865

Variability in Biaxial and Three-Point Flexure Strength of Dental Porcelain. K.J. ANUSAVICE* and Y.L. TSAI (Department of Dental Biomaterials, College of Dentistry, University of Florida, Gainesville, Florida, USA).

The objective of this study was to test the hypothesis that biaxial flexure (BF) strength data for feldspathic porcelain have a smaller coefficient of variation (CV) compared with data obtained from a three-point flexure (TPF) test. Three laboratories participated in the round-robin tests: University of Florida, Gainesville, Florida (F), Therapeutic Goods Administration, Victoria, Australia (T), and the Medical Devices Directorate, London, England (M). Each lab received experimental opaque and body porcelain frit material and three support bearings for the pin-on-three-ball BF test. Disks, 16 mm in dia. and 2 mm thick, were prepared for the biaxial flexure test and bars, 30 mm long x 5 mm wide x 3 mm thick, were prepared for the TPF test. These specimens were ground through 1 µm alumina abrasive. Loading was applied at a crosshead speed of 0.2 mm/min until fracture occurred. A 50 µm polyethylene or Teflon sheet was recommended between the pin and disk for the BF test. The mean BF strength and 95% confidence intervals for opaque porcelain ranged from 76.9 ± 8.0 MPa (T) to 97.1 ± 6.2 MPa (F). Corresponding BF strength values for body porcelain varied from 59.1 ± 2.5 MPa (M) to 66.7 ± 4.8 MPa (F). The mean TPF strength and 95% confidence intervals for opaque porcelain ranged from 73.7 ± 8.1 MPa (M) to 95.5 ± 12.8 MPa (T). Corresponding TPF strength values for body porcelain varied from 54.9 ± 5.6 MPa (F) to 68.3 ± 5.2 MPa (T). For the three labs, the mean coefficients of variation of BF strength values for opaque porcelain and body porcelain were 10.1% and 7.7%, respectively. The mean coefficients of variation of TPF strength values for opaque porcelain and body porcelain were 14.8% and 11.2%, respectively. Based on Tukey's multiple range test, there were statistically significant differences in strength ($p \leq 0.05$) among the three labs for both porcelains and the two test methods. For five of six cases, the CV of BF strength data was less than that of TPF strength values. This study was supported by NIH-NIDR Grants DE06672 and DE09307. We also acknowledge the porcelain provided by P. Cascone of J.F. Jelenko & Co and the data provided by P. Wassenar (T) and Stephen Crisp (M).

866

Evidence of a critical leucite particle radius for microcracking in dental porcelains. J.R. MACKERT, Jr.*, C.M. RUSSELL, and A.L. EVANS-WILLIAMS (Medical College of Georgia, Augusta, Georgia 30912-1260, USA)

The leucite particles in dental porcelains are often partially encircled by microcracks that are the result of the thermal expansion mismatch between leucite and the surrounding glass matrix. Although the magnitude of the stress at the particle-matrix interface is independent of the particle size (Selsing, *J Am Ceram Soc* 1961;44 419), Davidge and Green showed experimentally that there is a critical particle radius, R_c, below which microcracking is absent (*J Mater Sci* 1968;3:629-634). The critical radius is explained by a Griffith-type energy balance criterion—below R_c, the stress magnitude may be sufficient to cause cracking, but there is insufficient strain energy for the creation of the new surfaces of the microcrack. The purpose of the present study was to determine whether the leucite particle size of a dental porcelain influences the degree of microcracking in the porcelain. Microcrack density (cm^2/cm^3), leucite particle surface area (cm^2/cm^3), and leucite mean volume-surface diameter, $D_{3,2}$ (Underwood, 1970), were determined by quantitative stereology on 10 specimens each of six body porcelains and Component No. 1 of the Weinstein *et al.* patent (1962). The fraction of leucite particles with microcracks around them, f_{mc}, was estimated for each porcelain from the microcrack density and the leucite surface area. The values of f_{mc} were plotted against $D_{3,2}$, and a significant correlation was obtained ($p < 0.05$). Using the equations of Selsing (1961) and Davidge and Green (1968), the critical particle diameter, $D_c = 2R_c$, for leucite was calculated to be 4 µm. The porcelains were partitioned according to whether their mean leucite particle diameters fell above or below D_c, and their values of f_{mc} were analyzed using a permutation test by random resampling. The porcelains with leucite particle diameters below D_c had a significantly lower fraction of cracked particles compared with the porcelains with leucite particles above D_c ($p < 0.05$). Microcracking in dental porcelain can be minimized by reducing the leucite particle diameter to less than 4 µm. This work was supported by NIDR Grant No. DE07806.

