The use of slow heating and slow cooling regimens to strengthen porcelain fused to zirconia

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Statement of problem. Porcelain fused to zirconia prostheses are widely used. However, porcelain chipping, spalling, fracture, and delamination are common clinical problems. Residual stresses of thermal origin have received attention, but clear data and firing guidelines remain absent.

Purpose. The purpose of this study was to measure the influence of heating and cooling protocols on the strength of porcelain fused to zirconia.

Material and methods. A modified 4-point flexural testing technique was used to measure strength, and porcelain buttons were bonded to the beam between the 2 central loading points. Beams (n=54) were made of a tetragonal polycrystalline zirconium dioxide that was partially stabilized with an yttria core and a feldspathic dental porcelain. Three different heating rates and 3 different cooling regimens were used during firing. Two-way analysis of variance (ANOVA) was used to evaluate the 2 main effects of the heating and cooling regimens and their interaction with the delamination force (α=.05). The Tukey multiple comparisons test was used to identify differences among heating or cooling regimens.

Results. During loading, the porcelain buttons separated from the zirconia beams because of delamination within the porcelain, which was close to the porcelain to zirconia interface. ANOVA revealed that the effects of the cooling regimen and heating rate had statistically significant effects on failure load (P<.05). The effect of the cooling regimen was greater than that of the heating regimen.

Conclusions. Slow cooling and slow heating regimens should be used when firing porcelain to zirconia. Cooling regimens were found to be more influential than heating rates. Failure was localized to the porcelain adjacent to the porcelain-zirconia interface, not to the interface itself, indicating that the residual stresses of thermal origin within the porcelain dominated. The preparation of zirconia with 50 µm aluminum oxide at a pressure of 0.34 MPa was sufficient to prevent interfacial failure. (J Prosthet Dent 2012;107:163-169)

Clinical Implications

The failure of porcelain and its bond to zirconia is known to be the dominant clinical failure mechanism of zirconia based restorations. Meaningful knowledge of the porcelain to zirconia bond can be used to optimize zirconia surface preparation, firing schedules, and framework design.

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Alumina was the first solid-sintered industrial ceramic to be used for prosthodontic purposes.[1] More recently, zirconia frameworks, substructures, copings, and abutments have become widely used.[2-4] Solid sintered zirconia differs from alumina in several ways: increased strength, increased toughness, the property of transformation toughening, decreased elastic moduli, and decreased thermal diffusivity.[3-7] Many zirconia framework materials and associated porcelain products are now commercially available, together with a variety of fabrication techniques and services. For esthetic reasons, most zirconia frameworks or copings are veneered with porcelain.

Although zirconia-based prostheses have high survival rates, failure of the veneering porcelain is a common occurrence. Reported failure modes include surface crumbling, chipping, spalling, fracturing, and delamination.[8-21] Many of these defects occur entirely within the porcelain layer, but some occur close to or at the porcelain to zirconia interface, both clinically[8-21] and in laboratory modeling.[22-33] Residual stresses within porcelain due to thermal incompatibility may be a key cause of clinical porcelain problems.[26,29,31,34-43] Although porcelains designed for application to zirconia frameworks may have appropriate coefficients of thermal expansion, substantial internal stresses can still develop. Zirconia has a low thermal diffusivity; it is slow to adjust its temperature to that of its surroundings because it conducts heat more slowly than its volumetric heat capacity or thermal bulk would suggest. Zirconia has a slightly lower thermal diffusivity than alumina and a substantially lower thermal diffusivity than dental porcelain.[44,45] Therefore, during the firing of porcelain to zirconia substructures, the porcelain will increase in temperature and cool more quickly than the zirconia framework. Furthermore, the thermal diffusivity of zirconia is temperature dependent; it drops substantially at typical porcelain firing temperatures.[44] Thermal diffusivity is also density dependent; a less densely sintered zirconia with a lower density will have a lower diffusivity.[44] Thermal transfer problems are likely exacerbated by the geometry of crowns and partial fixed dental prostheses because porcelain is placed on the outer surfaces, tending to insulate the zirconia core. Also, zirconia frameworks, particularly fixed dental prosthesis connectors, tend to be bulkier than the veneering porcelain, again exacerbating the effects of differential heating and cooling rates. Residual stresses produced by differential cooling rates may produce fractures at stress levels far below the true value of a stress-free bond.[46] Residual stresses within layered ceramic prostheses could account for many of the reported failures of porcelain veneered to zirconia substructures.

