

RESEARCH AND EDUCATION

Fatigue failure load of feldspathic ceramic crowns after hydrofluoric acid etching at different concentrations



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The long-term success of ceramic restorations is determined by the durability of the adhesive bond and by the mechanical reliability of the ceramic material.¹ Ceramic restorations are constantly subjected to cyclic loads in wet conditions, and the combination of these factors has been identified as one of the main causes of crack initiation and subsequent growth, which may decrease the strength and increase the failure of the restorative materials.^{2,3}

Vitablocs Mark II (Vita Zahnfabrik) are fine-structure feldspar ceramic blocks used to produce inlays, onlays, veneers, and crowns with computer-aided design and computer-aided manufacturing (CAD-CAM) systems. Industrially prepared ceramic blocks are more structurally reliable materials for dental applications, although the machining process may induce flaws.⁴ Vita Mark II has a

relatively low flexural strength of 86.3 MPa, but it has a high Weibull modulus ($m=23.6$).⁴ Clinically, complete crowns of Vita Mark II showed similar survival rates to

ABSTRACT

Statement of problem. Hydrofluoric acid etching modifies the cementation surface of ceramic restorations, which is the same surface where failure is initiated. Information regarding the influence of hydrofluoric acid etching on the cyclic loads to failure of ceramic crowns is lacking.

Purpose. The purpose of this in vitro study was to evaluate the influence of different hydrofluoric acid concentrations on the fatigue failure loads of feldspathic ceramic crowns.

Material and methods. Eighty feldspathic ceramic crowns were cemented with resin cement to identical simplified complete crown preparations machined in a dentin-like polymer. The preparations were etched with 10% hydrofluoric acid for 60 seconds and received a primer coating. Before cementation, the intaglio of the ceramic crowns was treated with 1 of 4 surface conditionings ($n=20$): nonconditioned (control, CTRL), or etched for 60 seconds with different hydrofluoric acid concentrations: 1% (HF1), 5% (HF5), and 10% (HF10). A silane coupling agent was applied on this surface of all crowns, which were cemented to the preparations. Each crown was cyclically loaded in water with a G10 epoxy-glass piston positioned in the center of the occlusal surface. Fatigue failure loads of ceramic crowns were obtained by the staircase approach after 500 000 cycles at 20 Hz. Mean failure loads were analyzed by 1-way ANOVA and the Tukey test ($\alpha=.05$).

Results. Mean failure loads of groups CTRL (245.0 ± 15.1 N), HF1 (242.5 ± 24.7 N), and HF10 (255.7 ± 53.8 N) were statistically similar ($P>.05$), while that of the HF5 group (216.7 ± 22.5 N) was significantly lower ($P<.05$).

Conclusions. HF5 acid had a negative effect on the fatigue loads of the tested feldspathic ceramic crowns, while HF1 and HF10 acids did not change the fatigue resistance. (J Prosthet Dent 2018;119:278-285)

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Clinical Implications

Hydrofluoric acid etching does not have a weakening effect on resin-bonded feldspathic ceramic crowns. However, the clinical use of 5% hydrofluoric acid for etching this ceramic should be considered carefully.

crowns with Vita In-Ceram Spinell copings over a period of 2 to 5 years.⁵ Furthermore, appropriate bonding procedures can improve not only the bond strength but also the fracture strength of CAD-CAM crowns.⁶

During the bonding procedure, the intaglio surface of feldspathic ceramic restorations should be etched with hydrofluoric acid to provide the necessary surface alterations and surface roughness for mechanical interlocking.⁷ These surfaces are further primed with a silane coupling agent for chemical bonding of the ceramic, silane, and resin cement.^{7,8} The experimental evaluation of clinically failed complete ceramic crowns and finite element analysis results suggests that the majority of bulk fractures of single-unit crowns are initiated from the intaglio of the ceramic (cementation surface), where high tensile stresses develop during cyclic loading^{2,9-13} and where hydrofluoric acid etching is performed. Therefore, the internal surface characteristics of ceramic crowns are believed to play a crucial role in their probability of failure.¹⁴