867

Thermal Expansion of a Cesium Stabilized High Leucite Porcelain. S.T. RASMUSSEN,* C. I. MCLAREN, C. L. GROH and W.J. O'BRIEN (School of Dentistry, Univ. of Michigan, Ann Arbor, MI, 48109-1078).

Addition of Cesium to a leucite porcelain formulated to have a high content of leucite (Jeneric/Pentron, Inc., Wallingford, CT) was found to: 1) stabilize the high temperature polymorph of leucite (high leucite) at room temp., 2) make the material susceptible to a stress induced phase transformation from high leucite to the low temperature polymorph (low leucite) and 3) be reversible on heat treatment (J.D.R. 74: Abstract # 1232, 1995). Cesium could be useful for altering the properties of leucite based porcelains by replacing some or all of the low leucite with lower expanding high leucite. The components leucite porcelain, Cs_2O, SiO_2 and Al_2O_3 were fired at 1232 °C for 48 hr, quenched in water and ground to powder, which was then fired (20 min. at 1100 °C under vacuum) into specimens for x-ray analysis and thermal expansion measurement. Three solid specimens were ground flat and x-rayed, heat treated (10 min. at 650 °C) and x-rayed for a total of 5 times to obtain a conversion ratio (r = area high leucite/ area low leucite = 1.93) between x-ray peak areas for high and low leucite. A leucite crystal (Ward's Natural Science Establishment, Inc., Rochester, NY) ground to powder was mixed at various wt. % with silica and a copper standard and x-rayed to obtain a quantitative calibration curve for low leucite. Solid specimens were ground to powder, heat treated, mixed (9/1) with powdered Cu, and x-rayed to determine the wt. % of low leucite and high leucite. The wt. % high leucite was determined from the calibration curve using r = 1.93. Specimens containing 0.0, 0.5, 1.0, 1.5 and 2.0 mol % Cs_2O were found to have: 1) 63, 55, 52, 0 and 0 wt. % low leucite respectively, 2) 0, 3, 17, 62 and 67 wt. % high leucite respectively, 3) a total crystalline content of 63, 58, 69, 62 and 67 wt. % respectively and 4) thermal expansion coefficients of 16.6, 16.2, 15.1, 11.3 and 11.4 x 10^{-6}/°C respectively. Cesium was found to control the thermal expansion and proportions of low and high leucite for this porcelain. Supported by NIH(NIDR) grants: NRSA F33 DE05636 and DE09296-05.

868

Investigations into Technique Variability in Bonding Studies J.W. FUNDINGSLAND*, S.M. Aasen, P.D. Ario, E.A. Glasspoole (3M Dental Products Division, St. Paul, MN, USA).

During the development of an experimental dental adhesive, excessive operator variability became evident. The objective of this investigation was to determine the technique sensitive step of the bonding procedure. Two investigators who repeatedly obtained significantly different test results performed the bonding procedure from polishing to application of composite, exchanging samples after individual steps in the sequence. After isolation of the problematic step, additional experiments were conducted to verify the effect. Bonding studies were conducted using bovine enamel. Shear bond values were determined using an Instron testing machine. In the first experiment, the operator that applied the primer/etchant was found to be highly significant (P= .0001; n=5), while the preparation of the tooth (P=.41), application of the adhesive (P=.52), and application of the composite (P.59) were not significant. Mean bond values varied from 10.0 to 25.2 MPa, dependent upon who applied the primer/etchant. In a subsequent experiment, the effect of drying the primer/etchant was evaluated Again, highly significant differences (P=.0001; n=20) were noted between use of an Handi-Dry™ drier, drying using an air/water syringe and forceful drying using laboratory air pressure. Mean bond values ranged from 7.6 MPa using the Handi-Dry drier to 18.1 MPa using laboratory air pressure. In a final experiment, the effect of tooth orientation on the bond obtained with the primer/etchant was investigated. The liquid primer/etchant yielded 19.4 MPa when applied to a horizontal bonding sample, but only 9.6 MPa when applied to a vertical sample. This difference was significant (P=.0001; n=5). While differences in chemistry and test procedure can be significant, variability between operators can be equally significant. Interaction of the chemistry and operator effects are worth additional investigation.

