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Objectives: Compressive stress has been intentionally introduced into the overlay porcelain of zirconia-ceramic prostheses to prevent veneer fracture. However, recent theoretical analysis has predicted that the residual stresses in the porcelain may be also tensile in nature. This study aims to determine the type and magnitude of the residual stresses in the porcelain veneers of full-contour fixed-dental prostheses (FDPs) with an anatomic zirconia coping design and in control porcelain with the zirconia removed using a well-established Vickers indentation method.

Methods: Six 3-unit zirconia FDPs were manufactured (NobelBiocare, Gothenburg, Sweden). Porcelain was hand-veneered using a slow cooling rate. Each FDP was sectioned parallel to the occlusal plane for Vickers indentations (n = 143; load = 9.8 N; dwell time = 5s). Tests were performed in the veneer of porcelain-zirconia specimens (bilayers, n=4) and porcelain specimens without zirconia cores (monolayers, n = 2).

Results: The average crack lengths and standard deviation, in the transverse and radial directions (i.e. parallel and perpendicular to the veneer/core interface, respectively), were 67 ± 12 µm and 52 ± 8 µm for the bilayers and 64 ± 8 µm and 64 ± 7 µm for the monolayers. These results indicated a major hoop compressive stress (~40-50 MPa) and a moderate radial tensile stress (~10 MPa) in the bulk of the porcelain veneer.

Significance: Vickers indentation is a powerful method to determine the residual stresses in veneered zirconia systems. Our findings revealed the presence of a radial tensile stress in the overlay porcelain, which may contribute to the large clinical chip fractures observed in these prostheses.

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Residual Stresses in Porcelain-veneered Zirconia Prostheses

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Significance—Vickers indentation is a powerful method to determine the residual stresses in veneered zirconia systems. Our findings revealed the presence of a radial tensile stress in the overlay porcelain, which may contributed to the large clinical chip fractures observed in these prostheses.

Keywords

residual stress; porcelain-veneered zirconia; veneer chip fracture; Vickers indentation; fracture mechanics

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INTRODUCTION

Metal-free all-ceramic restorations offer better aesthetics and biocompatibility than porcelain fused to metal (PFM) prostheses [1–3]. Zirconia ceramic is nowadays widely used as a framework material in full-coverage crowns and fixed partial dentures (FPDs) due to its high flexural strength (900–1200 MPa) and fracture toughness (5–7 MPa·$m^{1/2}$) [4]. However, clinical research and practice have reported high incidence of veneer chipping and fracture in all major brands of porcelain-fused-to-zirconia (PFZ) systems, particularly in posterior restorations [5–14]. Examples of veneer chipping and fracture in porcelain fused to zirconia (PFZ) prostheses, after 6 months intra-oral service and mouth-motion fatigue loading *in vitro*, are shown in Fig. 1a and b, respectively. In both cases, cracks developed in the occlusal contact area, propagated downward along the axial direction and eventually intersected with the axial wall, resulting in significant veneer chipping.

It is generally believed that veneer chipping and fracture may be a result of residual tensile stress developed in the porcelain layer during the cooling process of firing cycles involved in sintering of ceramic veneers. Such residual stresses may arise from the thermal expansion mismatch between the porcelain veneer and the zirconia framework as well as from the rapid cooling after sintering, owing to the low thermal diffusivity of zirconia. In an attempt to estimate the magnitude of residual stresses, theoretical [15] and experimental [16–19] work have been conducted on flat models of PFZ bilayer systems. However, dental crowns and bridges have complex geometries with varying thickness of veneer and core. Therefore, stress analysis of model flat PFZ bilayers can only provide a qualitative illustration of the stress states in anatomically-correct restorations.

