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ABSTRACT

Residual stresses within the veneer are linked to the high prevalence of veneer chipping observed in clinical trials of zirconia prostheses. We hypothesized that the thermal mismatch between the zirconia infrastructure and the veneer porcelain, as well as the rate used for cooling zirconia-veneer crowns, would be directly proportional to the magnitude of residual stresses built within the veneer layer. Two porcelains with different coefficients of thermal expansion were used to veneer zirconia copings, to create high or low thermal mismatches. The crowns were cooled according to a fast- or a slow-cooling protocol. The retardation of polarized light waves was used to calculate the residual stress magnitude and distribution across the veneer, according to the photoelasticity principle, in 1.0-mm-thick crown sections. While thermal mismatch was an important factor influencing the maximum stress development in the veneer, cooling rate had a minor role. Curved surfaces were preferential sites for stress concentration regardless of thermal mismatch or cooling rate.

KEY WORDS: veneered zirconia restorations, internal stress, thermal mismatch, cooling rate, birefringence, photoelastic effect.

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INTRODUCTION

Tensile and compressive residual stresses created during manufacturing may affect the lifetime of ceramic prostheses as a direct influence on contact-induced crack propagation resistance. Overall, the resultant residual stresses in bilayer prostheses are a superimposition of stresses generated during cooling and stresses due to the mismatch ($\Delta\alpha$) in coefficients of thermal expansion (CTE) between the core and the veneer.

The low thermal diffusivity of the framework in zirconia-veneer bilayer systems results in higher temperatures at the interface during cooling in comparison with other infrastructure materials (Swain, 2009). Thermal gradients within the veneer are aggravated for faster cooling rates due to the less relaxed state of the veneer as the temperature falls through the glass transition temperature (T_g) range (Zhang *et al.*, 2010). These conditions place zirconia-based prostheses under high risk of veneer fracture, and the high incidence of chipping reported in clinical trials seems to support the mathematical predictions (Sailer *et al.*, 2007a; Al-Amleh *et al.*, 2010). However, available data on the magnitude and spatial distributions of residual stresses in zirconia-veneer bilayers are usually analytical or experimentally measured using simple geometries (Taskonak *et al.*, 2008; Dittmer *et al.*, 2009; Swain, 2009; Zhang *et al.*, 2009, 2010; Mainjot *et al.*, 2011a).

Birefringence measurements are largely used in optical industry to detect and quantify local residual stresses in glass components (*e.g.*, lenses, prisms) that could affect their functionality. Modern image measuring systems allow for the accurate and reproducible automatic measurements of the spatial distribution, orientation, and magnitude of stresses in small samples with relatively high resolution. *Birefringence*, or *double refraction*, is measured based on the photoelasticity principle and results from the retardation of polarized light through a translucent material with heterogeneous density. Stress-free glasses and ceramics are optically isotropic, but mechanical or residual stresses cause the 2 decomposed waves from the original polarized light to follow an orientation parallel to the principal stress vectors (σ_1 , σ_2). This is accompanied by a phase delay between these waves, which would otherwise travel in the same velocity. Different wave velocities in birefringent materials result in 2 indices of refraction (n_1 , n_2). The retardation (δ) between waves can be expressed by $\delta = \Delta n \cdot t$, where t is the thickness of the material. According to the Stress-Optic Law (Durelli and Riley, 1965), the difference between refraction indices is proportional to the difference between principal stresses, given by:

$$\Delta n = C (\sigma_1 - \sigma_2) \quad (1)$$

where C is the coefficient of photoelasticity (CPE) of the material. The stress can then be calculated according to the following equation:

$$\sigma = \delta/tC. \quad (2)$$

This study aimed to determine the CPE of 2 veneers used for veneering zirconia infrastructures and to measure the spatial distribution and magnitude of residual stresses built up across the veneer in zirconia crowns cooled following 2 different cooling protocols. Veneers with different CTEs were used to generate a high or low thermal mismatch to the zirconia core.