The choice of heating and cooling regimens during porcelain firing has the potential to increase or reduce internal stresses in layered porcelain and zirconia prostheses. Therefore, much attention has focused upon the effects of residual thermal stresses, thermal compatibility, firing schedules, and prosthesis design.[22-32,34-41,43,47-49] Several experiments have used conventional shear or microtensile bond tests; however, such tests are poorly suited to studying fracture in brittle interfaces with uneven stress distributions, flaws created during specimen preparation, load application difficulties, elastic modulus mismatches, pretest failures, and failure modes that may be variable, complex, or unknown.[38,49,50] Such technical difficulties were also encountered several decades ago when much attention was focused upon the bond between porcelain and the recently introduced base metal alloys. Caputo et al[51] developed a modified 4-point flexural test for that purpose. Their approach was subsequently analyzed and validated by others, and the necessary experimental parameters were defined.[52,53]

The purpose of this study was to measure the influence of heating and cooling protocols on the strength of porcelain fused to zirconia with a modified 4-point flexural test. The null hypothesis was that heating and/or cooling rates would not influence the strength of porcelain fused to zirconia beams.

MATERIAL AND METHODS

The modified 4-point flexural technique described by Caputo et al[51] was used (Fig. 1). In this test, porcelain is bonded to the beam between the 2 central loading points, only in areas of force magnification. Analysis has shown that, in this model system, bond separation is probably caused by tensile forces.[53] Advantages include ease of specimen fabrication, clinically relevant material thicknesses,

1 Beam design and test configuration. In this 4-point test, lower surface of each beam was placed in tension, whereas upper surface was placed in compression.

ease of testing, and failure occurrence at a predictable location; that is, under a line of force application.[53] The major disadvantage of this test is the absence of an analytical solution that would allow the calculation of bond strength and the influence of basic material properties such as elastic modulus and Poisson's ratio.[53] A pilot study was used to validate the test method for evaluating the porcelain to zirconia bond.

Zirconia beams, 31 mm in length, 6.5 mm in width, and 1.35 mm in height were fabricated (Lava All-Ceramic System; 3M ESPE, St Paul, Minn). The beams were prepared for porcelain application with airborne-particle abrasion; the tip was held 20 mm from the beam surface by using 50 μm diameter alumina particles at a pressure of 0.17 MPa. The zirconia beams were then steam cleaned (X3 Steamer; Amann Girrbach AG, Koblach, Austria), and the area for porcelain application was delineated with a wax pencil (VT741 Prismacolor Verithin; Newell Rubbermaid, Oak Brook, Ill). Rectangular porcelain buttons, 6.5 mm in length, 6.5 mm in width, and 4 mm in height were fired to the central parts of the beams. The height of the buttons was more than twice that of the beams to produce interfacial failure where the porcelain terminated under the line of force application.[53] The height of the porcelain buttons was important because thinner layers of porcelain generally undergo tensile failure on flexure, rather than interfacial delamination. To ensure a high degree of uniformity, the thickness of individual beams and buttons varied by less than 0.05 mm and the width by 0.1 mm, as measured with digital traveling micrometers with an accuracy of 0.0004 mm (Model 1337; Boeckeler Instruments, Tucson, Ariz) and a toolmaker's microscope (Unitron, Newton Highlands, Mass).