Over the past several decades, the materials engineering community has developed a number of techniques to evaluate the residual stresses in various materials systems. For example, the birefringence technique is able to measure the residual stresses by analyzing changes in the optical properties of a material that occur when stresses are present [20, 21]. Birefringence has obvious limitations for non-transparent materials and analysis of residual stress can be complicated by optical inhomogeneities in crystallite-containing glasses such as porcelain. X-ray or neutron diffraction techniques can effectively determine the residual stresses only in crystalline materials. The layer removal technique measures the uniaxial residual stress distributions in rectangular bar specimens by removing layers of known thicknesses and measuring the ensuing deflection of the specimens [22]. This approach places restrictions on the size and shape of the specimens, making it impractical to quantify residual stresses in dental crowns and bridges. Direct strain gauge applications and hole-drilling technique have also been used [19, 23]. However, these methods require a critical degree of expertise. In addition, the feasibility of positioning strain gauges and drilling holes in dental restorations is limited by their shapes. The Vickers indentation method (VIM) was first used to determine surface residual stresses in brittle materials by Marshall and Lawn 35 years ago [20]. In this method, surface residual stresses can be estimated by comparing the indentation crack length in stressed samples to that in unstressed samples [17, 18, 24–27].

The VIM has the potential for rapid evaluation of material properties in small samples with irregular shapes (such as dental restorations), which is a clear advantage over the various techniques described above. The disadvantage of the VIM is, as with many other mechanical testing methods such as hole-drilling, the requirement of a relatively smooth surface to assure an accurate measurement of indentation crack length. The VIM has been used to estimate the residual stresses in bilayer dental ceramics [17, 18]. However, all previous studies have used flat models of bilayer systems. This study aims to determine the type and magnitude of the residual stresses in the porcelain veneers of full-contour FDPs with an anatomic zirconia coping design using a well-established VIM. Such an exercise can take us one step closer to understand the nature of the residual stress, and thus the chipping and fracture problems of zirconia-based restorations.

MATERIALS & METHODS

Samples preparation

Six zirconia mandibular three-unit FDPs were obtained from NobelBiocare (Gothenburg, Sweden). All frameworks were CAD/CAM fabricated and consisted of the second premolar and second molar abutments and first molar pontic. The external surface of each framework was abraded with airborne alumina particles (d_{50} ~ 100 μm) at 1.0 bar pressure at a standoff distance 10 mm. A thin wash bake at 940°C was performed with Transpa Clear (NobelRondo, Nobel Biocare, Gothenburg, Sweden) for coloring purposes. The frameworks were hand-veneered by an experienced technician, where porcelain slurries were applied to the zirconia frameworks with a brush, condensed, and sintered. This procedure consisted of two firings at 930 and 910°C, followed by two glaze cycles at 890 and 850°C, respectively, according to the manufacturer's specifications. A slow cooling rate (30 °C/min) was utilized for each firing cycles and was controlled by keeping the furnace door closed until reaching 520ºC, which is around 50 ºC below the porcelain T_g temperature. Slow cooling is recommended by the manufacturer to reduce the amount of residual stresses that generate in the veneer due to temperature gradients during the cooling period of the firing cycle. The coefficients of thermal expansion (CTEs) of the NobelRondo porcelain and zirconia, measured at Wieland Dental Ceramics (Germany) using a well-known industrial dilatometer, were ~9.3×10^{-6} K^{-1} and ~10.4×10^{-6} K^{-1}, respectively. Twelve CAD/CAM-made zirconium-oxide abutments (Procera, Nobel Biocare, Gothenburg, Sweden) were screw-retained to Replace-straight-Groovy implants (Nobel Biocare, Gothenburg, Sweden). Abutments were then cemented on to the copings with glass-ionomer cement (Ketac Cem, 3M-ESPE), following manufacturer's instructions. All FDPs were then embedded in epoxy-resin (Epofix, Struers, Copenhagen, Denmark). For each sample, two cuts, approximately 3 mm apart, were made parallel to the occlusal plane, producing flat sections for indentation (Fig. 2a). A precision diamond saw (Isomet 2000, Buelher, Lake Bluff) was used. The sectioning directions were carefully chosen to preserve any hoop and radial stresses in the porcelain veneers. Four specimens included both the zirconia core and porcelain veneer (bilayer) (Fig. 2b). For the remaining two, the zirconia core was carefully removed, leaving only a monolithic porcelain layer (monolith). For all six specimens, a surface (Fig. 2a, blue arrows) was prepared for indentation testing by grinding with 600 grit SiC abrasive paper followed by polishing with diamond suspensions of 9, 3 and 1 μm particle size (Buehler, Lake Bluff, IL, USA).

