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Polymer Infiltrated Ceramic Network Structures for Resistance to Fatigue Fracture and Wear

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Abstract

Objective—To investigate fatigue fracture resistance and wear behavior of a polymer infiltrated ceramic network (PICN) material (ENAMIC, Vita Zahnfabrik).

Methods—Anatomically shaped ENAMIC monolithic crowns were milled using a CAD/CAM system. The crowns were cemented on aged dentin-like composite abutments (Z100, 3M ESPE) with resin-based cement (Vita DUO Cement, Vita). The specimens were subjected to 2 types of fatigue and wear tests: (1) accelerated sliding-contact mouth-motion step-stress fatigue test ($n = 24$) in water; and (2) long-term sliding-contact mouth-motion fatigue/wear test using a clinically relevant load ($P = 200$ N, $n = 8$) in water. Failure was designated as chip-off or bulk fracture. Optical and scanning electron microscopes were used to examine the occlusal surface and subsurface damage, as well as to reveal the material's microstructure. In addition, wear volume and depth were measured by x-ray micro-computed tomography.

Results—For accelerated mouth-motion step-stress fatigue testing, 3 out of the 24 ENAMIC crowns fractured following cyclic loading up to 1700 N. Minor occlusal damage and contact-induced cone cracks were observed in all surviving specimens, but no flexural radial cracks were seen. For long-term mouth-motion fatigue/wear testing under a 200 N load in water, a small wear scar without significant cracks was observed in all 8 tested ENAMIC crowns.

Significance—Monolithic CAD/CAM ENAMIC crowns showed superior resistance to sliding-contact fatigue fracture and wear.

Keywords

Polymer infiltrated ceramic network; CAD/CAM crowns; fatigue fracture; wear; damage modes

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1 Introduction

A polymer infiltrated ceramic network (PICN) material – VITA ENAMIC (Vita Zahnfabrik, Bad Säckingen, Germany), in which each phase is topologically interconnected throughout the microstructure has been recently launched in the dental CAD/CAM market. Although ENAMIC is so far the only dental material commercially available in this category, the concept of interpenetrating microstructures was first introduced to the field of dental materials in the mid-1980s [1,2]. This has led to the development of the In-Ceram systems (Vita Zahnfabrik), which consist of a porous ceramic network (alumina, zirconia toughened alumina, or spinel) that is infiltrated with a low viscosity glass. The In-Ceram restorative systems have been used successfully for 25 years [1–5]. However, the transition from glass infiltration to polymer infiltration of a porous ceramic network has taken quite a long time with a lot of efforts [6,7].

The ENAMIC material consists of 86 wt.% or 75 vol.% inorganic phase and 14 wt.% or 25 vol.% organic phase. The inorganic phase is a feldspathic ceramic, whereas the organic phase is composed of dimethacrylates (UDMA and TEGDMA). The three dimensional interconnected dual network structure [8] differs from traditional composites in which only the matrix phase is continuous [9]. The ceramic network provides resistance to deformation and wear, but it is brittle and susceptible to fracture. The ductile polymer network, capable of undergoing plastic deformation, yields resistance to fracture [10]. PICN materials combine enhanced resistance to distinct damage modes, exhibiting physical properties that are different and often superior than conventional composites [11].

A lot of attention has been drawn to developing and improving PICN materials since the release of ENAMIC in 2013 [10,12–16]. A comprehensive review of this class of materials has been recently published by Swain and collaborators [17]. The authors have concluded that PICN materials present a combination of low hardness and modulus coupled with higher toughness and R-curve behavior than glass-ceramic materials, enabling rapid milling with minimal chipping at the margin/edge, as well as the ability to cope with severe occlusal contact loading. However, the authors have also indicated that no information regarding wear and fatigue behavior of PICN was available. It has been discussed that resistance to fatigue fracture and wear damage are important determinants of the clinical longevity of direct and indirect dental restorations [18–20].

Accordingly, the aim of this study is to elucidate the resistance to fatigue fracture and wear damage of monolithic ENAMIC molar crowns by means of accelerated sliding-contact mouth-motion step-stress fatigue test and long-term fatigue/wear test with a clinically relevant load in water.