MATERIALS & METHODS

Specimen Preparation

An intact human second premolar was reduced at the occlusal surfaces, free surfaces, and proximal surfaces in 2.1, 1.7, and 1.2 mm, respectively. The margins were chamfered with a circumferential depth of 1 mm. After the preparation was scanned, 12 yttrium-stabilized tetragonal zirconia blanks (YZ Cubes, Vita Zahnfabrik, Bad Säckingen, Germany; Lot 30820; CTE: $\alpha_{Y-TZP} = 10.5$ ppm/°C) were milled with a Cerec3 Inlab (Sirona, Bensheim, Germany) machine and sintered at a final temperature of 1530°C for 2 hrs to produce copings of 0.7-mm thickness.

The copings were veneered with 2 different porcelains (VM9, Vita Zahnfabrik, CTE, $\alpha_{VM9} = 9.1$ ppm/°C; LAVA Ceram, 3M ESPE, Seefeld, Germany, CTE, $\alpha_{LAVA} = 10.2$ ppm/°C), to create a high or low thermal mismatch with the zirconia core ($\Delta\alpha = +1.4$ ppm/°C with VM9 and $\Delta\alpha = +0.3$ ppm/°C with Lava Ceram). The porcelains were applied by a single operator using only the enamel shade (EL1 for VM9, lot 19469; EA1 for Lava Ceram, lot 8790D) to have a homogeneous translucency across the thickness of the veneer. After the enamel firing, the thicknesses of the crowns were checked with a specimeter and ground with a low-speed superfine diamond bur under water lubrication down to 2.1 mm (cusp) and 1.7 mm (buccal surface) if necessary. At the buccal cusp, the core/veneer ratio was 0.5. At the end of the glaze firing, the crowns were cooled from final sintering temperature following a fast- or a slow-cooling program. Fast cooling consisted of the immediate opening of the oven's door (Vacumat 4000, Vita Zahnfabrik) after the end of the firing schedule, exposing the crown to contact with convective ambient air (~23°C). After the end of the firing schedule in the slow-cooling program, the door of the oven opened only 10%, opening completely as soon as the temperature inside the oven reached 200°C. Crowns from each group were fired together during the last firing ($n = 3$). A K-type thermocouple was used to determine the cooling rates

Figure 1. Determination of the CPE through the relationship between stress and polarised light retardation. (A) For the CPE calculation, a polarimeter was used in assembly with a universal testing machine to measure the polarized light retardation in the center of the porcelain discs during the diametral tensile test. (B) A close-up image of the porcelain disc during the diametral tensile test while transilluminated with polarized light.

Figure 2. Specimen fabrication and Stress Birefringence measurement. (A) Two parallel cuts were made across the occlusal plane of the crowns to obtain ~1.0-mm-thick slices. (B) A specimen is transilluminated with polarized light for the stress birefringence measurement.

according to the fast- or slow-cooling programs at the same position at which the crowns were sintered inside the oven. Between the maximum sintering temperature and 500°C, cooling rates of 600°C/min and 30°C/min were recorded for fast- and slow-cooling protocols, respectively.

The crowns were mounted on a cutting machine (Isomet, Buehler, Lake Bluff, IL, USA), and 1.05-mm-thick bucco-lingual slices from the center of the crown were cut by means of a diamond-impregnated copper disk (Buehler) under water lubrication. After that, the slices were polished by hand with a decreasing sequence of SiC papers (Buehler), from #600 to #4000 grit, under water lubrication, to a final thickness of 1.0 ± 0.02 mm.