Indentation testing

Vickers indentations were performed on the polished surface of the porcelain layer with a peak load of 9.8 N and a dwell time of 5 s using a microhardness machine (Leco, St. Joseph, MI, USA). To avoid interactions, indentations were performed at a distance at least twice the crack lengths from each other, defects, and porcelain edges [28]. Two rows of indents, approximately 1/3 and 2/3 thickness of the veneer layer away from the porcelain/zirconia interface, respectively, were placed in the veneer of the pontic, premolar and second molar with sharp corners oriented perpendicular (radial) and parallel (transverse) to the veneer/core interface (Fig. 2b). Indentations were performed in the bilayer specimens (n = 128) and in the monolithic porcelain (n = 28). Indentation crack patterns were captured immediately after testing using a calibrated imaging system incorporated in the microindentation tester (Buehler, Lake Bluff, IL, USA), so that moisture-induced slow crack growth had no significant influence on the crack length. Measurements were taken from the center of the indentation impression to the crack tip (Fig. 3) [29]. Indents showing significant lateral cracking or material spalling were not included in the analysis [30]. Scanning electron microscopy (SEM, S-3500 N, Hitachi Instruments, San Jose, CA, USA) was also used for better quality images and to confirm the measured crack length. Prior to SEM examination, specimens were gold coated typically within 15 min after the indentations. To assure accuracy of the measurements, for each crack length, the two values obtained from images captured using the calibrated optical system and SEM were averaged.

By measuring the crack length in stressed (i.e. porcelain fused to zirconia bilayers) and unstressed (i.e. standalone porcelain monoliths) veneering materials (Fig. 3), the magnitude of residual stresses, σ_R, in the porcelain veneer of a PFZ restoration can be estimated using the following equation [24]:

$$\sigma_{\mathrm{R}}=K_{1c}\left[\frac{1-(c_0/c_1)^{3/2}}{\psi c_1{}^{1/2}}\right] \quad (1)$$

where ψ is a crack geometry factor. For half-penny cracks, $\psi = 1.24$ [24]. c_0 and c_1 are the indentation crack lengths in unstressed and stressed materials, respectively (Fig. 3). K_{1c} is the fracture toughness of the porcelain veneer. Unfortunately, there is no reliable toughness value of this material available in the open literature using ASTM recommended methods, such as the single edge V-notch beam method (SEVNB). Quinn and co-workers [31] measured fracture toughness for various dental porcelain materials and brands using both SEVNB and single-edge pre-cracked beam (SEPB) methods. It was found that the toughness values of different types of commercial dental porcelains varied from 1.0 to 1.3 MPa·m$^{1/2}$. Therefore, residual stresses were computed across a range of reported fracture toughness values (between 1.0 and 1.3 MPa·m$^{1/2}$).

Statistical analyses

An ANOVA ($\alpha = 0.05$; SigmaPlot 11.0, Ashburn, VA, US) was performed to compare crack length in both transverse and radial directions among different locations of each specimen (pontic vs. premolar vs. molar), and among specimens of the same group (bilayer or monolith). A t-test ($\alpha = 0.05$; SigmaPlot 11.0, Ashburn, VA, US) was performed to compare crack length in both transverse and radial directions between row 1 and row 2 for each abutment (premolar or molar or pontic). The same test was used to compare crack length between radial and transverse directions for bilayers and monoliths.

RESULTS

A representative pattern of the Vickers indentation in monolithic porcelain is shown in (Fig. 4a). As can be seen, the location of the indentation did not affect the crack length. For each abutment and pontic, both radial and transverse cracks had no significant difference amongst rows ($p > 0.05$ for both radial and transverse directions). When both radial and transverse cracks in rows 1 and 2 were pooled together, no significant difference was observed between abutments ($p > 0.05$ for both radial and transverse). Crack lengths were 64 ± 8 μm and 64 ± 7 μm in transverse and radial directions, respectively. No difference was found between radial and transverse cracks ($p = 0.25$). This observation lends confidence to the accuracy of residual stress measurements using the VIM.

A typical indentation pattern in the porcelain/zirconia bilayer specimens is shown in (Fig. 4b). The location of the indentation did not affect the residual stresses values. In details, for each bridge section (i.e. pontic, molar abutment or premolar abutment), both radial and transverse residual stresses had no significant difference between the two rows ($p > 0.05$ for both radial and transverse). Therefore, radial and transverse values obtained in rows 1 and 2 were pooled together and when compared among different bridge sections (premolar, molar and pontic), no significant difference was found ($p > 0.05$ for both radial and transverse). In addition, no difference in residual stresses was found among different bilayer specimens ($p > 0.05$ within radial or transverse groups).

