Fatigue Behavior of the Resinous Cement to Zirconia Bond

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Abstract

Purpose: Resinous cements are widely used for luting zirconia restorations. Adhesive failures have occurred at the cement/zirconia interface, rather than at the cement/dentin interface, suggesting that the cement/zirconia bond may lack durability; however, few comprehensive, comparative evaluations of fatigue effects have been reported. The rate of fatigue-induced loss of bond strength may be a more important predictor of long-term success than a single snapshot of bond strength after an arbitrary number of thermocycles. Previous studies have failed to identify trends by investigating bond strengths at several different numbers of cycles. This may result in invalid conclusions about which cements have superior bond strengths. The purpose of this study was to investigate the effects of artificial aging by thermocycling and resinous cement type on bond strengths to zirconia.

Materials and Methods: The effect of the number of thermocycles (0, 1, 10, 100, 1000, and 10,000) on the bond strengths of five resinous cements, two of which were used with and without a primer, and an oxygen-inhibiting gel, was studied. Specimens were randomly assigned to thermocycle number/cement-type test groups. Because zirconia has a very low thermal diffusivity, exceptionally long thermocycle dwell times were used. Cylinders of zirconia were bonded end-to-end. One end of each bonded specimen was insulated, specimens were thermocycled and tested in shear, and bond strengths were calculated and analyzed.

Results: Two-way ANOVA revealed that the effects of cement type, the number of thermocycles, and their interaction all significantly affected bond strength (p < 0.0001). By 10,000 cycles, most cements had lost at least half of their initial bond strengths, and two cements effectively recorded zero bond strengths. Failure modes were cement specific, but adhesive modes predominated. Fatigue resistance of two cements was greatly improved by use of a primer and an oxygen-inhibiting gel, as recommended by their respective manufacturers.

Conclusions: Both the type of resin cement and the number of thermocycles influenced bond strength. Fatigue through thermocycling affected different cement types in different ways. Some materials displayed more rapid loss of bond strength than others. Cements differed in their failure modes.

Zirconia, an inert metal-oxide ceramic, has become widely used as a core material for fixed dental prostheses (FDPs) due to its high flexural strength, high fracture toughness, resistance to chemical corrosion, biocompatibility, and an acceptable appearance.1-4 A variety of processing techniques have been developed to form zirconia crown copings, FDP frameworks, implant abutments, and implant prosthesis frameworks.

Air abrasion, using alumina particles to create micromechanical retention at the surface area, is often used to improve bond strength and durability.5,8 Although zirconia has good mechanical properties, it is relatively difficult to roughen using air abrasion or chemical treatment.9,10 Because vigorous air abrasion may create microcracks and weaken ceramic materials, higher pressures and larger particle sizes have generally not been recommended for use on zirconia.

Resinous cements have stronger tensile and shear bond strengths to zirconia than other cement types, making them the preferred choice for clinical use.11-15 However, several bond strength studies have reported that adhesive failures have occurred at the resin/zirconia interface, rather than at the cement/zirconia interface.
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Figure 1 Lower right first and second molars. A zirconia-based crown fell off the first molar, but its resinous luting cement remained adherent to the tooth preparation. The porcelain on the zirconia-based crown on the second molar underwent cohesive failure.

Figure 2 Cylindrical zirconia test specimens were bonded end-to-end, and one side was insulated to ensure differential thermal expansion and contraction during thermocycling.

Figure 3 Plot of individual cement mean bond strengths to zirconia, and associated standard deviations, at different numbers of thermocycles. For individual cements, bond strengths peaked at a low number of cycles, after which bond strength generally decreased in a manner approaching log-linearity.

Anecdotal clinical reports of crown retention problems have also suggested that the cement/zirconia bond may sometimes be weaker than the cement/tooth bond, an unusual situation. Figure 1 displays a tooth preparation, still coated by an adherent layer of resinous cement, after loss of its zirconia crown.

Fatigue resistance is of obvious importance to long-term clinical performance. Thermocycling in an aqueous environment is widely used to simulate mechanical fatigue in the wet oral environment. Temperature changes produced by thermocycling apply mechanical stresses to interfaces between dissimilar materials through differential expansion and contraction. Although some prior research found that thermocycling decreased bond strength to zirconia, contrary results have also been reported.

Zirconia has a very low thermal diffusivity, so it is slow to adjust its temperature to that of its surroundings because it conducts heat slowly in comparison to its volumetric heat capacity or thermal bulk. However, prior studies of the cement-to-zirconia bond generally used short dwell times, typical for evaluation of bonding to metallic or resinous materials. Therefore, the thermocycling dwell times used in prior studies may have produced little differential expansion and little mechanical fatigue. Furthermore, most studies have only used a single number of thermocycles, that is a single "snapshot." This limits understanding of the fatigue process, which tends to be nonlinear. Although the exact cement-to-zirconia bond strength needed to provide long-term clinical performance remains unknown, knowledge of the fatigue behavior of the cement-to-zirconia interface could aid dentists in making material choices.