To simulate routine technical procedures, the porcelain buttons (VITA VM9, Vita Zahnfabrik, Bad Säckingen, Germany) were fabricated by using 4 firing cycles (Vita Vacumat 40T; Vita Zahnfabrik). This porcelain was recommended for this purpose by its manufacturer and by the manufacturer of the zirconia beams. Specimens each had a wash firing to 1000°C, 2 dentin firings to 930°C and 920°C, and a glaze firing to 910°C. The wash and dentin firings were under vacuum; the final glaze firing was not under vacuum. To avoid damaging the porcelain to zirconia bond, the porcelain buttons were not adjusted after the final firing.

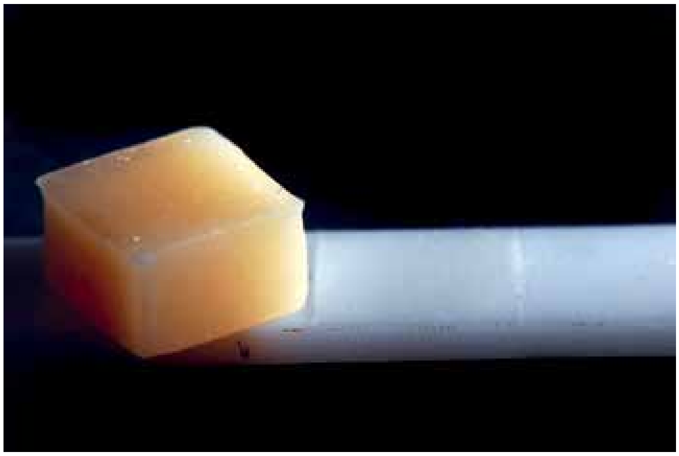
Table I. Porcelain firing schedules. Three heating rates and 3 cooling regimens were used. Specimens had 4 separate firings. Each of 9 heating rate/cooling regimen groups contained 6 individual specimens

Firing	Heating Rate	Vacuum	High Temperature	Cooling Regimen
Wash	25/50/75°C/min	Yes	1000°C	fast/moderate/slow
1st Dentin	25/50/75°C/min	Yes	930°C	fast/moderate/slow
2nd Dentin	25/50/75°C/min	Yes	920°C	fast/moderate/slow
Glaze	25/50/7°C/min	No	910°C	fast/moderate/slow

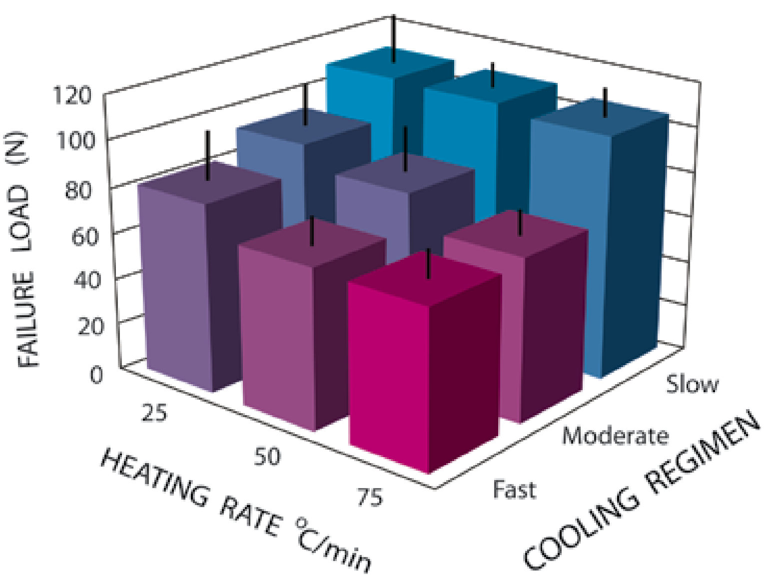
Three different heating rates, 25°C/minute, 50°C/minute, and 75°C/minute were used. Three different cooling regimens were used. The "fast" cooling regimen required removal of the specimen from the muffle as soon as the muffle had fully descended. The "moderate" cooling regimen required the specimen to be left in the fully open muffle for 7.5 minutes until a muffle temperature of 500°C was reached. The "slow" cooling regimen required the specimen to be left in the partially (30%) open muffle for 15 minutes until a muffle temperature of 500°C was reached. Upon removal from the muffle, the specimens were placed on their low density ceramic pillows away from the muffle until ambient temperature was reached, or for at least 1 hour. The oven was placed in a part of the laboratory unaffected by air conditioning ducts, ambient drafts, or other ovens.