The average crack length and standard deviation for the bilayers were 67 ± 12 μm and 52 ± 8 μm in the transverse and radial directions, respectively. As can be seen, transverse cracks were significantly longer than radial cracks ($p < 0.001$) (Fig. 4b). In addition, the transverse cracks were significantly longer in the bilayer specimens relative to the monoliths ($p = 0.006$). In contrast, the radial cracks were significantly shorter in bilayers relative to monoliths ($p = 0.001$).

Using Eq. 1 and the literature fracture toughness range for dental porcelain (i.e. 1 to 1.3 $MPa \cdot m^{1/2}$) [31], the estimated residual stresses in the porcelain veneer of our bilayers were -40 MPa to -53 MPa for hoop compressive stress (responsible for suppressing cracks in the radial direction), and 9 MPa to 11 MPa for radial tensile stress (leading to opening of cracks in the transverse direction) ($p < 0.001$).

DISCUSSION

In this work, the VIM was used to estimate the residual stresses in the porcelain veneer of hand build-up FDPs. The advantage of this testing technique was that residual stresses in both transverse and radial directions could be estimated by orienting the two orthogonal axes of a pyramidal indenter parallel and perpendicular to the porcelain/zirconia interface, respectively. Our findings revealed a large component of hoop compressive stress (−40 to −50 MPa) and a relatively small radial tensile residual stress (~10 MPa) in the bulk of the porcelain veneer. Therefore, any cracks in the porcelain veneer of these FDPs would have a tendency to propagate in the direction parallel rather than perpendicular to the porcelain/zirconia interface. Indeed, clinical research and laboratory testing have shown that veneer chip fractures propagated predominantly parallel to the interface, remaining near-orthogonal to the radial tensile component, and rarely reached the porcelain/zirconia interface (Fig. 1) [12, 32].

It is important to note that the residual stresses reported here are different from the actual stresses in whole FDPs, since they were measured on the planar section of truncated restorations. However, a previous work on veneered prostheses showed that, for a CTE mismatch of $2 \times 10^{-6}\ K^{-1}$ (veneer: $12 \times 10^{-6}\ K^{-1}$; core: $14 \times 10^{-6}\ K^{-1}$), the stress measured in the porcelain along the tooth axial direction was tensile and only 2 MPa, which was an order of magnitude smaller than that in the plane parallel to the interface [23]. Therefore, we sectioned our specimens parallel to the occlusal plane to preserve the most significant stresses.

The utilization of the VIM to determine residual stresses deserves further discussion. It has been recently suggested that the VIM is not a suitable technique for the measurement of the fracture toughness of brittle materials [30]. This is because the VIM produces a complex elastic-plastic zone beneath the indenter, which consists of an outwardly expanding hydrostatic core and its associated plastically deformed zone, surrounded by an elastic matrix [29]. In an isotropic brittle material like glass or porcelain, the VIM produces two orthogonal median-radial (halfpenny) cracks associated with the sharp corners of the indentation pyramid. Due to the 3-D nature of these cracks, there is no stress intensity solution available. Therefore, fracture toughness measurements using the VIM are based on empirical data fitting. However, in the present study, we use the VIM to determine the residual stresses of brittle materials. Despite the complex stress field and crack geometry produced by the VIM, in an isotropic material, cracks emanating from the four corners of the indentation impression should be symmetrical. This is supported by our experimental data where indentation crack lengths in monolithic porcelain layer were identical in all four corners. However, in porcelain/zirconia bilayer specimens the residual stresses contributed to indentation crack propagation, leading to different crack lengths in the transverse and radial directions.

It is true that the indentation fracture pattern changes with the applied load. At low loads, it appears as a pyramidal indentation impression free of cracks. At higher loads, cracks begin to emanate from the four corners of the impression. Finally, extensive lateral cracking often associated with considerable spalling around the impression occurs. This crack pattern–indentation transition occurs at different loads for different brittle materials. Therefore, care was taken to ensure that an appropriate indentation load was chosen to produce the orthogonal halfpenny cracks without any significant lateral crack and/or material spalling [30].