CPE Calculation

Three discs of each of the veneer ceramics were fabricated. The ceramic powders were mixed with Vita VM Modelling Liquid (Vita Zahnfabrik), poured into sectioned metallic molds of 16 mm in diameter, and condensed by manual vibration. The excess of liquid was blotted out with absorbent paper. The veneer discs were fired on a honey-comb tray with the same firing schedules used for the crowns and cooled slowly according to the slow-cooling protocol described in the previous section, to result in stress-free ceramic discs. The cooled discs were polished by

Figure 3. Residual stress map of Lava Ceram-veneered crowns. The dark gray represents the zirconia core. Magnitude and spatial distribution of residual stresses (in MPa) are represented by color gradients as a reflection of retardation intensity. The scale at the bottom describes the color representation of stress intensity and percentages of intensity ranges. The maximum value in the scale represents the maximum stress value measured in the veneer layer. Red-green lines surrounding the outer surface of the veneer and the inner surface of the core are artifacts due to the lack of perfect parallelism of the specimens in those regions. Lava Ceram-veneered crowns presented maximum residual stress in the order of 1.8 ± 0.1 MPa for fast cooling and 1.4 ± 0.1 MPa for slow cooling. Maximum stress in fast-cooled Lava Ceram-veneered crowns was concentrated in the occlusal concavity; stresses in the buccal cusp ranged from 0 to 0.6 MPa. Slow cooling of Lava Ceram-veneered crowns resulted in maximum stresses contouring the curved surfaces. (A) Represents a crown that underwent a fast-cooling protocol. The maximum stress of 1.6 MPa was concentrated at the occlusal concavity. Observe the thin band of stress peak in the midplane of the veneer. (B) Represents a crown that underwent a slow-cooling protocol. The maximum stress of 1.5 MPa concentrated on the curved surfaces near the interface.

hand with a decreasing sequence of SiC papers (Buehler), from #600 to #4000 grit, under water lubrication to a final thickness of 1.0 ± 0.02 mm.

The discs were mounted on an universal testing machine (Z 2.5, Zwick, Ulm, Germany) and subjected to a diametral tensile test while transilluminated by a beam of polarized light in a polarimeter (StrainMatic M4-50, ilis gmbh, Erlangen, Germany), which recorded the relative wave retardation at the center of the discs during load application at 50-N intervals up to 500 N (Fig.1). The diametral tensile stress was calculated by:

$$\sigma = 2P/\pi Dt \quad (3)$$

where P is the applied load, D is the diameter of the disc, and t is the thickness of the disc. We determined the photoelastic coefficient, C , by plotting stress vs. polarized light retardation. Three measurements were made for each disc of each veneer. The average C value of 3 discs was used.

Stress Birefringence Measurement

To measure the residual stress magnitude and spatial distribution through the birefringence measurement, we used an automatic imaging measuring system (StrainMatic M4-50, ilis gmbh), which consisted of an automatic polarimeter connected to a computer where the data were analyzed by software (StrainAnalyzer, ilis

gmbh). The polarimeter device was composed of a light source, a detector, 2 polarizers, and a quarter-wave plate. When a linearly polarized light is transmitted through an isotropic material, the field vector resultant is a circle. But when the polarized light goes through a birefringent material, the phase delay forms an ellipse. Determining the ratio between the ellipse axes, one can determine the retardation (Katte, 2009). The quarter-wave plate, placed between the specimen and the second polarizer (called ‘analyzer’), converts the elliptically polarized light back to linearly polarized light. The ellipticity angle is determined by rotating the analyzer. The retardation in nm is calculated by:

$$\delta = \alpha\lambda/180^\circ \quad (4)$$

where α is the angle of rotation of the analyzer, and λ is the light source wavelength in nm (in this study, λ was 587 nm). Fig. 2 shows a specimen on the polarimeter being transilluminated by a beam of polarized light. Due to differences in translucency, Lava Ceram specimens were exposed for 15 ms and VM9 specimens for 10 ms.

The residual stresses were calculated across the veneer thickness of the crowns according to Eq. (2) and could be assessed at any point within the veneer

by means of a software tool.