Although hydrofluoric acid etching modifies the intaglio surface of ceramic restorations to promote micro-mechanical interlocking, the impact of this acid on the ceramic strength remains uncertain. It appears to have a weakening effect on glass ceramics¹⁵⁻¹⁸ because of the modification of the resident surface flaw population,¹⁵ with a progressive increase in this effect as a function of the hydrofluoric acid concentration used for etching.^{16,19} With higher hydrofluoric acid concentrations, the increased number of defects can be attributed to newly introduced flaws.¹⁶ If these flaws reach a critical size, they can propagate on the internal surface and cause ceramic failure. Thus, the flaw distribution present in the material is directly related to the fracture strength of the ceramic.²⁰ However, some studies have reported that hydrofluoric acid in different etching regimens did not negatively impact the strength of ceramics^{21,22} and that unfilled resin or resin cement application had a positive effect on flexural strength after hydrofluoric acid etching and silane treatment,^{14,22} minimizing the influence of flaws.²³ The strengthening effect occurs as a consequence of the interaction of the resin with the entire surface defect population²⁴ and may depend on the behavior of the resin penetrating the ceramic surface.²⁵

In addition, this acid is also known as a chemical with extremely hazardous effects because of its toxicity.²⁶

Therefore, potential damage to health has motivated dental research to test low hydrofluoric acid concentrations.²⁷

The performance of complete ceramic crowns is determined by a complex combination of factors including the material selected, thickness, damage introduced, adhesive/luting system used, tooth substrate (natural dentin or foundation restoration²⁸), and the fatigue response to complex loading.²⁹ In terms of failure prediction, cyclic mechanical loads applied under wet conditions could simulate the damage accumulation that occurs in ceramic restorations in an oral environment³⁰ and can lead to modes of crack initiation and growth not seen under monotonic loads.³¹ However, the authors are unaware of studies that did not perform monotonic tests to evaluate the effect of hydrofluoric acid etching on the ceramic surface, which can be less clinically relevant than fatigue failure tests. Likewise, the authors are unaware of studies that tested the influence of the restoration geometry on stress distribution or the cementation effects, such as flaw "healing" by resin cement. In addition, the influence of dentin elastic properties and the manufacturing method of the ceramic specimens—related to population defects—need to be studied. Currently, studies that replicated clinical situations that have assessed the influence of hydrofluoric acid etching in different concentrations on the fatigue failure loads of adhesively cemented feldspathic ceramic crowns are lacking.

Therefore, the purpose of this *in vitro* study was to evaluate the influence of different hydrofluoric acid concentrations on the failure loads of feldspathic ceramic crowns machined by CAD-CAM systems using wet mechanical cyclic tests. Two hypotheses were tested: acid etching would not reduce the fatigue failure loads in comparison with untreated crowns, and mean failure loads would not be influenced by the different hydrofluoric acid concentrations.

MATERIAL AND METHODS

The design of a simplified complete crown was adapted from one previously developed by Gressler May et al.³⁰ They observed that fractures were initiated from the cementation surface of feldspathic crowns. Furthermore, the lack of contact damage on the loaded surface was attributed to the use of a flat-end piston made from G10 epoxy glass.