We acknowledge that our current Vickers indentation impressions covered an area of approximately 150 × 150 μm and were intended to measure the residual stresses at 1/3 and 2/3 distances from the veneer/core interface. The expected variability in stresses along the direction from veneer/core interface to veneer outer surface did not result in significantly different values between the two areas. A technique allowing for more indents from the veneer/core interface to the porcelain outer surface, such as nanoindentation, might be needed in addition to the VIM for further investigation. In this way, more insights might be provided on the variability of residual stresses at different distances from the veneer/core interface.

CONCLUSION

Our findings revealed the presence of a radial tensile stress in the overlay porcelain of zirconia-ceramic prostheses, which may lead to the large clinical chips and fractures of these prostheses. In addition, we have demonstrated that Vickers indentation is a powerful method to determine the residual stresses in veneered dental prostheses.

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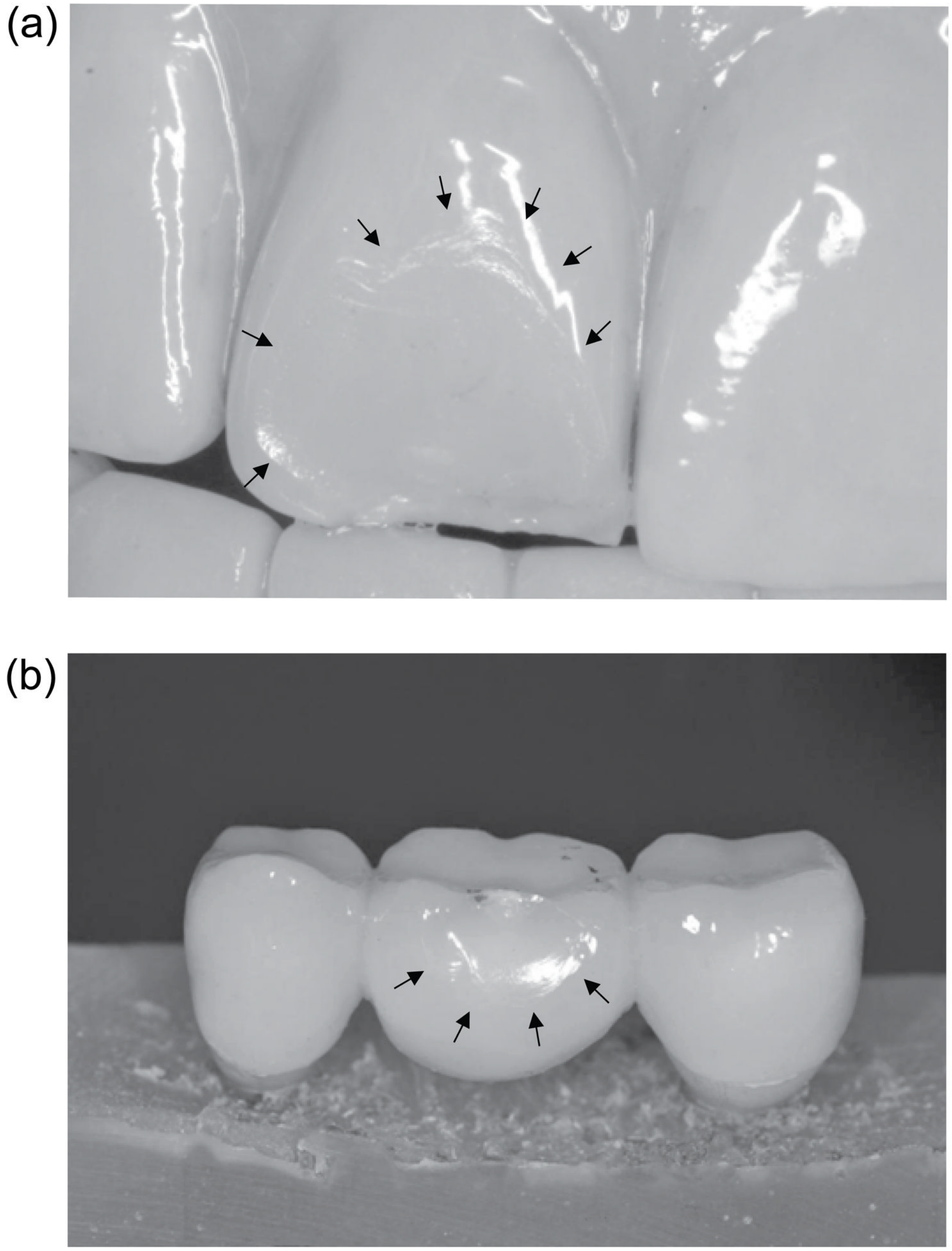

Figure 1.