These heating rates and cooling regimens were chosen to bracket the range used in routine laboratory practice. The zirconia manufacturer's instructions recommended a heating rate of 45°C/minute (Lava Technical Product Profile; 3M ESPE); the porcelain manufacturer's instructions recommended a heating rate of 60°C/minute (VITA VM9 Working Instructions; Vita Zahnfabrik). The zirconia and porcelain manufacturers' firing instructions did not include cooling regimens. After the presentation of pilot data from this study at a meeting, a summary appeared in an unofficial newsletter (Espertise Scientific Facts, Zirconia-supported ceramic restorations: uncovering the mysteries, 2009, 3M). Based upon the pilot data from this current study, a heat rise of 30°C/minute and slow cooling were then recommended by the zirconia manufacturer (3M ESPE). The heating and cooling regimens used in this current study bracketed both the original and the updated manufacturer's recommendations.

A randomized full block design containing all 9 possible heating rate and cooling regimen combinations was used (Table I). Each of the 9 heating rate/cooling regimen groups contained 6 specimens. A power analysis based upon pilot data indicated that this sample size was approximately twice that needed to identify a 20 N difference in failure load with a .05 probability. Descriptive statistics, means, and associated standard errors for delamination forces were calculated. Two-way analysis of variance (ANOVA) was used to evaluate the 2



2 Specimen after testing. Porcelain button separated from zirconia beam. However, thin layer of porcelain remains on zirconia beam; failure occurred within porcelain, close to, but not at porcelain-zirconia interface.



3 Failure loads of porcelain fused to zirconia specimens plotted against heating rate and cooling regimen. Group means and associated standard deviations are shown. Slower cooling regimens and slower heating rates resulted in higher failure forces. Effect of cooling regimen as greater than that of heating rate.

TABLE II. Analysis of variance for main effects of cooling regimen and heating rate and their interaction on specimen strength

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Squares	F	*P*
Cooling regimen	12243	2	6122	45	<.001
Heating rate	1790	2	895	7	.002
Interaction	654	4	163	1	.3
Residual	5775	45	128		
Total	20461	53			

main effects of the heating and cooling regimens and their interaction with the delamination force (α=.05). In the event of the main effects being statistically significant, the Tukey multiple comparisons test was used to identify differences among heating or cooling regimens (α=.05).

RESULTS

Fractography

During flexural loading, all of the porcelain buttons separated from the zirconia beams because of delamination close to, but not quite at, the porcelain to zirconia interface. A thin layer of porcelain remained on every zirconia beam (Fig. 2). This finding indicated that the porcelain adjacent to the interface was the weak link, not the interface itself. The failure location suggested that substantial internal stresses existed within interface-adjacent porcelain. The survival of the interface indicated that the surface preparation of the zirconia was adequate. The porcelain buttons remained intact, indicating that tensile failure of the bulk porcelain buttons did not occur. Similarly, the zirconia beams remained intact. The fracture surfaces of the porcelain buttons often became slightly concave after separation from the zirconia beams, indicating the presence of internal stresses (Fig. 2). This methodology produced a single simple failure mode.

