

Strength and fatigue of polyacid-modified restorative materials (compomers)

KEN G. BOBERICK

Temple University School of Dentistry, 3223 N. Broad Street, Philadelphia, PA 19140, USA

JOHN I. MCCOOL

Penn State Great Valley, 30 E. Swedesford Road, Malvern, PA 19355 USA

GEORGE R. BARAN*

Temple University College of Engineering, 1947 N. 12th Street, Philadelphia, PA 19122, USA

E-mail: GRBaran@astro.temple.edu

The purpose of this study was to compare the fatigue behavior of a hybrid composite, four compomers, and two viscous glass ionomers after short- and long-term soaking in distilled water.

Bars with dimensions of 30 mm × 2 mm × 2 mm were formed in stainless steel molds, finished with 600 grit SiC, then soaked for either 24 h or one year, and tested in 3-point flexure at stressing rates between 0.001 and 2500 MPa s⁻¹.

Data were plotted as fracture stress vs. stressing rate, and the exponent *N* in the power law for crack growth rate was computed from the slope of these plots. All compomer and resin composite materials tested exhibited subcritical crack growth (i.e. a reduction in strength with a decrease in stressing rate). Soaking lowered the slope for all materials, that is, increased the crack propagation rate. F2000 exhibited an increase in fracture strength while Hytac and Compoglass exhibited a decrease following long-term soaking in distilled water. Crack-growth exponents obtained from these fatigue data were used to estimate the stresses which would result in a five-year lifetime for these materials.

© 2002 Kluwer Academic Publishers

Introduction

Until recently, dentists were limited to two major groups of direct tooth-colored restorative materials: conventional glass ionomer materials and composite resins. Glass ionomers undergo setting through an acid–base reaction between an ion-leachable glass and an aqueous polyacid, and have as an advantage the ability to release fluoride [1]. Resin composites are materials composed of inorganic filler particles, usually silica, embedded in a polymerizable organic matrix, usually a blend of BIS-GMA and TEGDMA. Their toothlike appearance and insolubility in oral fluids has made them popular as restorative materials for aesthetic applications; however their high shrinkage on polymerization and high coefficient of thermal expansion can lead to micro-leakage and premature failure [2].

Attempts to combine glass ionomers with resin composites have resulted in the development of hybrid materials called polyacid modified resin composites (compomers). Compomers contain the essential components of a glass-ionomer cement but at levels that are insufficient to produce an acid–base reaction in the dark [3]. With a high resin content, it is expected that these

compomers behave more like resin composites than glass ionomers, and the physical properties and handling characteristics of these materials are determined by the degree to which the material has been modified. These new hybrid materials are intended to overcome some disadvantages of conventional glass-ionomer cements, for example short working time, long setting time, poor fracture strength, and sensitivity to water during the early stages of setting [4–6], while preserving their clinical advantages (aesthetics, self-adhesion to dental tissues, and fluoride release) [6–8]. Studies have also shown that the hybrid materials are not in every way superior to conventional glass ionomers; for example, the compomers exhibit lower wear resistance [6, 9].

Because composites are now routinely used in load-bearing restorations, they are subject to fatigue and are evaluated for their response to fatigue in laboratory testing. If compomers are to be considered as alternatives to composites, then it is of interest to characterize their fatigue behavior as well. The purpose of this study was to examine and compare the fatigue behavior of a hybrid resin composite, four polyacid modified resin composites (compomers), and two highly viscous glass ionomer

*Author to whom all correspondence should be addressed.

TABLE I List of materials tested

Material/manufacturer		Lot number
Z100/3M (St. Paul, MN, USA)	Composite	19981109
Hytac/ESPE (Seefeld, Germany)	Compomer	029
F2000/3M (St. Paul, MN, USA)	Compomer	19981110
Dyract/Dentsply (De Trey-Dentply, Konstanz, Germany)	Compomer	9605178
Compoglass/Ivoclar-Vivadent (Amherst, NY, USA)	Compomer	800045
Fuji IX capsulated/GC (Tokyo, Japan)	Highly viscous glass Ionomer	020687
Ketac-Molar Aplicaps/ESPE (Seefeld, Germany)	Highly viscous glass Ionomer	Fw0038691

materials (VGI) using a dynamic fatigue test. This test was first described by Evans [10] and later defined as a distinct test modality by Ritter [11]. In dynamic testing, fracture strength is determined as a function of constant stressing rate. Typically, the fracture strength of materials exhibiting fatigue decreases with decreasing stressing rates due to the growth of the critical flaw during the test. Fairhurst *et al.* [12] successfully used dynamic fatigue testing to evaluate the fatigue parameters of a model experimental porcelain, but the test has also been applied to less brittle materials such as polymers [13].

