time graph was generated with noise analysis software (pcRTA, v2.30; LinearX Systems). In the noise-analysis control panel, the pink noise generator was selected, and an American National Standards Institute A weighted filter was used with the dynamic range fixed between –60 and 120 dBm. The noise analysis was started simultaneously with the contact pressure test. After glazing, specimens (Fig. 1B) were prepared in the same manner as before glazing, with the addition of a natural glaze (manufacturer-recommended firing cycle) after devesting and reactionary layer removal. This simulated a finished laboratory restoration.

Previous work determined that the 2-parameter Weibull distribution with a maximum likelihood curve fitting is best practice for small data sets.[28] The 2-parameter Weibull distribution is characterized by a shape (Weibull modulus, β) and a scaling (characteristic strength, ŋ) parameter, and is estimated from fracture data. A likelihood contour method was used to determine whether 2 Weibull distributions are statistically different (SuperSMITH Weibull 5.08-32 and Super SMITH Visual 5.08-32; Fulton Findings LLC). The plot has the 95% confidence bounds of the estimate for the Weibull shape parameter (β) on the Y-axis and the 95% confidence bounds for the estimate of the characteristic strength (ŋ) on the X-axis. If contour plots intersect, Weibull parameters are not statistically different.26, 28

Results

The Weibull and likelihood ratio contour plots for the equibiaxial flexural strength test on the before-glazing specimens demonstrated a significantly higher failure strength for 1EC than for 1EG, 1EGA, and 1EGG. 1EC exhibited the greatest characteristic strength (188MPa), and 1EGA ranked the weakest (160 MPa). A significant difference was found between 1EG and 1EGG, but no significant difference was found between 1EGA and 1EGG. 1EC possessed the greatest reliability (β=5.5), while the lowest was observed with 1EGG (β=3.3) (Fig. 2). Glazing after diamond-adjustment was the most beneficial after the damage procedure (1EGG, 176 MPa).

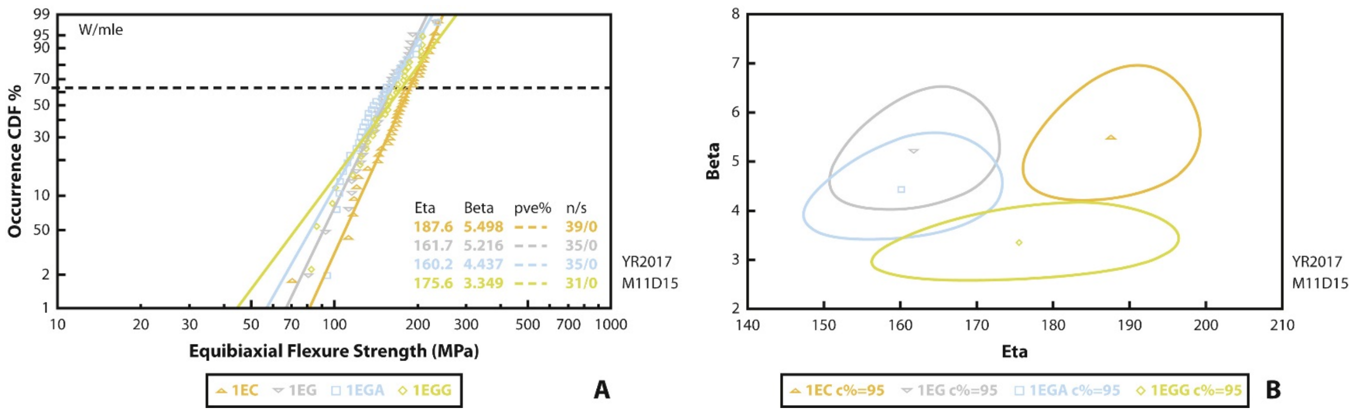

Figure 2. Equibiaxial flexural strength before glazing. A, Two-parameter Weibull plot. B, Likelihood ratio contour plot.

Regarding the contact pressure test, the Weibull and likelihood ratio contour plots revealed no significant difference between the 1PC (98 MPa) and 1PGG (98 MPa) groups. 1PG (95 MPa) exhibited a significantly lower contact pressure than 1PC and 1PGG (Fig. 3).