Eighty identical dentin analog prosthetic preparations (preparation height=5.32 mm, internal angle radii=0.5 mm, axial wall convergence=16 degrees, cervical preparation depth=1.2 mm, and round shoulder radii=0.5 mm) were machined from 11 mm diameter rods of an epoxy-glass cloth (NEMA G10; International Paper) in a mechanical lathe (Diplomat 3001; Nardini), as seen in Figure 1A. One G10 preparation was scanned, and the 3-dimensional images were processed in CAD software

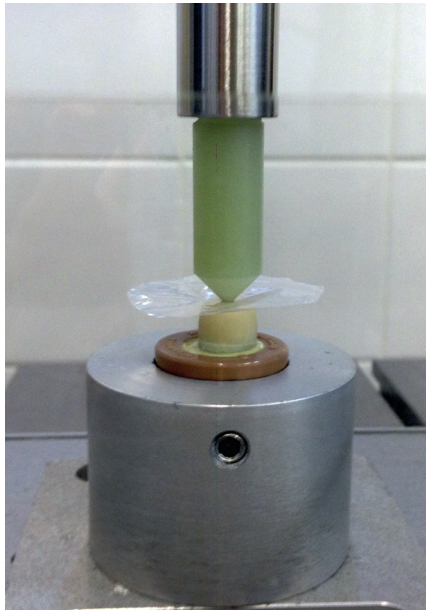


Figure 2. Experimental arrangement. Specimen centrally loaded with G10 piston in fatigue machine.

Sinusoidal cyclic loading was applied to ceramic specimens, with amplitudes ranging from a minimum of 10 N to the maximum tensile load, at a frequency of 20 Hz, and for 500 000 cycles. The initial load and the step size were determined based on the results of the monotonic tests from the 10% hydrofluoric acid (HF10) group ($n=3$; mean monotonic load for fracture=300 N). A load that was 60% of the mean monotonic failure load was assumed as the initial load (180 N). A step size of 20 N was applied up or down to the next specimen, according to the examination for subsurface crack formation by transillumination. If the tested specimen failed, the next specimen was cycled at a lower load by decreasing 1 step size. If the specimen survived the 500 000 cycles, the subsequent specimen was cycled at a higher load by increasing 1 step size.

The mean failure load (L_f) and the standard deviation (s) were calculated on the basis of the data of the least frequent event (survival or failure) by using the method described by Collins.³²

$$L_f = L_{f0} + d \left[\sum in_i / \sum n_i^{\pm 1/2} \right] \quad \text{Eq. (1)}$$

$$S = 1,62d \left\{ \left[\left(\sum n_i \sum i^2 n_i - (\sum in_i)^2 \right) / (\sum n_i)^2 \right] + 0,029 \right\} \quad \text{Eq. (2)}$$

$$\text{if : } \left[\left(\sum n_i \sum i^2 n_i - (\sum in_i)^2 \right) / (\sum n_i)^2 \right] \geq 0.3,$$

Table 2. Mean fatigue failure loads (L_f) and standard deviation (SD) of CAD-CAM feldspathic crowns from staircase tests

Group	L_f (N) (\pm SD)
CTRL	245.0 \pm 15.1 ^A
HF1	242.5 \pm 24.7 ^A
HF5	216.7 \pm 22.5 ^B
HF10	255.7 \pm 53.8 ^A

CAD-CAM, computer-aided design and computer-aided manufacturing. Different superscript letters indicate statistically significant differences ($P < .05$).

where L_{f0} is the lowest load level considered in the analysis, d is the step size, and n_i is the number of failures or survivals at the given load level. In Eq. (2), the negative sign is used if the least frequent event is a failure; otherwise, the positive sign is used. The lowest load level considered is designated as $i=0$, the next level as $i=1$, and so on. n_i is the number of failures or survivals at a given load level.

Topographical analysis was performed by field emission scanning electron microscopy (FE-SEM) (FEI Inspect F50; FEI) at different magnifications. For this analysis, a machined crown was sectioned into 4 pieces. Each piece was treated using different conditioning methods, and the occlusal surfaces of cementation (internal surface) were sputter coated with a gold-palladium alloy before being examined.

After the fatigue test, the crowns were analyzed under a light microscope (Stereo Discovery V20; Carl Zeiss) to determine the region of crack origin. Then the failure crowns were longitudinally sectioned into halves perpendicular to the track of the radial crack. Representative specimens from each group were analyzed by FE-SEM.