869

Relation between Water Content in Acetone/Alcohol Based Primer and Interfacial Ultrastructure S.H.Y.WEI*, A.J.GWINNETT[1], F.R.TAY (University of Hong Kong. [1]SUNY at Stony Brook, New York)

The "Overdry Phenomenon" and the "Overwet Phenomenon" were used to describe certain micromorphological attributes along the resin-dentin interface under different dry and moist conditions following acid conditioning of dentin. This study investigated the ultrastructure of the resin-dentin interface when a two-bottle primer system containing *NTG-GMA and BPDM was used with different concentration of water as a primer solvent: a)* an experimental version of All-Bond 2 with no water in primer A; b) commercial version of All-Bond 2 (Bisco, Itasca, IL) with 5% water in primer A; and c) former version of All-Bond 2 with 17% water in primer A. Thirty-six 1 mm dentin discs prepared from third molars were each conditioned with 10% H_3PO_4 for 20 seconds and rinsed for 20 seconds. They were randomly divided into 3 groups: Group I: conditioned dentin surface blow-dried for 30 sec; Group II: blow-dried for 3 sec; and Group III: blot-dried so that dentin surface remained visibly moist (Kanca technique). The 3 categories of primers were applied to each disc in 8-10 coats resulting in 9 sub-groups. Discs in each sub-group were bonded together to form a disc-pair using a chemical cure resin, demineralized in EDTA and prepared for TEM examination. With the use of water-free primer version, sub-optimal hybridization characteristic of the "Overdry Phenomenon" was observed whenever dentin was dried prior to bonding (Gp Ia & Ib). In the 5% water version, prolonged desiccation resulted in compromised hybridization (Gp. IIa) while resin globules were observed on the surface of the hybrid layer when a moist technique was employed (Gp. IIc). In *the 17% water version, surface blisters and globules characteristic of the "Overwet Phenomenon" were observed* in Gp. IIIb & IIIc. It was concluded that between the two extremes of a spectrum of bonding conditions, there was a shift of the "window of opportunity" for optimal hybridization and tubular seal depending on the water content of the primer system investigated. (Supported by CRCG Grant, University of Hong Kong)

870

Silane-Modified Dentin Bonding Systems. JE CODE[1], JM ANTONUCCI[2], GE SCHUMACHER[1], PS BENNETT[3]. ([1]NIH, Bethesda, MD; [2]NIST, Gaithersburg, MD; [3]ADAHF, NIST, Gaithersburg, MD).

Silane coupling agents are commonly used to mediate the bonding of dental polymers to siliceous substrates. The aims of this study were to assess by shear bond testing: (a) the effect of the silane 3-methacryloxypropyltrimethoxysilane (MPTMS) on a dentin bonding system comprising N-phenylglycinedioctic acid (PIDAA) as the surface-activating conditioner and photoactivated PMDM as the adhesive monomer and (b) the effect of an intermediary unfilled bonding resin (UR) on these bonding systems. A 2-step bonding protocol was used in (a) and a 3-step protocol in (b). The PIDAA concentration was 0.3 mol/L in 1:1 v/v acetone: H_2O. Two PMDM adhesives, A1 and A2, were prepared: A1, 10% w/w PMDM, 0.05% w/w CQ in acetone (Control); A2, 10% w/w PMDM, 5% w/w MPTMS, 3% w/w HEMA, 0.05% w/w CQ in acetone. A photoactivated unfilled resin based on bis-GMA, TEGDMA, and HEMA was used as an intermediary bonding resin in the 3-step bonding protocol.. The dentin bonding protocols involved the following steps sequentially applied: 1) 20 µL of PIDAA; 2) 20 µL of A1 or A2 light cured (LC) 30 s; 3) addition of UR, LC 30 s. A photoactivated composite was then added to the dentin surface and LC 60 s. Shear bond strength (SBS) results of the modified bonding systems in MPa after 24 h storage in H_2O at 23°C were:

Protocol:	1 & 2 SBS (SD)	1,2 & 3 SBS (SD)
Adhesives	No UR	UR
A1	36.9 (5.1) n=12	36.4 (9.1) n=9
A2	32.4 (9.1) n=13	39.6 (7.2) n=9

ANOVA indicated no significant differences between bonding systems based on adhesives A1 and A2 or among bonding systems based on the adhesives plus the bonding resin ($p \leq .05$). Results indicate that SBSs are not adversely affected by the addition of other bonding components to the PIDAA/PMDM/CQ dentin bonding system. These results suggest that unique bonding systems for other applications, e.g., composite-to-porcelain, utilizing PIDAA/PMDM/CQ may be feasible. Supported by NIST, NIH, and the ADAHF Paffenbarger Research Center.