Materials and methods

One hybrid composite, four polyacid-modified resin composites (compomers), and two conventional glass ionomers were investigated as listed in Table I. Bar-shaped specimens were made of each product in a split stainless steel mold. The materials were syringed into the mold, and the assembly was then sandwiched between two pieces of clear glass. The molds had been sprayed with a dry Teflon separating medium to facilitate separation of the specimen from the metal mold.

The hybrid composite and the compomers were placed into a Triad[™] light-curing oven and cured for two minutes on each side. Light-cured materials were finished using 600 grit SiC paper immediately after curing, and stored in distilled water at 37 °C for 24 h or one year before testing. The highly viscous glass ionomer materials (Ketac-Molar, Fuji-IX) were fabricated according to instructions received from the manufacturer. They were allowed to bench cure in the molds for 20 min at 100% relative humidity, removed from the molds, coated lightly with resin, and stored for 24 h in distilled water at 37 °C. They were then finished with 600 grit SiC paper, and stored in distilled water at 37 °C for either an additional 24 h (dry specimens) or for one year before testing (soaked specimens).

The flexural strength of the materials was measured in three-point bending. Fifteen to twenty beams were fabricated for each speed tested. Specimens were tested in bending mode after placing the beams in a jig with a distance of 20.0 mm between the lower supporting members. The jig was mounted in a servohydraulic testing machine and the beam was centrally loaded with stressing rates ranging between 0.001 and 2 500 MPa s⁻¹. Both the load at fracture of the specimen and the deformation of the beam at fracture were recorded. Flexural strength (σ) was calculated using the following formula:

$$\sigma = 3PL/2bd^2 \quad (1)$$

where P = load at fracture; L = distance between the lower supporting members; b = width of the beam (measured at fracture point); and d = thickness of the beam (measured at fracture point).

Results

Plots of log of fracture strength vs. log stressing rate for both wet and soaked specimens are shown in Figs 1–7. The slopes of the regression lines shown in these plots and their standard deviations were computed and are shown in Table II. The slopes are directly related to the exponent in a crack-growth law as discussed below. It may be noted that for each material, the soaked specimens had a lower slope than the dry specimens, a finding strongly indicative of an effect of soaking on subcritical crack growth. To formally assess the effect of soaking on the slope and hence on the crack-growth exponent N , the difference in the dry and soaked slopes for each material were computed and divided by the estimated variance of the difference

$$\left(\sigma_d = \sqrt{\sigma_{dry}^2 + \sigma_{skd}^2}\right)$$

Under the hypothesis that soaking had no effect, the computed value of the scaled difference z follows an approximate standard normal distribution. If soaking lowers the slope, the computed value of z will be large and the p value for a one-sided hypothesis test will be small. The resultant values of z and p are shown in Table II. Significant ($p < 0.01$) reductions in slope with soaking are exhibited by Compoglass, Dyract, and F2000.

To compare the slopes and hence the crack-propagation exponent among the materials, we normalized each slope by its estimated standard error and the resultant values are shown in Table III for dry and soaked specimens. They are approximately normally distributed random variables with unit variance. If the slope were zero, that is, if there were no subcritical crack growth, the standardized slopes would, with 95% probability vary over the interval ± 1.96 . Since none are smaller than about 4.0, we take this as evidence that some subcritical crack growth occurred in all seven materials in both the dry and soaked states. To discover if slopes differ among materials, we first observed that if there were no difference in slopes among the seven materials within each set, the range should be consistent with the Studentized range q within samples of size 7 drawn from a normal population with unit variance. From tables of the Studentized range the upper 10% critical value in

TABLE II Pairwise comparisons between the slopes of wet and dry materials, and standard deviations of slopes

Material	Dry (slope)	σ Dry (slope)	Soaked (slope)	σ Soaked (slope)	Z	p
Compoglass	0.094545	0.005169	0.050951	0.002936	7.3265	0.0000
Dyract	0.054749	0.004340	0.042415	0.002656	2.4144	0.0078
Hytac	0.040620	0.010240	0.037025	0.002752	0.3390	0.3672
Z100	0.036373	0.003596	0.031056	0.005228	0.8464	0.1986
F2000	0.122809	0.004414	0.037661	0.004893	12.919	0.0000
Ketac-Molar	0.057647	0.007067	0.050430	0.010290	0.5781	0.2816
Fuji IX	0.051895	0.006063	0.037377	0.007535	1.470	0.0704

groups of size 7 is 3.81. Any normalized value in Table III among the dry and soaked specimens which differ by more than 3.81 may be said to differ significantly with an experimentwise type I error rate of 10%. The table below shows the absolute pairwise differences among the seven materials in the dry state. Non-significant differences are shown shaded.