Statistical analysis was performed using statistical software (IBM SPSS Statistics for Windows v21; IBM Corp). All load values (failure or survival steps) were analyzed by 1-way ANOVA and the post hoc Tukey test ($\alpha=.05$) because the data presented homogeneity of variances ($P>.05$ based on the Levene test) and normal distribution ($P>.05$ based on the Shapiro-Wilk test).

RESULTS

Significant differences were found among the groups (ANOVA, $P<.001$). The crowns etched by 5% hydrofluoric acid (HF5) had a lower mean fatigue failure load than that of the CTRL, 1% hydrofluoric acid (HF1), and HF10 groups, which were statistically similar (Table 2). The patterns of runouts (survivals) and failures from the staircase experimental design for the different groups after 500 000 cycles are shown in Fig. 3.

The FE-SEM analysis revealed that slight topographical changes were promoted by HF1 compared with

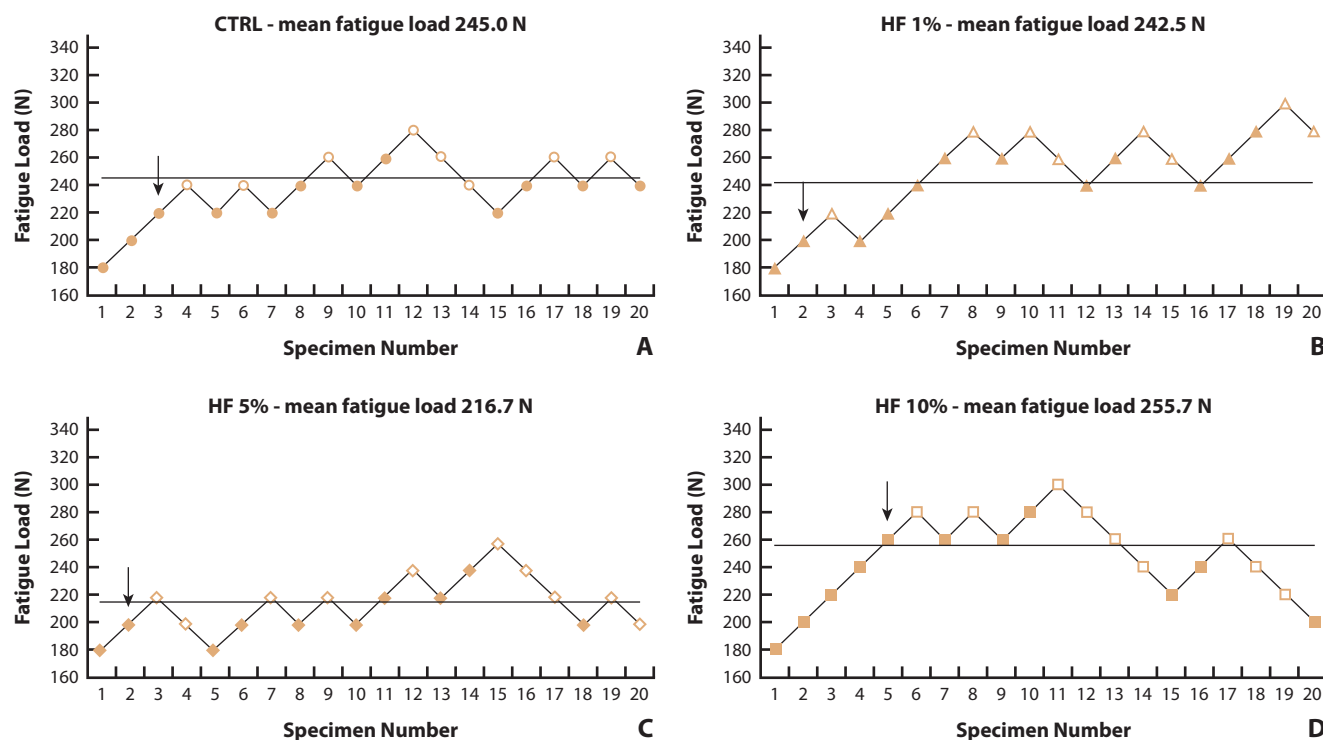


Figure 3. Pattern of runouts (survival) and failures for each group (CTRL, HF1, HF5, HF10) observed during wet mechanical cycling (500 000 cycles). Arrows indicate load level at which up-and-down character started. Horizontal lines indicate mean fatigue load, filled scorers show runout, and empty scorers are failure.