2 Materials and Methods

2.1 Specimens fabrication

Anatomically correct mandibular first molar crowns were designed and milled from VITA ENAMIC CAD/CAM blocks, using a CEREC System (Sirona, Charlotte, NC, USA). A standard die of a mandibular first molar preparation was scanned into the system and

adjusted to compensate for the cement layer thickness (50 μm) [21]. Tooth preparation was modeled by reducing the proximal walls by 1.5 mm and the occlusal surface by 2.0 mm. The anatomically correct crown specimens were polished using the VITA ENAMIC technical polishing set (Vita Zahnfabrik), as recommended by the manufacturer. A single operator performed all polishing procedures with the aid of a magnification loupe (4.5 magnification Zeiss, Jena Germany).

For ENAMIC restorations, which cannot be placed in the oven for traditional glazing treatments, fine polishing after milling is a very important step to reduce the roughness and population of surface defects. It is well-established that fatigue and wear behavior are governed by material properties, surface characteristics, contact geometry, and the interfacial friction coefficient [22,23]. Polishing also reduces the friction between the contacting surfaces [23]. In surfaces subjected to cyclic sliding, pre-existing defects act as stress-concentrators and can become the origins of cracks. Propagation of these cracks is associated with mechanical fatigue, such as hydraulic pumping, as well as moisture-assisted slow crack growth [18,24,25].

All crowns were cemented to aged resin-based composite dies (Filtek Z100, 3M ESPE, St. Paul, MI, USA), which were stored in distilled water at 37°C for 3 to 5 weeks. Z100 was selected as abutment material because it has an elastic modulus of 18 GPa [24], which is similar to the elastic modulus of 16 – 18 GPa for human dentin [26,27]. The effective elastic moduli of the supporting abutment and luting cement were found to play a governing role in the fracture resistance of dental crowns [26–29].

All cementation procedures followed the manufacturers' instructions: Z100 abutments were thoroughly cleaned with ethanol and air-dried, followed by application of VITA A.R.T. BOND adhesive (Vita Zahnfabrik), rubbing it for 20 s and air drying for 5 s. The crowns were cleaned in ultrasonic bath and air-dried. The inner surfaces were then thoroughly cleaned with ethanol and air-dried. A 5% hydrofluoric acid gel (VITA CERAMICS ETCH, Vita Zahnfabrik) was applied to the inner surfaces of the crowns for 60 s, rinsed in tap water, and air dried for 20 s. VITASIL (Vita Zahnfabrik) silane coupling agent was applied to the etched surface, followed by 60 s waiting time, and air drying. The adhesive VITA A.R.T. BOND was rubbed on the cementation surface for 20 s and air-dried. VITA DUO CEMENT (Vita Zahnfabrik) 1:1 base & catalyst pastes were mixed and applied to the inner surface of the crown. The crowns were then pressed onto the abutment by hand. Caution was taken to ensure that marginal gaps were closed uniformly. Excess cement was chipped away after 2–3 s light curing. Photopolymerization of the luting agent was carried out by exposing each surface of the crown to a dental curing light with an irradiance of 850 mW/cm² for 40 s (Ultra Lume LED 5, Ultradent Products Inc, South Jordan, UT, USA). All cementation procedures were carried out by a single operator. After cementation, the specimens were stored in distilled water at 37°C for 5 to 7 days for continued polymerization and the hydration of the cement layer prior to mechanical testing.

2.2 Mechanical testing

Fatigue and wear resistance of ENAMIC was tested by means of two methods: (1) accelerated sliding-contact mouth-motion step-stress fatigue test ($n = 24$) and (2) long-term

sliding-contact mouth-motion fatigue/wear test using a clinically relevant load ($n = 8$). Crowns were immersed in distilled water during all tests.

To determine the loading cycling profiles that constitute the accelerated sliding-contact mouth-motion step-stress fatigue test, four crowns were subjected to single cycle load to failure test [30,31]. The crown/tooth replica assembly was mounted in a universal testing machine (Model 5566, Instron, Canton, MA, USA), and load was applied axially at the central fossa of the occlusal surface of the crown through a tungsten carbide (WC) spherical indenter ($r = 3.18$ mm). Specimens were loaded monotonically at a constant rate of 1 mm/min until failure.