871

Stabilization Effect of NPG on Demineralized Dentin and its Mechanism. T. NIKAIDO[1]* and J. M. ANTONUCCI[2] ([1]Tokyo Medical and Dental University, Tokyo, Japan, [2]NIST, Gaithersburg, MD, USA).

N-Phenylglycine (NPG) has been shown to be an excellent primer in dentin bonding systems. In this role, NPG functions as a coinitiator for interfacial polymerization. In this study, the role of NPG and analogous aliphatic and aromatic amino acids in the stabilization of demineralized collagen was assessed. Human molars were used to make dentin discs. Superficial or middle portions of the occlusal dentin surfaces were ground with 320-grit SiC under water. Then a microfilled resin was placed on the center of the dentin surface, light-cured for 60s to preserve the original ground surface against the subsequent acid etching. Dentin surfaces were treated with 20 µL of 40% H_3PO_4 for 30s, rinsed and dried (control). Then 20 µL of either acetone (A), 0.3M NPG in acetone (NPG), 0.3M N-phenylalanine in acetone (NPA), saturated N-methylglycine in acetone (NMG/A), 0.3M NMG in acetone/water (1:1v/v) (NMG/AW), or 0.3M ethyl benzene in acetone (EB) was then applied. The specimens were stored at 23°C for 1d, and then dried under vacuum for 2h. The discs were then fractured into two halves. SEM photographs of the saggitally-sectioned dentin surfaces were taken, and the difference (Δ) in the height of the collapsed demineralized dentin surface from that of the undemineralized dentin was measured; n=6 for each group. The data were statistically analyzed using one-way ANOVA at the 5% level. The values Δ (µm) were; 6.5±0.5 (control), 7.4±0.6 (A), 3.3±1.0 (NPG), 4.0±0.6 (NPA), 6.2±1.1 (NMG/A), 5.4±0.9 (NMG/AW), and 7.0±1.1 (EB). Of the amino acids, only NPG and NPA, the surface-active aromatic amino acids, showed significant potential for stabilizing the demineralized collagen network. Neither the hydrophilic solvent, acetone, nor the hydrophobic ethyl benzene showed any stabilization potential compared to the control. The mechanism of this stabilization may involve several factors such as the formation of new hydrogen and ionic bonds between the amino acids and the demineralized collagen network. This study was supported by Tokyo Medical and Dental University and NIST.

872

Chlorhexidine's effect on shear bond strength of orthodontic brackets. PL DAMON*, SE BISHARA, ME OLSEN, and JR JAKOBSEN. (The University of Iowa, Iowa City, IA USA).

The purpose of this study was to determine whether the application of chlorhexidine to etched enamel as an antibacterial agent will affect the shear bond strength and debonding failure modes of orthodontic brackets.

Forty recently extracted teeth were cleaned and randomly divided into two groups; the first group was etched with a 37% phosphoric acid gel and then a primer was applied containing a chlorhexidine varnish. Metal orthodontic brackets were then bonded using the Transbond XT bonding system (3M/Unitek). The second group was bonded using the same system but without chlorhexidine. A Zwick Universal Testing Machine was used to determine shear bond strengths. The residual adhesive on the enamel surfaces was evaluated using the Adhesive Remnant Index. The analysis of variance and Chi square tests were used to compare the two groups.

The results indicated that there was no significant difference in bond strengths between the chlorhexidine ($\bar{x}$ = 11.8 ± 2.1 MPa) and non-chlorhexidine ($\bar{x}$ = 12.4 ± 3.1 MPa) treated teeth (p = 0.29). The Chi Square test evaluating the residual adhesive on the enamel surfaces also showed no significant difference (p = 0.136) between the two groups. In conclusion, the use of a primer containing chlorhexidine did not significantly affect the shear bond strength nor the bond failure location during debonding. Supported by the Univ. of Iowa College of Dentistry Dows Institute Research Program.