the untreated condition (CTRL) (Fig. 4). A progressive effect of the different hydrofluoric acid concentrations was observed on the ceramic microstructure, indicating that higher concentrations promoted larger and deeper craters and pits.

The crown failure analysis under a light microscope showed that all fatigue cracks were radial cracks starting from the cemented surface, and there was no Hertzian cone crack. Representative FE-SEM micrographs of the fracture surfaces are presented in Fig. 5. The irregularities created by acid etching on the ceramic surface were often not filled by resin cement, especially in the HF5 group.

DISCUSSION

The first hypothesis—that the acid etching would not reduce the fatigue failure loads in comparison with untreated crowns—was partially accepted. Mean fatigue failure loads were significantly lower when ceramic surfaces were etched with HF5, while the groups CTRL, HF1, and HF10 were not statistically different (Table 2). The second hypothesis was rejected because the fatigue failure loads were influenced by the distinct hydrofluoric acid concentrations.

Malament and Socransky²⁸ reported that the intraoral survival rates (Kaplan-Meier) of glass-ceramic crowns (Dicor) over 16 years were higher when they were acid

etched before being bonded to the dentin cores, as opposed to not being acid etched. Of the luting agents tested in that study, acid-etched Dicor restorations luted with composite resin exhibited more favorable survivor functions than restorations luted with glass ionomer or zinc phosphate cement.²⁸ These data highlight the importance of resin-bonded adhesion and topographical changes promoted by hydrofluoric acid etching to achieve better clinical results.

Nevertheless, hydrofluoric acid can be harmful and particularly aggressive to soft tissues.²⁶ Considering the potential hazards of hydrofluoric acid in dental applications, low concentrations of this acid have been studied to promote durable bond strengths without weakening the ceramics. Venturini et al²⁷ tested 4 hydrofluoric acid concentrations for etching a feldspathic ceramic (Vita Mark II), concluding that 3%, 5%, and 10% hydrofluoric acid promoted stable resin adhesion after long-term aging, while 1% hydrofluoric acid showed a significant decrease in bond strength after aging/thermocycling. In another study,¹⁸ they tested the effect of the same hydrofluoric acid concentrations on the flexural strength and reported that acid etching has a weakening effect on feldspathic ceramic when compared with untreated ceramic, regardless of its concentration. Regardless of these previous monotonic findings, the mean fatigue failure loads were not different among the groups CTRL,