Accelerated sliding-contact mouth-motion step-stress fatigue test were used to determine cyclic fatigue behavior of ENAMIC. Three step-stress profiles (mild, moderate, and aggressive) were designed [31] based upon results from the single load to fracture test. Crowns were distributed across the three profiles in the ratio of 4:2:1 for mild ($n = 12$), moderate ($n = 6$), and aggressive ($n = 3$) profiles, respectively. The mild profile starts at 400 N indentation load and goes to 1200 N at 170k cycles; the moderate profile starts at 500 N and goes to 1400 N at 120k cycles; and the aggressive profile at 600 N and goes to 1700 N at 70k cycles. Three additional ENAMIC crowns were subjected to the extended aggressive profile with load up to 1700 N and 300k cycles (including 30k at 1700 N). It is well-established that ceramic [32] and composite [33] materials are sensitive to load/stress rate. Therefore, a clinically relevant loading rate (1000 N/s) was used [34,35]. As a result, the loading frequency, including load, slide, and lift-off phases, varied from 0.3 Hz at 1700 N to 2 Hz at 200 N. The crowns were subjected to accelerated step-stress fatigue by sliding a spherical WC indenter ($r = 3.18$ mm) 0.7 mm down the distobuccal cusp towards the central fossa, using an electrodynamic fatigue testing machine (EL-3300, Enduratec Division, TA Instruments, Minnetonka, MN). At the end of each load cycle, all specimens were inspected under a polarized light stereomicroscope for cracks and damage. Bulk fracture and chip-off fracture of the crowns were considered as failures.

In an attempt to simulate loading conditions relevant to those occurring during chewing, 8 crowns were subjected to long-term sliding-contact mouth-motion fatigue/wear by sliding a spherical steatite indenter ($r = 3$ mm) 0.7 mm down the distobuccal cusp towards the central fossa, using the electrodynamic fatigue testing machine at a maximum load of 200 N and 2 Hz frequency for 1.25 million cycles in water. These fatigue testing conditions are believed to simulate 5 years of clinical service [36,37].

A steatite indenter was chosen for the long-term sliding-contact mouth-motion fatigue/wear test because it presents physical properties in the midrange of those of dental porcelains, glass-ceramics, and dental enamel [38]. Therefore, simulating the wear of the crown and its antagonist would undergo clinically relevant conditions. On the other hand, a hard WC indenter was used for accelerated step-stress test in order to continually apply higher loads (stresses) during cyclic sliding, avoiding contact wear. Relatively soft indenter materials like steatite would undergo greater wear, increasing the contact area as the number of cycle increases, thus decreasing contact pressure, which goes against the fundamentals of the step-stress testing method. A WC indenter was also used for the single cycle load to failure test,

since the results of the latter are needed to guide the design of step-stress profiles. The radii of both steatite and WC indenters (3 mm and 3.18 mm, respectively) were in the midrange of the molar cuspal radii (2 – 4 mm) [39].

2.3 Characterization of Fatigue and Wear Damage

2.3.1 Microstructural analysis—For better understanding of the material fatigue and wear behavior, a scanning electron microscope (SEM – S3500N, Hitachi, Tokyo, Japan) was employed to analyze the microstructure of ENAMIC. Energy-dispersive x-ray spectroscopy (EDX) was used for elemental and phase analyses. Two randomly chosen crowns were cross-sectioned, polished with 3 µm diamond suspension and treated by one of two different etching techniques. One specimen was thermally etched at 500°C for 30 min. The other specimen was acid etched with 5% hydrofluoric acid gel for 60 s. Prior to SEM examination, specimens were carbon coated to prevent electrostatic charge accumulation.