	Compoglass	Dyract	Hytac	Z100	F2000	Ketac-Molar	Fuji IX
Compoglass	*	5.68	14.31	8.0	9.54	10.12	9.73
Dyract	—	*	8.63	2.4	15.2	4.44	4.04
Hytac	—	—	*	6.31	23.9	4.19	4.59
Z100	—	—	—	*	17.52	10.12	1.72
F2000	—	—	—	—	*	19.7	19.26
KetacMolar	—	—	—	—	—	*	0.4
Fuji IX	—	—	—	—	—	—	*

Thus, among the dry specimens, the slopes of Z100, Fuji IX and Ketac-Molar are not significantly different from each other, and Dyract and Z100 cannot be distinguished from each other. All other pairwise differences are significant.

The following table shows the paired differences for the soaked specimens.

	Compoglass	Dyract	Hytac	Z100	F2000	Ketac-Molar	Fuji IX
Compoglass	*	1.39	3.9	11.41	9.66	12.45	12.53
Dyract	—	*	2.52	10.03	8.28	11.07	11.15
Hytac	—	—	*	7.51	5.76	8.55	8.63
Z100	—	—	—	*	1.76	1.04	1.12
F2000	—	—	—	—	*	2.8	2.88
KetacMolar	—	—	—	—	—	*	0.8
Fuji IX	—	—	—	—	—	—	*

Among the soaked specimens, the results cluster into two groups: Compoglass, Dyract and Hytac are indistinguishable from each other as are Z100, F2000 Ketac-Molar and Fuji IX. Every material in the first group differs significantly from every material in the second.

TABLE III Standardized slope values

Material	Dry	Soaked
Compoglass	18.2829	17.3539
Dyract	12.6037	15.9695
Hytac	3.9668	13.4539
Z100	10.2836	5.9403
F2000	27.8206	7.6969
Ketac-Molar	8.1572	4.9009
Fuji IX	8.5593	4.8207

The strength values observed at the highest stress application rate were taken to represent the fast fracture condition. Two-parameter Weibull distributions were fitted to these data using the method of maximum likelihood. The estimated Weibull parameter values are shown for the dry and soaked conditions in Table III. η denotes the Weibull scale parameter with units of MPa, and β denoting the dimensionless Weibull shape parameter. The first question of interest is whether soaking has affected the Weibull shape parameter for any of the materials. McCool [14] has shown that the homogeneity of the shape parameter among sets of k Weibull samples may be based on the ratio of the largest to the smallest maximum likelihood shape-parameter estimate among the k groups. Tables of critical values of this ratio have been developed for $k = 2$ to 10 and sample sizes ranging from 5 to 30 [15]. The tables are developed for the case where the sample sizes for the k samples are equal. In comparing the soaked with the dry sample shape parameter values, $k = 2$ but unfortunately the sample sizes are unequal. From tables referenced above, it is found that for two samples both of size 20, the ratio of the largest to smallest shape parameter will not exceed 1.56 by chance in more than 10% of such comparisons. For two samples of size 10 for a 10% level significance test, the value is 1.87, and for two samples of size 15, the critical ratio is 1.65. It is thus conservative to use the critical value based on both samples being of size 20. By this standard and from the shape-parameter ratios listed in Table IV we may conclude that soaking has only affected the shape parameter for Hytac for which it has been reduced by a factor of two.

To assess the effect of soaking on fast-fracture strength we use a similar multiple comparison analysis to compare the median fast-fracture strengths $x_{0.50}$, before and after soaking. The test statistic is:

$$t = \bar{\beta} \ln \left(\frac{(x_{0.50})_{\max}}{(x_{0.50})_{\min}} \right) \tag{2}$$

where $\bar{\beta}$ is the average shape-parameter estimate among the k groups to be compared, and $(x_{0.50})_{\max}$ and $(x_{0.50})_{\min}$ are the largest and smallest values of the estimated medians among the k groups. The method applies when the shape parameter does not differ among the groups. The estimated median is computed in terms of the estimated scale and shape parameters as:

$$x_{0.50} = \eta \cdot (\ln 2)^{1/\beta} \tag{3}$$

The computed estimates of median strength are given in Table V. The computed values of the t statistic are shown in the last column of Table IV. The critical value of t for a

TABLE IV Fitted Weibull parameters for the highest stress rate (fast fracture).