The purpose of this study was to investigate the effects of resinous cement type, fatigue through thermocycling, and their interaction on the strength of the cement-to-zirconia bond. Four null hypotheses were addressed: the type of cement does not influence bond strength; the number of cycles does not influence bond strength; the number of cycles does not influence bond strength; and the interaction of cement type and number of cycles does not influence bond strength.
bond strength; cement type and cycle number do not interact to influence bond strength; and the type of cement does not influence failure mode.

Materials and methods

Cylinders of yttria-stabilized tetragonal polycrystalline zirconia (8 mm long, 6 mm diameter) were milled from partly sintered zirconia blocks (Aadva Zirconia, GC Advanced Technologies, Costa Mesa, CA) and then fully sintered. The machined ends of the cylinders were abraded using 320 grit sandpaper and air abraded using 50 µm alumina for 10 seconds at 50 psi from a distance of 1 inch. The cylinders were bonded end-to-end using the manufacturer’s protocols for seven adhesive resinous cement groups: GCem Automix (GC, Tokyo, Japan), MaxCem Elite (Kerr, Orange, CA), Multilink with and without Zirconia Primer (Ivoclar Vivadent, Schaan Liechtenstein), Panavia F2.0 with and without Oxyguard (Kuraray, Okayama, Japan), and RelyX Unicem (3M ESPE, St Paul, MN) (Fig 2).

After cementation, specimens were stored in 100% humidity for 1 hour and water for 23 hours before being thermocycled for 0, 1, 10, 100, 1000, or 10,000 cycles. Temperatures of 5°C and 55°C were used. An exceptionally long dwell time of 2 minutes, with a transfer time of 11.5 seconds, was used to ensure that the zirconia cylinders adjusted to temperature before being transferred. A rubber insulating sleeve was placed over one side of each bonded pair of cylinders during thermocycling to produce differential thermal expansion on either side of the cement layer, and consequent mechanical stress.

The specimens were then tested in shear loaded at a 0.1 in/min crosshead speed using a screw-driven universal testing machine (Model 1122, Instron, Canton, MA). The maximum load was recorded in N, and shear bond strengths were calculated in MPa. Failure modes of the specimens were identified using light microscopy (Universa!, Carl Zeiss, Gottingen, Germany) at up to 400× magnification to study the pairs of debonded specimens.

A power analysis for seven levels of cement, six levels of thermocycling, \( p < 0.05 \), and an effect size of eight, assuming a spread of group means from 0 to 40 MPa and a mean standard deviation of 5 MPa suggested that a minimum group size of four specimens was necessary; whereas six specimens were used per group. Seven cement groups were tested at each of six numbers of thermocycles, with six specimens being included within each cement/fatigue subgroup, for a total sample size of 252. Descriptive statistics and means (and associated standard deviations) were calculated for each cement type and thermocycling group. Two-way ANOVA was used to evaluate the significance of cement type and number of thermocycles, as well as their interactions, on bond strength (\( p < 0.05 \)). A priori one-way ANOVA and Tukey multiple pairwise comparisons testing were used to determine which of the seven cement types differed from one another both before any thermocycling and after 10,000 thermocycles (\( p < 0.05 \)).

Results

All four null hypotheses were rejected. Cements responded differently to thermocycling; some types maintained bond strength more effectively than others (Fig 3). Two-way ANOVA revealed that the effects of cement type, number of thermocycles, and their interaction all significantly affected bond strength (\( p < 0.0001 \)) (Table 1). Cements also differed in failure mode. Multiple comparisons testing before thermocycling grouped materials, from strongest to weakest bond strengths, such as [GCem, Panavia, Panavia F2.0 without Oxyguard, MaxCem], [Panavia F2.0 without Oxyguard, MaxCem, RelyX], [MaxCem, RelyX, Multilink], and [RelyX, Multilink, Multilink without Primer].

At 10,000 cycles most cements had lost at least half of their initial bond strengths, and two cements recorded zero (or nearly zero) bond strengths (Multilink without primer and MaxCem) (Fig 3, Table 2). Multiple comparisons testing by cement type of bond strength after 10,000 thermal cycles showed that Panavia F2.0 differed from all other materials; GCem, RelyX, and Multilink were ranked together as a secondary group. RelyX, Multilink, Panavia F2.0 without Oxyguard, MaxCem, and Multilink without primer were ranked together as a tertiary group.

For individual cements, substantial fatigue effects only became evident after a number of thermocycles; thereafter, bond strength was lost in an approximately log-linear manner (Fig 3). These individual bond strength fatigue trends were described by simple mathematical functions of the form: \( y = -m \ln (n \text{ of cycles}) + c \), where \( y \) = loss of strength, \( m \) = slope, and \( c \) = projected x-axis intercept (Table 3).