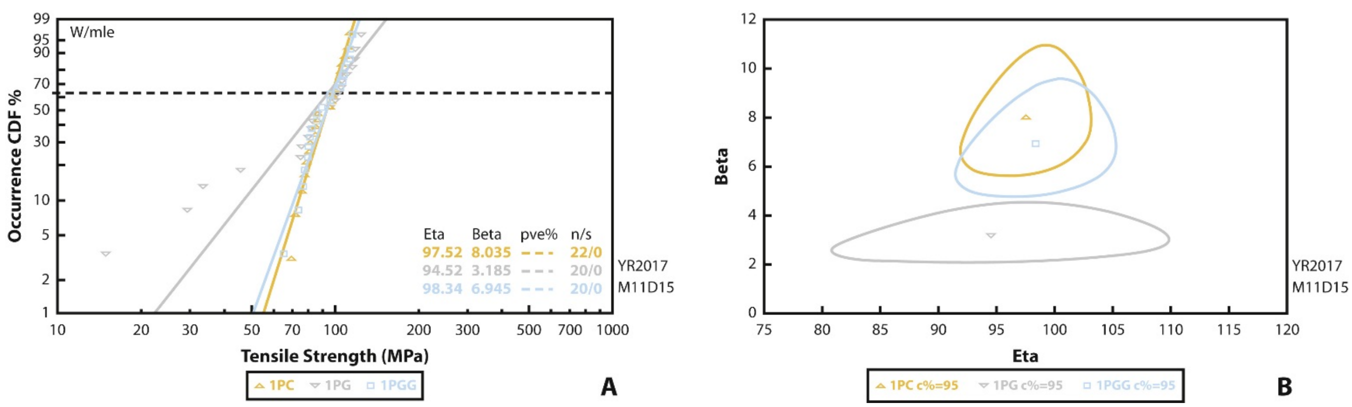

Figure 3. Contact pressure before glazing. A, Two-parameter Weibull plot. B, Likelihood ratio contour plot.

Diamond-adjustment without glazing (1EG and 1PG) resulted in the next-to-lowest equibiaxial flexure strength and the lowest contact pressure. Acid-etching after diamond-adjustment (1EGA) improved Weibull modulus but not failure load.

After glazing, the strength of all the groups when subjected to glazing after devesting increased in comparison with corresponding groups in the before-glazing part of the study. As in before glazing, the equibiaxial flexural strength test demonstrated a significantly higher failure strength for 2EC (240 MPa) than for 2EG, 2EGA, and 2EGG (Fig. 4). A significant difference was found between 2EG and 2EGG, but not between 2EG and 2EGA. 2EC specimens were the most reliable (β=6.8), and the lowest reliability belonged to 2EGG (β=4.1). Subsequent glazing after diamond-adjustment (2EGG) was the most beneficial post damage procedure (204 MPa).

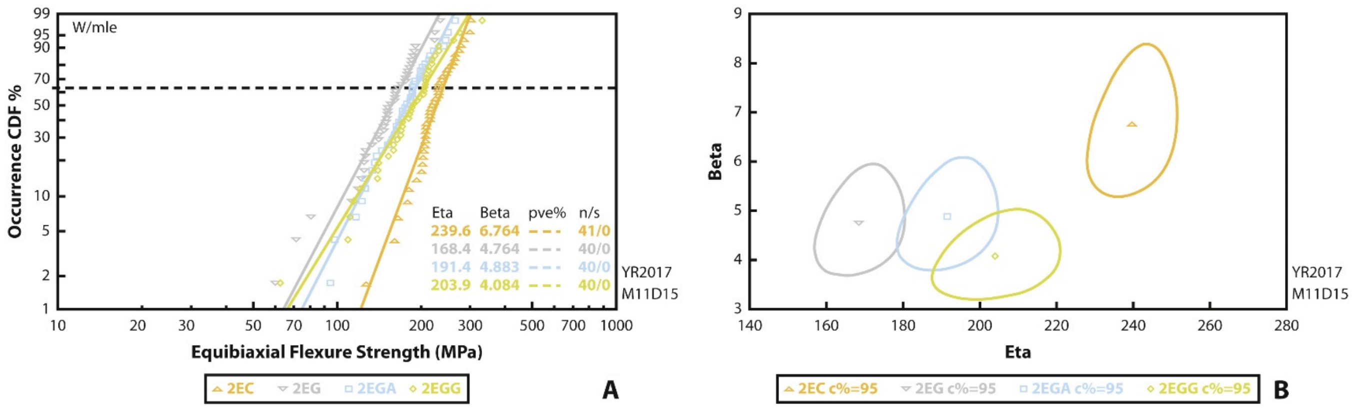

Figure 4. Equibiaxial flexural strength after glazing. A, Two-parameter Weibull plot. B, Likelihood ratio contour plot.