2.3.2 Accelerated step-stress fatigue damage analysis—All crowns were examined for occlusal surface damage associated with the fatigue scar and its surrounding areas using polarized light stereomicroscopy (Leica MZ-APO, Wetzlar, Germany). Failures were designated as chip-off fracture or bulk-fracture through the crown. Crowns that did not present fracture were evaluated for subsurface damage using a sectioning technique [40] rather than a residual strength measurement using the monotonic load to failure test [41–43]. The authors believe that more information can be obtained by identifying surface and subsurface structural changes than from very high load fracture patterns or reduction in initial strength. Therefore the crowns were embedded in the clear epoxy resin, cross-sectioned buccolingually along the direction of the sliding contact and slightly away from the center of the fatigue scar, using a water cooled low speed saw with a diamond wafering blade (Isomet, Buehler, Lake Buff, IL). Cross-sectioned crowns were polished up to the center of the fatigue scar using a sequence of diamond grinding discs of 15, 9, 6, and 3 microns (Apex, Buehler), and analyzed using polarized light stereomicroscopy for the presence of cracks and damages.

2.3.3 Long-term fatigue/wear damage analysis—Long-term sliding-contact mouth-motion fatigued crowns were evaluated by a micro-computed tomography scanner (µCT Scanco 40 medical AG, Bassersdorf, Switzerland) operated at 70kVp energy, intensity of 114 µA (0.5 mm AL Filter), integration time 248 ms, and threshold 440. Each crown was scanned in high resolution for 18.2 min, resulting in 305 slices, with a voxel size of 8 µm. The standard error of measurement for the testing conditions was 4.95 µm. The top (occlusal) 171 slices were selected for evaluation and 3D reconstruction. Advanced software (Dataviewer version 1.5.0.2, CT An version 1.13, CT vox version 2.6, Adobe Photoshop CC) was used for image processing to compare the original topography of the occlusal surface with that after fatigue/wear testing, and determine the maximum wear depth and volume loss at the fatigue scar.

3 Results

3.1 Microstructural characterization

Secondary electron imaging (SE), back-scattered electron imaging (BSE), and a semi-quantitative EDS analysis were used to obtain the following results. To reveal the interconnected ceramic network structure, the ENAMIC specimen was polished and then thermally etched at 500°C for 30 min to selectively burnout the polymer phase. A representative SE image is shown Fig. 1a, where a ceramic network structure consisting of ~25% of porosity is clearly evident. Interestingly, a BSE image of the same area revealed some inclusions within the ceramic phase (see arrows in Fig. 1b). These inclusions appeared to vary in size and shape and have a random distribution across the specimen. The semi-quantitative elemental analysis (EDS) was performed on the polished and heat-treated specimen surface. An elemental mapping was conducted on selected areas (50 x 50 µm) but deliberately avoiding those large inclusions. The most abundant element was silicon, followed by aluminum, potassium, and sodium. These are common elements found in feldspathic ceramics. EDS was also used to analyze the elemental composition of the inclusions, revealing a major peak of yttrium. For better visualization of the polymeric phase, a second specimen was polished and then acid etched (5% HF acid for 60 s) to selectively remove the surface ceramic material. Both SE and BSE images confirmed the presence of an organic polymer phase (Figs. 1c & 1d) not dissolved by the hydrofluoric acid.

3.2 Single load-to-fracture testing

Under monotonic loading, the maximum load to fracture was recorded for 4 ENAMIC crowns. The highest fracture load value measured was 2536 N, the lowest 1775 N, and the mean fracture load $\pm$ standard deviation was 2329 ± 411 N. In all cases, fracture occurred by splitting the crowns mesio-distally (Fig. 2). Splitting is induced by axial loading with a hard indenter wedged between the molar cusps. It is a common mode of fracture in single cycle axial loading of anatomically-correct monolithic crowns [44].

3.3 Accelerated sliding-contact mouth-motion step-stress fatigue results

Out of the 24 crowns subjected to accelerated step-stress fatigue (Tables 1 & 2), 21 crowns survived, 2 chipped (one from the moderate profile and one from the mild profile), and 1 crown underwent bulk fracture in the mild profile. Through fractographic analysis, one could observe a series of fracture features that indicated fracture origin at the occlusal surface of the fatigue scar. One crown presented a bulk fracture and the fractographic features are presented in Fig. 3. The fracture surfaces showed fractographic features such as coarse hackles and arrest lines, which indicate that fracture propagated from the occlusal surface [45].