Only GCem exclusively recorded cohesive failure modes. Only Panavia F2.0 and Panavia F2.0 without Oxyguard recorded adhesive failure modes exclusively. All other cements exhibited mixed failure patterns, but adhesive modes were predominant. Failure modes were cement specific, but were not influenced by the number of thermocycles.

Discussion

This study used a full block design, large sample size, and long dwell times to provide rigorous thermocycling, six different numbers of cycles, and a powerful statistical approach to evaluate the effect of fatigue by thermocycling. This allowed fatigue trends to be more clearly identified than in most prior studies, which generally used shorter dwell times and fewer numbers of cycles, or just a single number of cycles. 5,7,9,15,19,21,24-29

Certain cement types maintained higher bond strengths to zirconia than others through the progression of thermocycling. The highest overall bond strengths were attained using Panavia F2.0 and GCem cements; whereas RelyX, Panavia F2.0 without Oxyguard...
Individual bond strengths tended to peak after some thermocycling (Table 2, Fig 3). This can be attributed to the heat from the 55°C water bath aiding in resinous polymerization of the cements, and to stress relaxation following water imbibition. Prior studies have also found that low numbers of thermocycles can increase bond strengths of resinous cements to a variety of substrates, as was found at 100 cycles in this study.15,34 Individual bond strengths tended to decrease after additional cycling. The decrease in strength was attributed to the fatigue of the bond by the expansion and contraction of the zirconia specimens and the innate weakening of the bond through the fatigue process. Some cements fared better than others through the fatigue process (Tables 1 – 3, Fig 3).

The interaction between cement type and number of thermocycles was highly significant (p < 0.0001) (Table 1). That is, thermocycling affected different cements in different ways. This explains in part why different studies that used “snapshots” of a single number of thermocycles have sometimes produced conflicting results. In this study, the use of many different numbers of thermocycles allowed fatigue trends to be discerned.

Interestingly, the two cements with the highest final bond strengths (GCem, Panavia F2.0) underwent completely different failure modes; GCem failing cohesively, and Panavia F2.0 failing adhesively. The cement that underwent cohesive failure without adhesive failure, GCem, may have had superior bond strength, or inferior cohesive strength, or both, in comparison to the other materials.

It was clear that failure modes differed among cements and that they underwent different types of failure mechanisms, but the precise effects on clinical performance are unknown. The ex vivo setting of this study limits direct clinical application of the results; however, the effects of two clinically important variables were isolated and measured, and trends were clearly identified. The relationship between number of thermocycles and duration of clinical service is unknown. Likewise, the relationship between bond strength and clinical performance is unknown. The role of bond strength may be confounded by a variety of clinical variables such as preparation form, masticatory force, and habit; however, some investigators have suggested thresholds of 10 MPa or more for “acceptable” or “sufficient” clinical bond strength.25,30 After 10,000 thermocycles, only Panavia F2.0 and GCem exceeded this value.

The importance of compliance with manufacturers’ instructions was clearly demonstrated. Although Panavia F2.0 cemented without Oxyguard recorded bond strengths comparable to the other cements, the application of Oxyguard to Panavia F2.0 produced significantly stronger bonds and a marked improvement in fatigue resistance (Fig 3, Table 2). Blocking oxygen allows for more complete polymerization of the Panavia F2.0 in the clinically and mechanically important marginal region. Likewise, the application of a Multilink primer to zirconia showed significantly higher mean bond strengths and also increased the durability of the bond. The Multilink zirconia primer is an unfilled surface-active resin designed to create a superior surface physico-chemical interaction for the filled Multilink cement. Without the primer, Multilink demonstrated the lowest bond strengths at every thermocycle interval and only one of the six specimens was able to endure 1000 cycles.

The laboratory setting facilitated precise manipulation of the cements that had more complicated instructions and more steps.
The only previous study but a couple of studies found that thermo-cycling had significant effects on bond strength to zirconia. This study showed that the weakest bond strength to zirconia was achieved after 10,000 cycles, but that its bond strength rapidly deteriorated to negligible levels at 10,000 cycles. Some studies have identified that included GCem Automix ranked it as having the highest shear and tensile bond strengths among the included cements.

In summary, cement rankings changed with numbers of thermocycles. After a low number cycles, a near logarithmic loss of bond strength occurred for this material class. These data emphasize the need for including a high number of cycles, and examining trends, not snapshots, before drawing conclusions or making clinical material choices.

Conclusions

Within the limitations of this study, the following conclusions could be drawn:

1. The type of resinous cement very strongly affected bond strength to zirconia.
2. The number of fatiguing thermocycles very strongly affected bond strength to zirconia.
3. Thermocycling affected different cement types in different ways.
4. Cements differed in their failure modes.

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