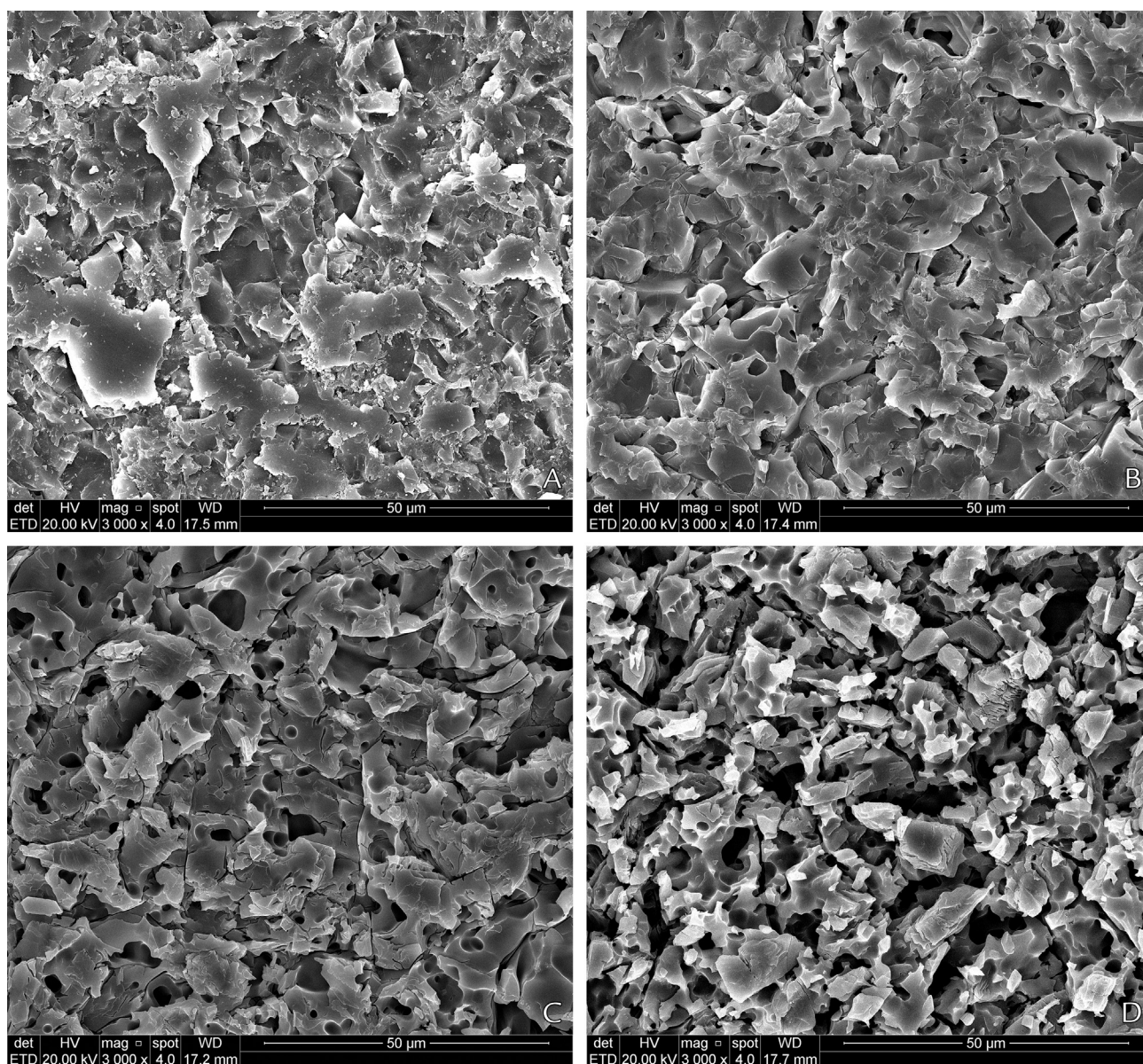


Figure 4. Representative field emission scanning electron microscopy micrographs (original magnification $\times 3000$) of different ceramic surface treatments showing topography pattern alteration generated by machining procedure and hydrofluoric acid etching. A, CTRL group. B, HF1 group. C, HF5 group. D, HF10 group.

HF1, and HF10 in the current study. The explanation for this could be the cementation procedure, which provides support to the ceramic crown by a cement layer. A positive influence of the resin cement application on the ceramic flexural strength has been proposed by some authors.^{22,24,25} The theory of resin strengthening ceramics may be explained by the combination of the Poisson constraint and the creation of a resin interpenetrating layer sensitive to the elastic modulus of the resin.²⁵ A resin layer that is bonded to the flawed surface would change the ceramic material to a ceramic-composite resin.¹²