Two crowns presented chip-off fractures (Fig. 4). The fractures had a typical scallop-shaped morphology with a maximum length extending near or all the way to the cervical margin, and involved two cusps (distal and distobuccal cusps). Closer inspection from the buccal aspect of the crown revealed arrest lines, suggesting the fracture originated at the occlusal contact point [45]. The fractured surface did not expose the Z100 abutment. No radial fractures were observed.

Crowns that survived the accelerated step-stress fatigue—aggressive (1700 N/70k cycles) and mild (1200 N/120k cycles) profiles were inspected under the stereomicroscope for surface and subsurface (after cross-sectioning) damage. Representative images of an ENAMIC crown following fatigue loading (mild profile) are shown in Fig. 5. The occlusal aspect revealed two fatigue damage modes: sliding-contact induced occlusal surface herringbone cracks on the disto-buccal cusp [23,29]; and, impact induced concentric ring cracks on the disto-lingual cusp [25,46,47]. Under the cyclic loading in wet environments, both herringbone and ring cracks are capable of propagating deep into the material, forming partial [24,31] and inner [18] cone cracks. As demonstrated in the cross-sectional views in Fig. 5: disto-buccal cusp sliding-induced occlusal surface partial cone cracks (solid arrows) were observed in all sectioned crowns, whereas disto-lingual cusp impact-induced inner cone cracks (open arrows) were present in most cross-sectioned crowns. However, the number and length of these cracks varied greatly among the crowns inspected. Radial cracks originating from the cementation intaglio surface were not observed in any of the cross-sectioned crowns.

3.4 Long-term sliding-contact mouth-motion fatigue/wear results

Under a clinically relevant load of 200 N applied with a steatite antagonist ($r = 3$ mm) for 1.25 million cycles in water, the specimens showed only a small wear scar at the sliding surface and no catastrophic failure. The average of maximum wear depth $\pm$ SD was 66 ± 14 μm , and volume loss was 0.051 ± 0.006 mm^3 . Material loss in this *in vitro* test corresponds to clinical wear of about 5 years [36,37].

4 Discussion

The present study investigated the sliding contact fatigue and wear response of a recently developed PICN (Vita ENAMIC) for CAD/CAM applications. Following the most harsh step-stress profiles that the chewing simulator could deliver (a maximum fatigue load of 1700 N), only 3 out of the 24 tested crowns experienced catastrophic failure. These results suggest that a microstructure composed of dual interpenetrating networks with distinct elastic moduli [11,48] yields good resistance to mouth-motion fatigue by toughening effects [11,49]. Coldea and collaborators [10] reported that cracks in a PICN propagate predominantly through the ceramic network and along the polymer/ceramic interface with polymer deformation bridges across the crack, which enhances the resistance to crack propagation.

A consistent failure pattern was observed in single load-to-fracture testing. The failure was ascribed as splitting from the central fossa through the composite abutment to the crown base into two halves. Splitting was induced by axial loading with a hard indenter wedged between the buccal and lingual cusps. Interestingly, the splits often deviated slightly from the symmetry plane along the central fissure between the buccal and lingual cusps, suggesting that cracks can sometimes propagate as readily through part of a cusp as through the fissure. The crack trajectories depend on the distribution of tensile stresses in the cuspal region. Variations in the cuspal geometry of molar teeth and the radius of indenting spheres can alter the stress distributions. The average splitting load under the current test conditions

was over 2000 N, well above the nominal biting force [50]. However, under extreme situations with high extrinsic forces (trauma) or in bruxer patients, peak tooth loading could reach 1000 – 1400 N [51–53]. It is well established that the slitting load diminishes as the indenter radius decreases [44]. Thus, under extreme circumstances, splitting of ENAMIC crowns may occur if the indenter radius reduces to 1 – 2 mm—a value that lies at the lower border of the nominal molar cuspal radius range 2 – 4 mm [39].