Material	η (dry)	β (dry)	η (soaked)	β (soaked)	Sample size (soaked)	Sample size (dry)	$\beta_{\max}/\beta_{\min}$	t
Compoglass	155.0	10.32	114.2	7.19	15	20	1.43	3.25
Dyract	138.6	14.32	144.0	15.96	15	20	1.11	0.180
Hytac	144.3	13.30	98.24	6.77	15	20	1.96	4.57
Z100	172.8	5.13	156.9	4.37	15	20	1.17	0.558
F2000	62.34	6.95	112.1	7.16	15	20	1.03	3.65
Ketac-Molar	25.3	3.56	20.1	4.99	10	20	1.40	0.667
Fuji IX	24.15	4.25	22.36	3.41	10	20	1.25	0.426

10% significance level test for the equality of $k=2$ median values is 1.01 for two groups of size 10, 0.798, for two groups of size 15 and 0.675, and for two groups of size 20. For two groups of different sizes such as 15 and 20 one expects the critical value to be intermediate to 0.675 and 0.798. Based on this reasoning applied to the computed t values in Table III, we can say that soaking significantly decreased the strength of Compoglass and Hytac and significantly increased the strength of F2000. For the other materials, there was no significant effect of soaking on median strength. The test is approximate for Hytac since the shape parameters of the soaked and dry Hytac samples were shown above to be significantly different.

The same multiple comparison methodology may be used to compare the soaked and dry Weibull parameters among the seven materials ($k = 7$). From tables of critical values, the upper 10% point for the ratio of maximum to minimum shape-parameter estimates in seven samples of size 20 is 2.00. Any pair of materials tested in the dry condition for which the shape parameter ratio exceeds 2 may be declared different.

The following table gives the ratio of the larger to the smaller shape parameter among all pairs of materials as tested in the dry condition.

	Compoglass	Dyract	Hytac	Z100	F2000	Ketac-Molar	Fuji IX
Compoglass *	—	1.39	1.29	2.01	1.48	2.89	2.43
Dyract —	—	*	1.07	2.79	2.06	4.02	3.37
Hytac —	—	—	*	2.59	1.91	3.73	3.13
Z100 —	—	—	—	*	1.35	1.44	1.21
F2000 —	—	—	—	—	*	1.95	1.64
KetacMolar —	—	—	—	—	—	*	1.19
Fuji IX —	—	—	—	—	—	—	*

TABLE V Computed median strengths $x_{0.50}$ (MPa)

Material	Median strength (dry)	Median strength (soaked)
Compoglass	154.990	108.512
Dyract	138.610	140.763
Hytac	140.357	93.059
Z100	160.963	144.292
F2000	59.129	106.472
Ketac-Molar	22.794	18.669
Fuji IX	22.152	20.077

There is no difference among Compoglass, Dyract and Hytac or among Z100, F2000, Ketac-Molar and Fuji IX. Compoglass differs only from Ketac-Molar and Fuji IX.

For the soaked groups, the critical value based on a sample size of 15 is 2.19. The following table gives the ratio of the larger to the smaller shape parameter among all pairs of materials as tested in the soaked condition.

	Compoglass	Dyract	Hytac	Z100	F2000	Ketac-Molar	Fuji IX
Compoglass *	—	2.22	1.06	1.65	1.00	1.44	2.11
Dyract —	—	*	2.35	3.65	2.22	3.19	4.68
Hytac —	—	—	*	1.55	1.06	1.35	1.99
Z100 —	—	—	—	*	1.64	1.14	1.28
F2000 —	—	—	—	—	*	1.43	2.10
KetacMolar —	—	—	—	—	—	*	1.46
Fuji IX —	—	—	—	—	—	—	*

It is seen that in the soaked condition, Dyract is different from the other six materials which are virtually indistinguishable from each other. Although $7.19/3.41 = 2.10$ is close to significant, the fact that the 3.41 was computed from a sample of only 10 means that somewhat more variability should be expected.

Since high values of the Weibull shape parameter correspond to low variability in breaking strength, we may summarize by saying that in the dry condition the materials fell into a high variability group and a low variability group. In the soaked condition only Dyract exhibited low variability.

We now compare the median strength in the dry condition among the specimens. Since the shape parameters differ between the two groups of materials identified above, we conduct the comparisons separately

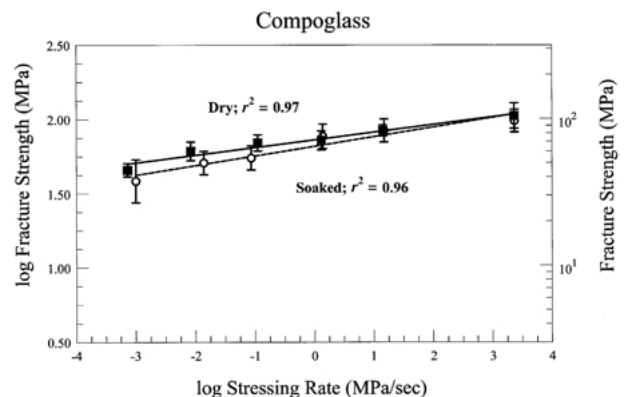


Figure 1 Dynamic fatigue plot for the light-cured composite Z100.