Clinical and laboratory fractures in porcelain-veneered zirconia prostheses (Procera, Nobel Biocare, Gothenburg, Sweden). a) Clinically fractured zirconia FDP: a hand-veneered zirconia upper right central incisor crown of a Procera Implant Bridge fractured by porcelain chipping six months after restoration. Protrusive contact was documented on the incisal edge of the crown. b) Laboratory fractured zirconia FDP: an over-pressed zirconia Procera Implant Bridge loaded under mouth-motion step-stress accelerated life testing fractured by buccal chipping of the porcelain.

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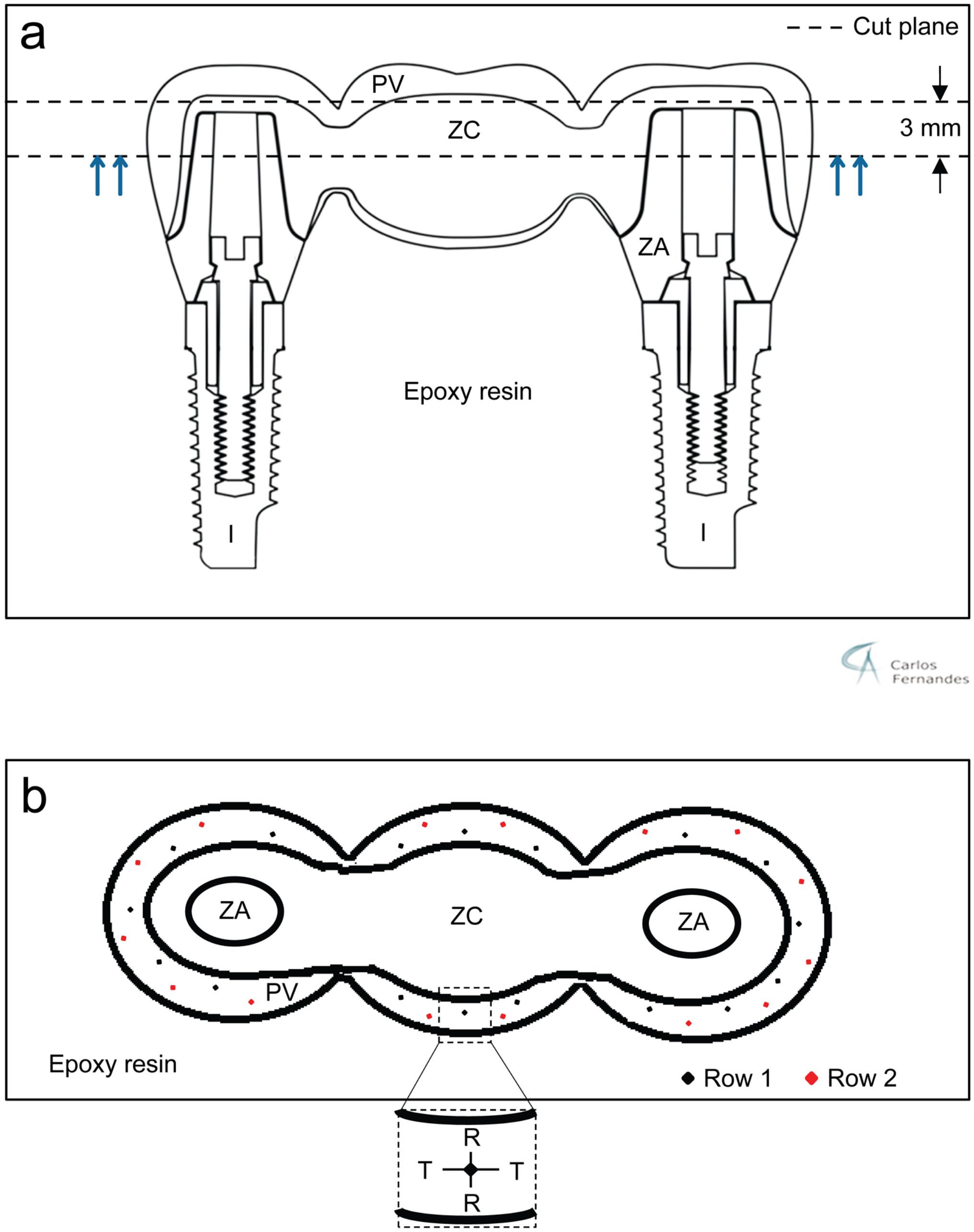