Etching the cementation surface and bonding with a low-viscosity resin cement can minimize the influence of flaws at cementation surfaces.²³ However, crowns etched with 5% hydrofluoric acid were significantly less resistant than the other groups tested here. The FE-SEM fractography images (Fig. 5) show nonhomogeneous penetration of the resin cement into the ceramic irregularities created by 5% hydrofluoric acid through the cement voids present at the interface, mainly in the failure origin area. The presence of a large flaw and cement voids along the internal surface of a glass-ceramic crown may raise internal stresses, leading to failure.¹² Under loading, the

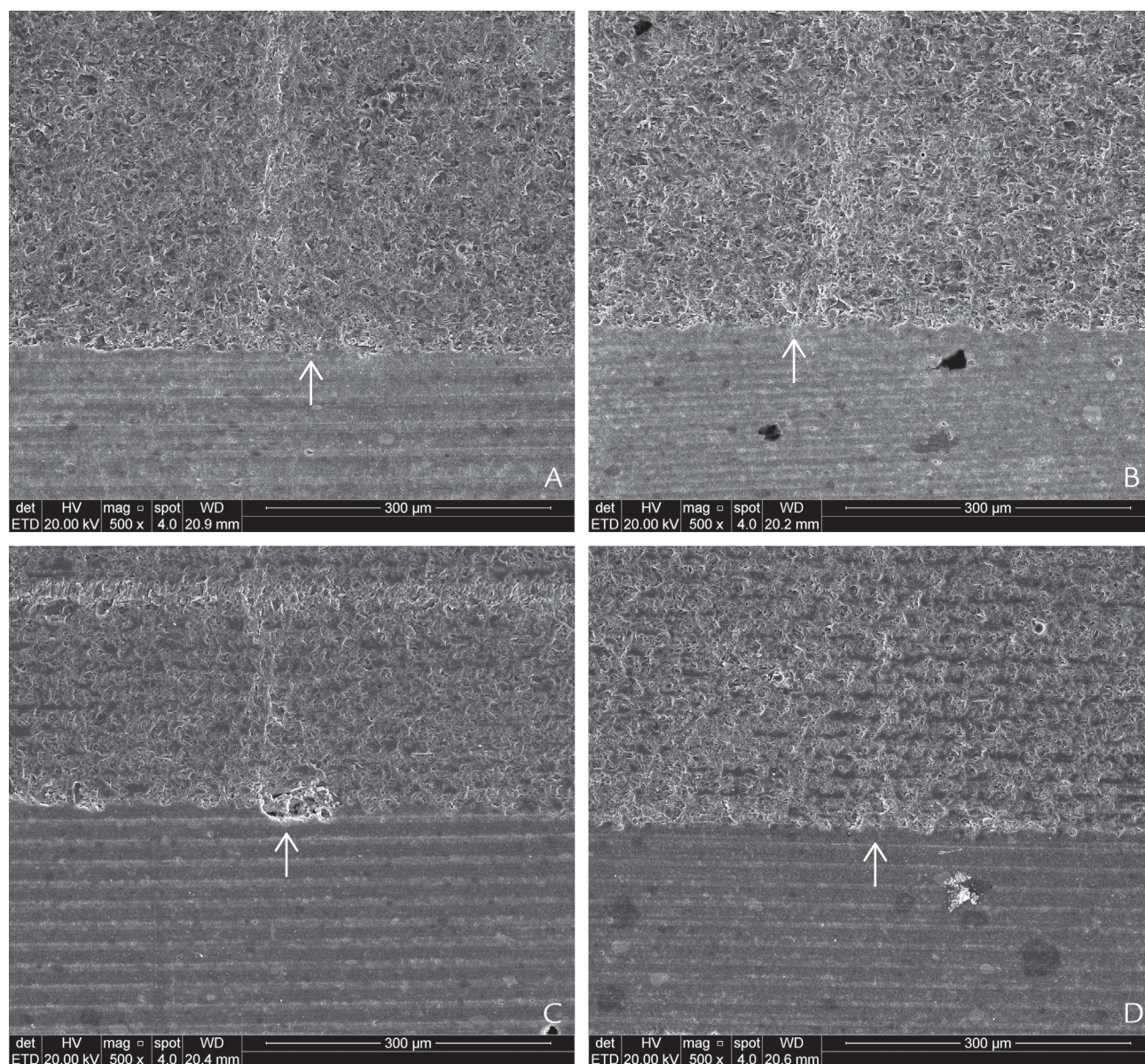


Figure 5. Representative images (original magnification $\times 500$) of fracture surfaces from crowns subjected to fatigue. A, CTRL group. B, HF1 group. C, HF5 group. D, HF10 group. Radial cracks pointing to fracture origin can be observed in cementation surface under tensile stresses.

stress at the flaw tip in an air space cannot be transferred to the resin, thereby increasing the susceptibility of the ceramic to crack initiation.¹² The resin cement viscosity is also important, as more fluid resin cement could penetrate irregularities on the intaglio ceramic surface.

Previous investigations of clinically failed glass-ceramic crowns revealed that the majority of failures were initiated from flaws and tensions existing at the cementation surface, indicating this surface as the location of the highest tensile stress and/or the largest flaws.^{9,11,13} In the current study, all failures occurred as radial cracks from the cementation surface, which approximate to the clinical failure reports. In addition, the use of a flat-end piston made from G10 epoxy glass and a

plastic strip between the piston and the crown during the test may justify the absence of contact damage on the loaded occlusal surface.

The load to initiate a radial fracture is influenced by the crown thickness and the relative elastic modulus between crown and the supporting tooth substrate.²⁹ Hence, the current study used a crown design with a simple geometric configuration because an irregular geometry makes it difficult to determine the effect of crown thickness. In addition, the substrate supporting material was an epoxy filled with woven glass fibers (G10), which has an elastic modulus similar to that of dentin. Kelly et al¹⁰ reported that G10 was not significantly different from hydrated dentin in terms of blunt contact elastic