The surviving crowns from the accelerated step-stress fatigue test showed consistent damage modes, which comprised a series of partial cone cracks at the sliding contact sites [23,24,29]. Outer and inner cone cracks have been observed as well [18,23]. Propagation of cone cracks subjected to cumulative sliding contact is associated with mechanical fatigue such as hydraulic pumping as well as moisture-assisted slow crack growth [18]. Extending this type of damage to crack propagation leading to catastrophic failure would require much higher indentation loads or more loading cycles [24,25]. Clinically, since molar crowns are subjected to high chewing stresses, they are vulnerable to fracture; particularly restored teeth [20]. The cumulative chewing cycles would be the cause of such fatigue mechanisms in molar crowns, providing a persistent driving force for crack propagation. It is important to note that the fatigue damages described above were observed in the ENAMIC crowns that survived cyclic loading up to 1700 N and 300k cycles. To the authors' knowledge, there are no other studies investigating fatigue damage in ENAMIC posterior crowns that can be directly compared to the results of the present work. There have been only two recent publications concerning fatigue behavior of ENAMIC [54,55]. One evaluated residual strength of anterior ENAMIC crowns following 50 – 200 N/3.7 million R-ratio fatigue testing [54]. The other determined fatigue limit of ENAMIC in 3-point bending mode [55]. Other materials to be directly compared to this PICN would be CAD/CAM resin composites and glass-ceramics. Molar crowns of Lava Ultimate (CAD/CAM resin composite) presented 100% survival to a similarly harsh step-stress protocol [21]. In contrast, the glass-ceramic IPS Empress CAD did not survive mouth-motion sliding fatigue above 650 N [21], while e.max CAD seems to have a threshold for damage/bulk fracture in the range of 1,100 to 1,200 N [31]. Reaching loads higher than those seen in routine chewing (100–150 N) [50] yields the exploration of thresholds for damage accumulation changes in failure modes that might occur at extremely high occlusal loading. Further, male bruxers are known to exhibit bite forces up to 1000 N during sleep with force durations many times longer than those observed in normal chewing [53,56]. The ENAMIC crowns showed good fatigue resistance, indicating that this material is well indicated for full contour restorations, and may even be indicated for patients with parafunctional activity. However, all ENAMIC full coverage crowns in this study were supported by dentin-like composite abutments, so caution is advised if ENAMIC is to be used to fabricate bridges.

The wear resistance of ENAMIC against steatite antagonist was investigated by using mouth-motion cyclic loading at a frequency of 2 Hz with a clinically relevant load of 200 N [57] for 1.25 million cycles, which approximately corresponding to about 5 years in the oral cavity [36,37]. None of the crowns tested in the present study showed fractures, and the ENAMIC wear depth recorded was $66 \pm 14 \mu\text{m}$. Other studies have tested the wear response of this PICN [17,58,59]. Swain et al. [17] performed a fatigue test simulating 5 years of mouth-motion sliding-contact in similar conditions to those presented herein and in

association with thermal cycling. Similar to the results presented here, none of the 14 ENAMIC crowns fractured or presented significant fatigue damage other than minor wear. In that study, 12 out of 14 Vita Mark II (feldspar ceramic) crowns revealed significant fractures, while 6 out of 14 e.max CAD (lithium disilicate ceramic) crowns present minor cracking. The authors did not quantify wear in their study.

In two studies with ENAMIC [58,59] wear depth was quantified but the test conditions were quite different from those of the present study, hindering direct comparisons. Stawarczyk et al. [58] investigated wear simulating 5 years of mouth-motion sliding-contact of human enamel against various dental CAD/CAM materials: resin composites, PICN, and glass-ceramics. The PICN showed less wear than the resin composites and similar to glass-ceramics, while enamel antagonists wore more against the latter. Enamel-enamel *in vivo* wear results vary significantly. Esquivel-Upshaw et al. [57] reported a 40 – 80 µm enamel wear after 1 year, while other studies reported yearly enamel wear around 10 µm [60], and 29 – 38 µm for molar crowns [61]. Esquivel-Upshaw et al. [57] also reported a 40 – 100 µm antagonist enamel wear depth against three distinct veneer ceramic materials after 1 year in occlusal function. Taking all into account, ENAMIC seems to have similar wear resistance to glass-ceramics [58], which are reported to yield acceptable antagonist enamel wear *in vivo*. Therefore one could expect that this PICN exhibits relatively good wear performance clinically. The use of the dual microstructure with interpenetrating networks also fully capitalizes the advantage of the CAD/CAM technology (i.e. easy to machine and polish with no post-machining firing required), thus offering a viable one-visit treatment option, even for patients with a high bite force.