Regarding the contact pressure test, the Weibull and likelihood ratio contour plots revealed no significant difference between 2PC (123 MPa) and 2PGG (124 MPa) or between 2PG and 2PGG (Fig. 5). Diamond-adjustment and no glazing (2EG and 2PG) resulted in the lowest failure loads for both the equibiaxial flexure and the contact pressure tests.

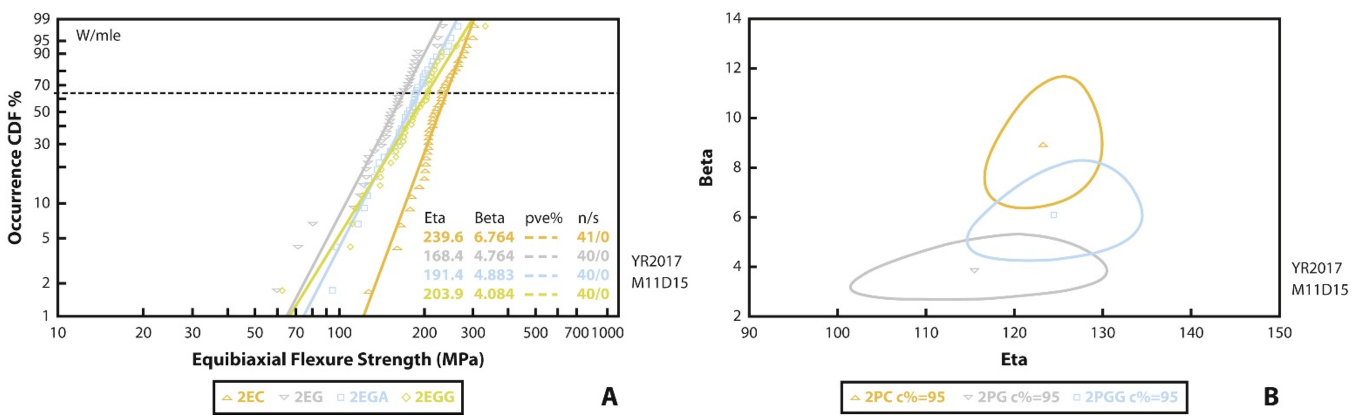

Figure 5. Contact pressure after glazing. A, Two-parameter Weibull plot. B, Likelihood ratio contour plot.

Discussion

The first null hypothesis was rejected because a significant difference was found between the equibiaxial strength of the devested control and the devested, damaged, and repaired specimens (Fig. 2B). The second null hypothesis was not rejected because no statistical difference was found between the contact pressure of the devested control group and the devested, damaged, and repaired groups (Fig. 3B). The third null hypothesis was rejected because a significant difference was found between the equibiaxial strength of glazed control specimens and the glazed, damaged, and repaired specimens (Fig. 4B). The fourth null hypothesis was not rejected because no statistical difference was found between the contact pressure of the glazed control group and the glazed, damaged, and repaired groups (Fig. 5B).

Shrotriya et al[24] investigated the effect of indenter size on cemented ceramic restorations. A small spherical indenter will not reproduce the modes of damage observed clinically, while a large spherical indenter can.[24] Similarly, Kelly et al[25] reported that a large spherical indenter should be used. This investigation used a 50-mm-radius load point, and failure loads were clinically relevant (390 to 800 N).

A ring-on-ring test was used in the present study because it produces an equibiaxial stress state, the load is distributed over a larger area of the specimen and failures from contact stresses are minimized, and equibiaxial tests have not been compared with contact pressure tests.[28] Many load-to-failure tests use a compliant material beneath the load point to reduce stress singularities at the point of contact with specimens. Compliant materials may decrease load-to-failure; more of the load point is in contact with the test specimen, and a critical flaw is more likely to be contained in the stress field.

Glazing improved equibiaxial flexure strength and load-to-failure of the pressed LDGC in general and damaged specimens in particular. The before-glaze equibiaxial test specimens, 1EG and 2EG, exhibited lower strength and reliability after diamond-adjustment compared with 1EC and 2EC (Figs. 2B, 4B). However, when disks received an after-adjustment glaze, 1EGG and 2EGG, these specimens became significantly stronger (Figs. 2B, 4B). Moreover, in a contact pressure test, glazing resulted in damaged-and-repaired specimens (1PGG) that were not significantly different from controls (Fig. 3B).

In the after-glazing part of the study, the Weibull modulus generally increased compared with the before-glazing part, and it is believed that glazing repaired devesting damage. Additionally, magnitude of strength and contact pressure increased significantly compared with before-glazing specimens. Figure 6 shows that glazing after devestment or diamond-adjustment improved load tolerance compared with no glazing, indicating perhaps that manufacturing processes have a significant effect on LDGC material.