behavior or resin cement bond strength. They found a small difference (approximately 5%) in fatigue behavior when testing was performed at 20 Hz versus 2 Hz and concluded that further tests can be performed at 20 Hz, greatly increasing the rate of data accumulation. Therefore, the current fatigue test was performed at 20 Hz.

Regarding the fatigue test, the staircase approach was used to determine the fatigue strength; it involved subjecting specimens to a high number of cycles at a low load. At least 15 specimens were necessary to evaluate the fatigue strength at the life of interest.³² Coefficients of variation of approximately 10% were achieved by Kelly et al,¹⁰ with a modest number of specimens (≤ 20) in a staircase sensitivity testing. Because of its low variability, a sample size of 20 crowns was used in the present study.

There are limitations in this in vitro study, and some clinical conditions were not simulated. The direction of the load application was only axial, without simulation of lateral forces and sliding that may occur clinically during mastication or clenching. Furthermore, the brief water storage times (7 to 14 days), without long-term aging, might be insufficient to damage bond strength. The chemical bond between silane and resin cement might deteriorate over time because of hydrolysis, and the micromechanical retention promoted by the minimal acid etching (HF1) or nonacid etching (CTRL) may be insufficient to provide stable bond strength. Therefore, the adhesion in CTRL and HF1 may have been promoted mainly by chemical bonds, with minimum micromechanical bonding. Thus, these findings should be considered carefully, and future studies should stress the clinical plausibility of using lower concentrations of hydrofluoric acid.

CONCLUSIONS

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

1. Etching with 5% hydrofluoric acid on the intaglio surface of feldspathic ceramic crowns reduced the fatigue failure loads and thus would not be recommended for the tested ceramic.
2. The mean fatigue failure loads of the feldspathic crowns was not influenced by 1% and 10% hydrofluoric acid, since these etched crowns did not differ from untreated crowns.

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