RESULTS

The photoelastic coefficients calculated for Lava Ceram and VM9 were 9.25 1/TPa and 10.4 1/TPa, respectively. Figs. 3 and 4 show the image results for the residual stress magnitude and spatial distribution for Lava Ceram and VM9, respectively. In Figs. 3a and 4a are illustrated the specimens that were fast-cooled, and on 3b and 4b those that underwent the slow-cooling protocol. Due to the relatively low translucency of zirconia, the coping appears in dark gray, and the veneers are colored. For detailed descriptions of residual stress values and stress gradients across the veneer, refer to the legends of Figs. 3 and 4.

DISCUSSION

Conventional understanding of stress gradients in bilayer systems states that compressive stresses are generated within the veneer near the infrastructure with higher CTE, with tensile stresses developing at the surface of the veneer. That respective stress profile has been observed for porcelain-fused-to-metal specimens with various $+\Delta\alpha$ values (DeHoff *et al.*, 1996, 1998; DeHoff and Anusavice, 1998). For zirconia-Lava Ceram discs, Taskonak *et al.* (2008) observed that fast cooling developed

compressive stress on the surface, whereas tensile stresses were calculated for slow cooling. Finite element simulations conducted by Aboushelib *et al.* (2008) for zirconia-porcelain bars calculated compressive residual stresses of -42.8 MPa at the interface for a core-veneer mismatch of $+1.3$ ppm/ $^{\circ}\text{C}$ and -16.1 MPa for the zirconia-Lava Ceram combination. At the surface of zirconia-porcelain bilayers having 1.0 ppm/ $^{\circ}\text{C}$ mismatch, Swain (2009) calculated compressive stresses for cooling rates of 10 and $50^{\circ}\text{C sec}^{-1}$. For the faster-cooling protocol and thicker porcelain layers (core/veneer ratio of 0.33), due to the low diffusivity of zirconia, high tensile stresses within the veneer were developed, placing the surface under extreme compression. At a core/veneer ratio of 1 , compressive stresses developed throughout the thickness of the veneer, with higher magnitude close to the interface. On a disc made of 1 mm zirconia core and 2 mm VM9 porcelain, Mainjot *et al.* (2011a) reported a parabolic stress profile with high compressive stresses at the surface (-96 MPa), and at the deepest measured point (-40 MPa at 1.3 mm from the surface), with a slight tensile peak ($+4$ MPa) around 0.5 mm from the surface.

These studies show a general trend of compressive stresses being developed in the porcelain near the core and tensile stresses at the surface in metal-ceramic and zirconia-porcelain systems for cooling protocols other than tempering. Nevertheless, most studies calculate or measure stress gradients using simple geometries. For complex geometries, closer to clinical relevance, tensile stresses were found to develop within the porcelain in some FE analyses (De Jager *et al.*, 2005; DeHoff *et al.*, 2006). Moreover, tensile stresses generated within the veneer of lithium-disilicate sphere and cylinder cores were the cause of spontaneous fracture for some CTE combinations (DeHoff *et al.*, 2008). In a recent study by Mainjot *et al.* (2011b), zirconia-VM9 specimens (core/veneer ratio of 0.5) resulted in tensile stresses within the veneer close to the interface for 2 slow-cooling protocols and compressive stresses across the thickness of the veneer for a tempering protocol.

The light retardation as an analogue of residual stresses was measured in the present study as the ratio between the resulting ellipse axes based on the phase delay of polarized light waves. However, the shape change of the resulting field vector during the transition from different stress regions is limited by a 180° ellipticity amplitude. This results in retardation gradients that give no distinction between tensile or compressive stresses. Therefore, any retardation measured and indicated by the color