Effects of Heating and Cooling Regimens

Slower cooling and heating regimens resulted in higher failure forces; these trends were clearly visible when group means were plotted in a 3-dimensional (3-D) bar graph (Fig. 3). ANOVA revealed that the effects of the cooling and heating regimens had substantial and highly significant effects on failure load, <.001, and .002 respectively (Table II). These 2 factors accounted for most of the experimental variance. The effect of the cooling regimen was almost an order of magnitude larger than the effect of the heating regimen. The interaction between heating and cooling regimens was small in magnitude and was not statistically significant; therefore, the main effects of the cooling and heating regimen operated separately and independently. Multiple comparisons testing revealed that all 3 cooling regimens differed from one another, whereas only the fastest and slowest heating regimens differed from one another (*P*<.05).

DISCUSSION

The data support rejection of the null hypothesis (Table II). The slow heating/slow cooling group was approximately twice as strong as the fast heating/fast cooling group. Although the results based on this in vitro model cannot be directly extrapolated to clinical outcomes, such a difference is likely to have clinical relevance given that the cohesive failure of veneering porcelain is a common failure mode for porcelain fused to zirconia prostheses. It is the responsibility of manufacturers, technicians, and dentists to provide patients with predictable products. Slow heating and slow cooling regimens should be routinely used during the fabrication of porcelain fused to zirconia prostheses.

By necessity, the exact conditions of the experiment were unknown. Although nominal heat rise rates could be programmed into the oven, the actual heating rates of the specimens were unknown. The cooling rates of the oven could not be programmed or controlled, so different regimens were chosen, as they would be in routine laboratory practice. During the pilot study, unsuccessful attempts were made to measure specimen surface temperatures with an infrared sensor during the cooling phase of pilot experiments. However, valid comparisons could be made among different heating and cooling groups because other experimental variables were held constant. This approach allowed meaningful recommendations to be made because overall trends were clearly discerned and because the experimental protocols reproduced the steps that dental laboratory technicians perform to fabricate porcelain fused to zirconia prostheses.

The measured temperatures and heating or cooling rates were those determined by the thermocouple sensor located within the oven itself. Direct measurement of the surface or the internal and interfacial specimen temperatures was not possible. The placement of thermocouples within the small specimens would have been technically challenging and would have altered and confounded the measurement of "normal" temperatures. Unsuccessful attempts were made to use an infrared sensor to measure surface temperatures of the specimens after the muffle opened, that is, during cooling.

In this study, the cooling rate was controlled by setting the time for the muffle thermocouple to reach a target temperature of 500°C upon completion of the firing cycle. However, this target temperature was the thermocouple temperature, not the specimen temperature. Somewhat paradoxically, if a slower cooling regimen is used, the specimen remains closer to the thermocouple and its temperature estimation is more accurate. Additionally, commercial porcelain furnaces differ in shape, size, and opening mechanism. Although most furnaces move a stage up to a muffle, others move a muffle down over a stage, and some open and close like a clam-shell. Uniform cooling may be more difficult to accomplish when a clam-shell furnace opens because the heating element moves eccentrically. Another practical method to control the cooling rate is by controlling the amount that the muffle opens after the firing cycle has been completed. This study was performed in a well-controlled environment, distant from doors, windows, air conditioning vents, drafts, air filters, and other ovens to minimize temperature fluctuations. These all had measurable effects on muffle temperature during the cooling phases of pilot studies.

The use of slower heating and cooling regimens considerably lengthens firing cycle times; this has obvious impacts on technician productivity, furnace use, and cost. The benefit is invisible to the technician, dentist, and patient. Whereas use of slower heating and cooling rates may reduce the risk of porcelain failure, problems may take months or years to manifest themselves. Yet, these problems will be expensive, no matter who pays for a replacement restoration. Although ion-exchange strengthening is known to increase the strength of veneering porcelains,[54] the process does take additional time and its advantages are not evident to the technician, dentist, and patient; it has yet to achieve widespread usage. Perhaps, the presently unacceptable porcelain failure rates will be sufficient to drive the adoption of slower heating and cooling rates.