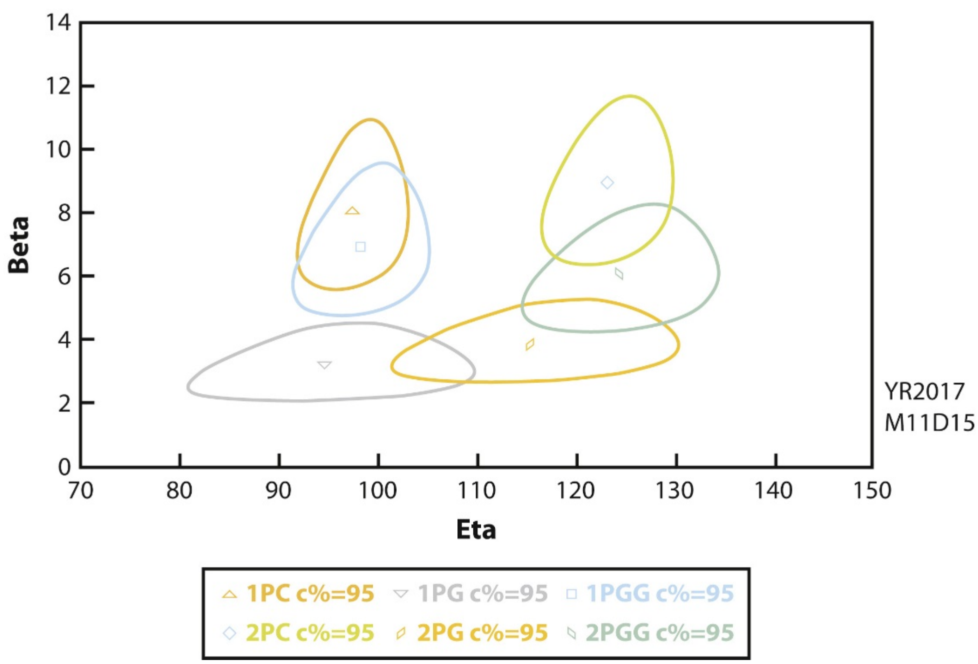

Figure 6. All contact pressure groups before and after glazing.

Statistical outcomes differed depending on the mechanical test used. Results of the equibiaxial flexural strength test showed a significant difference between controls and all other groups before glazing and after glazing (Figs. 2B, 4B). By comparison, no significant difference was found between the controls and diamond-adjusted-and-glazed groups when bonded and tested by contact pressure (Figs. 3B, 5B). The equibiaxial test (315 to 460 N) and contact pressure test specimens (390 to 800 N) failed at similar loads.

Hydrofluoric acid has been shown to increase the surface roughness and consequently weaken LDGC.[19] However, when LDGC is etched and bonded, the strength of the specimen was improved.[20] Bonding with resin cement may improve the performance of adjusted ceramic materials by healing defects and interfering with crack propagation.20, 21, 22 In the present study, simply cementing damaged specimens (1PG, 2PG) without a glazing treatment resulted in Weibull distributions that were significantly different from the controls (1PC, 2PC) in the before-glazing and after-glazing contact pressure tests. Acid etching and resin bonding may be unable to heal critical size defects. Glazing treatments and not bonding may be responsible for restoring groups 1PGG and 2PGG to predamaged strength. The controls in both tests, and for both parts of the study, exhibited the highest Weibull modulus. In addition, groups that received a glazing treatment after adjustment demonstrated a higher Weibull modulus than that of the devested or before-glaze specimens.

The results are related to the specific LDGC pressed material used, and the in vitro study did not simulate oral conditions. That is, fatigue (cyclic loading) or chemical and thermal changes may affect the performance of evaluated materials. In addition, the specimen geometry differs from that of a typical dental restoration.

Conclusions

Within the limitations of this in vitro study, the following conclusions were drawn:

1. A glazing treatment improved the mechanical properties of adjusted IPS e.max Press disks when evaluated with equibiaxial flexural and contact pressure tests.
2. Diamond-adjustments made to lithium disilicate glass-ceramic reduced the reliability of the material. When adjustments are made on the intaglio surface of IPS e.max Press, a subsequent glazing treatment can be recommended.
3. The average load-to-fracture values of the equibiaxial and cemented disks were within the recorded range of human biting forces.
4. Before glazing and after glazing, contact pressure groups displayed a similar rank order in terms of treatment and contact pressure; the control was the strongest, while the diamond-adjusted specimens were the weakest.

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