Figure 2.
Schematic plan view showing specimen sectioning direction and the incipient surface for Vickers indentation. a) Two cuts (dashed lines) were made parallel to the occlusal plane; one surface (blue arrows) was indented. ZA: zirconia abutment; ZC: zirconia core; PV: porcelain veneer; and I: implant. b) The incipient surface of bilayer specimens, including two abutments, a zirconia framework, and a porcelain veneer. Indentations were performed with the two orthogonal axes parallel and perpendicular to the zirconia/porcelain interface so that crack length in both transverse (T) and radial (R) directions could be measured.

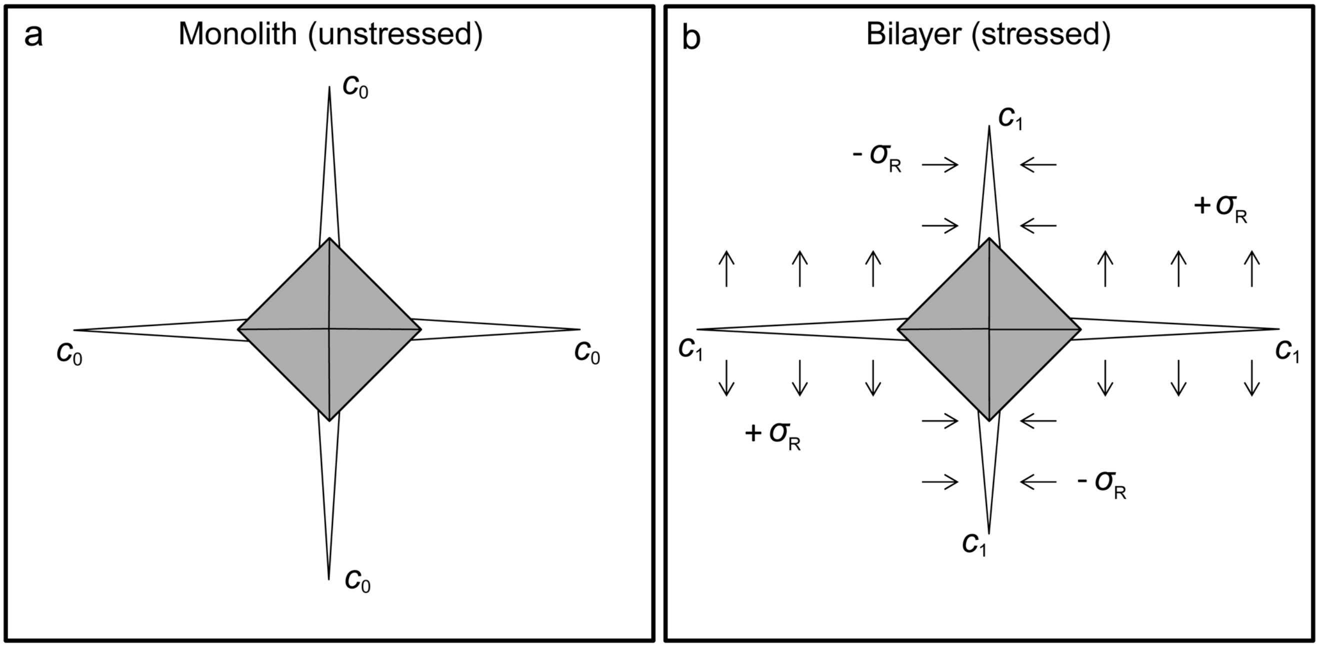

Figure 3.
Schematic plan view of fracture patterns produced by Vickers diamond pyramid indentation on porcelain. Crack lengths were measured from the center of the indentation impression to the crack tips for all directions. a) In unstressed material, cracks emanating from the four corners of the impression have identical length (c_0). b) In stressed material, the crack lengths (c_1) can vary depending on the nature and direction of the residual stresses. For cracks propagating near-perpendicular to a major component of tension, $c_1 > c_0$. For cracks extending near-perpendicular to the major component of compression, $c_1 < c_0$.