Figure 4. Residual stress map of VM9-veneered crowns. The dark gray represents the zirconia core. Magnitude and spatial distribution of residual stresses (in MPa) are represented by color gradients as a reflection of retardation intensity. The scale at the bottom describes the color representation of stress intensity and percentages of intensity ranges. The maximum value in the scale represents the maximum stress measured in the veneer layer. Blue lines surrounding the inner surface of the core are artifacts due to the lack of perfect parallelism of the specimens in that region. The stresses in VM9-veneered crowns were distributed similarly for both cooling protocols. Starting at 0.5 mm from the surface, residual stress in the veneer increased toward the core, with maximum stresses located 0.1 mm from the interface along the whole extension of the core, except in the occlusal concavity. Slightly higher stresses were measured within VM9 when the crowns were fast-cooled (6.9 ± 0.2 MPa) than when slow-cooled (5.8 ± 0.2 MPa), with the maximum stress located in the buccal cusp for both cooling protocols. (A) Represents a crown that underwent a fast-cooling protocol. The stresses increased from 0.5 mm from the surface and reached the peak at about 0.1 mm from the interface. The maximum stress of 7.1 MPa was concentrated at the curvature of the buccal cusp, with regions of high stress at the curvature of the palatal cusp and buccal surface. The occlusal concavity developed no residual stress. (B) Represents a crown that underwent a slow-cooling protocol. The stress gradient across the thickness of the veneer and the distribution of stresses are very similar to those of the fast-cooled crown, with a slightly lower maximum stress value (5.9 MPa).

gradients may be interpreted as either tensile or compressive stress (Figs. 3, 4). Nonetheless, ongoing cyclic fatigue experiments being conducted in our lab, in crowns equivalent to those used herein, have found a significantly higher reliability for Lava Ceram-veneered crowns (unpublished observations). Chipped VM9-veneered crowns have shown fractographic patterns suggestive of a weaker plane within the veneer near the interface, matching the high stress band close to the core for VM9 found in the present study. Similar patterns in fractured zirconia-based prostheses, in which the crack plane never reaches the interface, have also been described clinically (Sailer *et al.*, 2007b) and in *in vitro* fatigue experiments (Coelho *et al.*, 2009; Rosentritt *et al.*, 2009). Since the cooling-rate influence has been shown to be small, the stress near the interface as a result of CTE mismatch is expected to be tensile, due to radial and hoop stress components (Tholey *et al.*, 2011), explaining the decreased lifetime for the high-mismatch veneer/core combination.

The cooling rate influence was restricted to the stress distribution for the low-mismatch and to a slight increase in stress magnitude for the high-mismatch combination. Indeed, studies

have shown that the greater alteration in stress magnitude occurs when a tempering protocol is undertaken (Anusavice *et al.*, 1989; Asaoka *et al.*, 1992; DeHoff *et al.*, 1996). For 3 different cooling rates, Choi *et al.* (2011) found a high correlation between residual stress magnitude in zirconia-veneered specimens with different mismatches and the temperature difference between surface and inner porcelain at T_g . Among the cooling rates evaluated, residual stresses were low for the slow- and the fast-cooling protocols, while tempering resulted in high stress values. The small difference in stress values between fast- and slow-cooling protocols recorded in the present study indicates that the fast-cooling rate used (600 °C/min) was insufficient to create a temperature gradient within the veneer that would result in high stress build-up. Nonetheless, cooling rates determined by the opening of the furnace door are dependent on the door-opening system and may vary (Tholey *et al.*, 2011).

The concentration of stresses and maximum stress values occurred, in general, over curved surfaces of the coping for both veneers and cooling protocols, indicating the importance of geometry in the stress distribution. The stress vector orientation has shown a sudden change (~180°) at the tip of the cusps in the bucco-palatal plane (not shown), pointing to a more complex strain behavior over curved surfaces, which is aggravated on cusps due to the small radius and two-dimensional convexity. The low values recorded for residual stresses in the present study in comparison with those in the aforementioned studies might be the result of neglect of the stress component in the z-direction and the effect of a possible elastic strain release due to sectioning. Nevertheless, this study showed a four- to five-fold stress magnitude escalation, with an increase in thermal mismatch from +0.3 ppm/°C to +1.4 ppm/°C for the core/veneer ratio used, evidencing the main role of thermal mismatch in stress build-up.

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