The findings of this current study are broadly consistent with those of prior studies. Localization of the failure site to porcelain adjacent to the biomaterial interface has been reported by others.[22-33,42] Residual stresses of thermal origin have previously been implicated as causes of decreased biomaterial specimen strength and in veneer chipping.[26,29,31,34-41,43] Comparable data on the effects of heating and cooling regimens are rare. Komine et al[31] concluded that the duration of cooling from firing temperature to room temperature may affect the shear bond strength of veneering porcelain to a zirconia material, depending on the porcelain material used. However, their study only used 2 cooling regimens: removing the specimen directly from the muffle, analogous to the "fast" regimen in this current study; or leaving the specimen beside the muffle for 4 minutes, intermediate between the "fast" and "moderate" regimens used in this study.[31] Guazzato et al[39] concluded that the incidence of spontaneous cracks increased with thicker layers of porcelain and with faster cooling. The authors used 2 cooling regimens, one analogous to the "fast" regimen in this current study and a "normal" regimen that followed manufacturers' instructions, probably intermediate between the "fast" and "moderate" regimens used in this current study.[39] Gostemeyer et al,[41] using a layered beam subjected to flexure and 2 cooling regimens, concluded that slow cooling of zirconia-based prostheses in the region between the porcelain sintering temperature and its glass transition temperature may increase the risk of adhesive delamination failures. However, the different cooling regimens in this current study and in those of Komine et al and Guazzato et al were applied below the porcelain glass transition temperatures.[26,31,39,41,54] No prior study has used a randomized block design to measure the influence of a broad range of heating and cooling protocols on a porcelain fused to zirconia system. This current study greatly extended the breadth of cooling and heating regimens and determined that the effects of cooling and heating regimens were simply summative. Consistent trends were identified with multiple (9) data points, allowing these trends to be generalized.

Although visual inspection of the failed specimens clearly demonstrated that residual porcelain remained on the zirconia beams (Fig. 2), additional fractographic information may be obtained by using scanning electron microscopy (SEM), but the failure mode was clearly cohesive failure of the porcelain, not of the zirconia-porcelain interface. However, SEM and X-ray diffraction analyses may be relevant to understanding the leucite composition, distribution, and consequent thermomechanical properties of veneering porcelains. Leucite content probably varies among commercial products and is generally increased by slower cooling.

This current study focused upon the effects of heating and cooling regimens on internal stresses as manifested by failure forces. Thermal stresses manifested themselves even though a porcelain with a coefficient of thermal expansion (CTE) appropriate to that of its zirconia substrate was used. Previous studies indicate that CTE mismatches can be destructive.[26,36,37] Whereas manufacturers carefully match porcelain CTEs to their intended substrates, it is possible that porcelains may sometimes be used for substrates that were not intended or advised. Porcelain fused to zirconia crowns may often be prescribed without specifying the particular type of zirconia, the desired fabrication process, or the type of porcelain to be used. Knowledge of laboratory processes by dentists and excellent communication between dentists and technicians may help to prevent the adverse consequences of inappropriate material selection or fabrication procedure.

CONCLUSIONS

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

1. Slow cooling regimens should be used when firing porcelain fused to zirconia prostheses.

2. Slow heating rates should be used when firing porcelain fused to zirconia prostheses.

3. Cooling regimens influenced failure loads of porcelain to zirconia more than heating rates.

4. The strength of a model porcelain fused to a zirconia beam was approximately doubled by the use of slow heating and slow cooling regimens.

5. Failure was localized to the porcelain adjacent to the porcelain-zirconia interface, not to the interface itself, indicating that the residual stresses of thermal origin within the porcelain led to its cohesive failure very close to the zirconia cores.

6. The surface preparation of zirconia for porcelain application, with 50 μm aluminum oxide at a pressure of 0.34 MPa was sufficient to prevent interfacial failure.

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