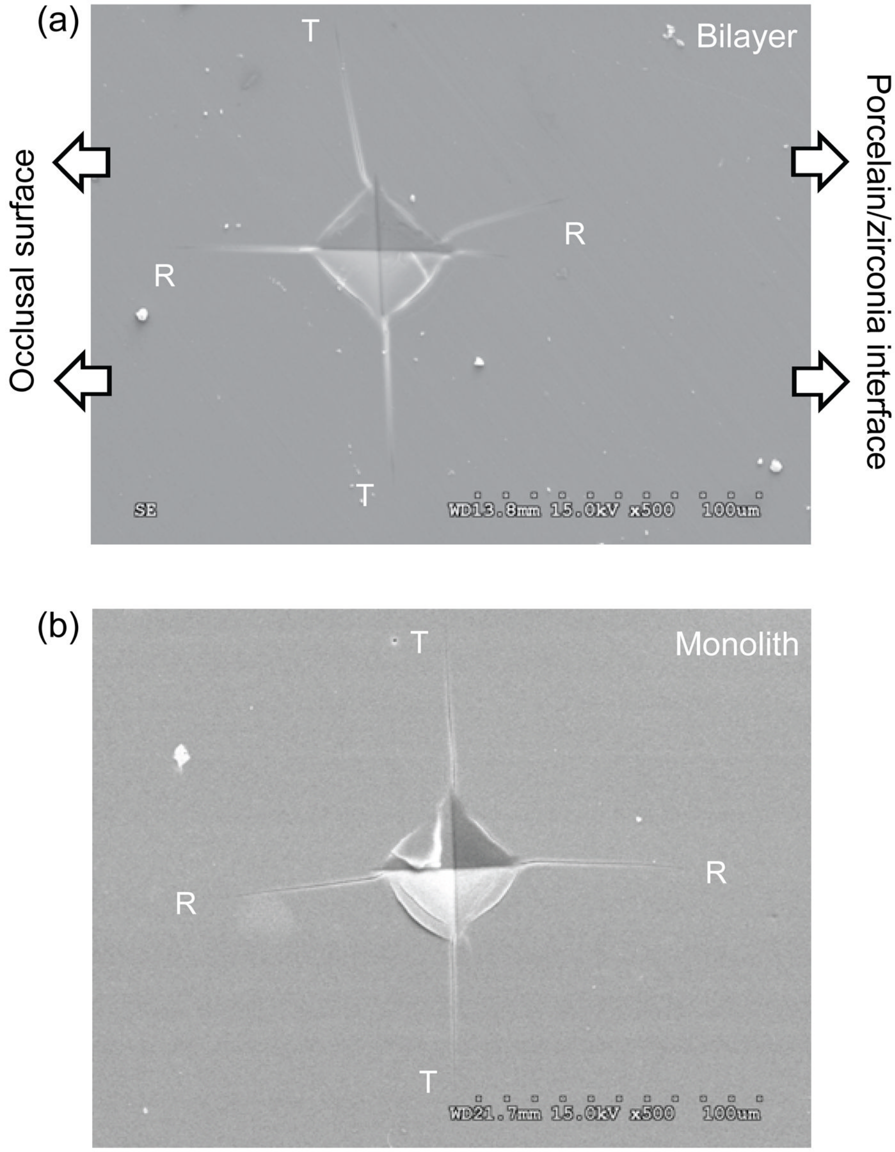

Figure 4.
Representative crack patterns of Vickers indentation on porcelain with and without a zirconia framework. a) For porcelain/zirconia bilayered specimens, crack lengths in the transverse direction (T) parallel to the zirconia/porcelain interface are significantly longer than those in the radial direction (R). b) For monolithic porcelain specimens, crack lengths in all directions are identical.