5 Conclusions

The polymer infiltrated ceramic network monolithic crowns showed excellent resistance to fatigue contact and flexural damage. Failure could only be observed at very high loads (> 1,000 N) by using step-stress accelerated life testing. This suggests that early clinical abrupt fractures of ENAMIC posterior crowns are unlikely and may only occur under extremely high traumatic loads, especially when the radius of the antagonist cusp is relatively small. In addition, the PICN material exhibited good wear resistance under the clinically relevant loading conditions. However, all ENAMIC full coverage crowns in this study were supported by dentin-like composite abutments, so caution is advised if ENAMIC is used to fabricate bridges.

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Highlights

- The ability of polymer infiltrated ceramic network (PICN) monolithic crowns to sustain high bite force and wear challenge has been evaluated.
- PICN crowns can withstand mouth-motion cyclic loading up to 1700 N and 300K cycles.
- PICN crowns have only suffered minor wear following prolonged sliding-contact cyclic loading under clinically relevant conditions.
- Under extreme loads (> 2000 N), fracture may occur by splitting the crowns mesio-distally.

Figure 1.

Microstructure of ENAMIC observed by SEM. (a) SE image of a polished and then thermally etched ENAMIC specimen, revealing a ceramic network structure consisting of ~25% of porosity following selective removal of the polymer phase. (b) BSE image of a polished and thermally etched specimen, arrows point to inclusions within the ceramic phase. (c) SE image of a polished and then acid etched ENAMIC specimen, showing the polymer network after selective removal of the surface ceramic material. (d) BSE image of a polished and acid etched specimen, confirming the presence of an organic polymer phase.

Figure 2.
The dominant failure mode in ENAMIC crowns subject to the single-cycle load-to-fracture test. Fracture occurred by splitting the crowns mesio-distally under very high loads (above 2000 N).

Figure 3.

One ENAMIC crown presented bulk fracture during the accelerated step-stress test. (a) & (b) Occlusal & buccal views of the fractured crown. (c) The image shows twist hackles (open arrows), marking the crack propagation direction from the occlusal surface downward. (d) The broken part of the specimen. The image shows hackle lines (coarse hackles and twist hackles, solid arrows) and arrest lines (open arrows), indicating that the fracture origin is on the disto-buccal cusp ridge at the occlusal surface (white arrows).

Figure 4.

Two chipped ENAMIC crowns. (a & b) An ENAMIC crown chipped at 1150 N after 158,728 cycles (mild profile). The buccal view shows twist hackles (solid arrows) and arrest lines (open arrows), indicating that the direction of crack propagation is from the occlusal surface downward. Fracture origin is marked by a white arrow. The chip extended to the cervical third of the buccal surface. (c & d) An ENAMIC crown chipped at 1300 N after 115k cycles (moderate profile). The chip extended to the cervical margin of the crown. The occlusal views show the damage at the disto-buccal cusp due to cyclic contact loading. Note: white arrows in (c & d) indicate the fracture origin.

Figure 5.

Damage sustained in crowns following accelerated step-stress fatigue loading. (a) An overview of the cross-sectional surface of a survival crown from mild profile. (b) Higher magnification view of partial cone cracks emanating from the sliding contact area on the disto-buccal cusp (solid arrows). (c) Inner cone cracks emanating from the impact site on the disto-lingual cusp (open arrows).

Table 1

Distribution of crowns per loading profile, number of survivors and failures, and survival rate (%) for the accelerated step-stress fatigue.

Profiles (Load/No. of cycles)	No. of crowns tested	No. of survivors	No. of failures	Survival rate %
Extended Aggressive (1700N/300k)	3	3	None	100
Aggressive (1700 N/70k)	3	3	None	100
Moderate (1400 N/120k)	6	5	1	83
Mild (1200 N/170k)	12	10	2	83

Table 2

Description of failed crowns during accelerated step-stress fatigue test.

Specimen ID	Profile	Failure load (N)	No. of cycles	Failure mode
sp 9	Moderate	1300	115,000	Chipping
sp 14	Mild	1150	158,728	Chipping
sp 15	Mild	1050	132,494	Bulk fracture