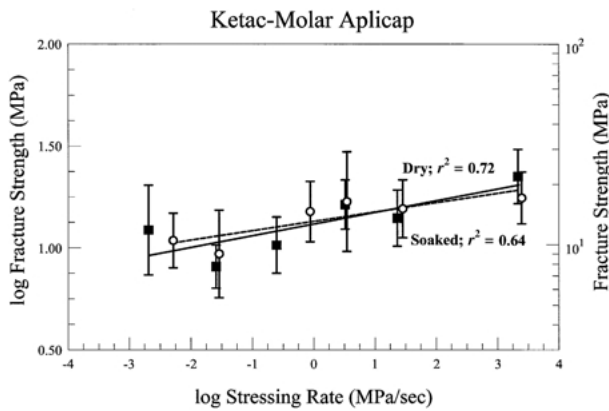


Figure 2 Dynamic fatigue plot for the highly viscous glass ionomer Fuji IX.

for the first three materials listed in Table IV and then for the remaining four materials. For the first three materials, the average shape-parameter estimate is 12.648. The critical value of the t statistic at the 10% significance level, based on samples of size 20, is 0.8365. The computed t statistic (Equation 2) using the largest and smallest value of $x_{0.50}$ among all pairs of three materials is given below:

	Compoglass	Dyract	Hytac
Compoglass	*	1.41	1.25
Dyract	—	*	0.163
Hytac	—	—	*

These values establish that in the dry condition Dyract and Hytac do not differ in strength from each other, but both have lower strength than Compoglass.

For the second set of four materials the average shape parameter value is 4.973 and the critical value of t for $k = 4$ and 10% significance is 0.9238. The computed t for all pairs within this set of four materials is tabled below.

	Z100	F2000	KetacMolar	Fuji IX
Z100	*	4.98	9.72	9.86
F2000	—	*	4.74	4.88
KetacMolar	—	—	*	0.142
Fuji IX	—	—	—	*

The results show that Fuji IX and Ketac-Molar do not differ, but all other comparisons are significant. Since we have already compared dry to soaked within each material we omit undertaking a comparison of the median strengths among the seven materials in the soaked condition.

Discussion

In visible-light-cured composite resins, the additional degree of cure after the initial 24 h period is insignificant, so that the principal cause of strength changes for the material tested here is water sorption. Soaking a composite in water results in both a rapid elution of unbound molecules and a much slower sorption of water

that is controlled predominantly by diffusion into the polymer matrix [16]. Most of the water molecules occupy free volume between polymer chains, as well as the microvoids created during polymerization [17]. The hydrophilicity of the polymer used also affects the degree and rate of water absorption observed in a composite.

Adsorbed water plasticizes the polymer matrix, and may hydrolyze the silane bond and attack the silica filler. These phenomena all result in a diminution of composite strength. After the network is saturated with water and stabilization occurs, there is no further reduction in mechanical properties. Further degradation, such as filler/matrix interfacial hydrolysis or polymer matrix crazing, may be absent or may not continue significantly once the composite has become saturated and remains wet [18]. The hybrid composite Z100 showed no significant difference in strength after soaking, and the slope of the dynamic fatigue plot (Fig. 1) was unaffected by soaking. Its high filler content and matrix composition limit the degree of water sorption and subsequent loss of strength in this composite.

The viscous glass ionomers studied here do not contain a resin component, so that changes in their properties are presumed to be due to water-mediated chemical reactions. Prior studies have shown that traditional glass-ionomer cements increase in strength after water exposure [19] as a result of the development of a silica network structure [20]. However, no significant effects of aging in water on flexural strength were found in this study, though again soaking lowered the slope of the dynamic fatigue plot (Figs 2 and 3).

The compomer materials have chemical compositions lying between that of traditional resin composites and glass ionomers. The complex chemical composition of the four compomers tested undoubtedly affects the degree of water sorption possible in each material, and the extent of the subsequent acid-base reaction. Compoglass contains BISGMA, UDMA, TEGDMA, and DCDMA. Dyract contains UDMA and TCB, an acid polymerizable monomer which has two methacrylate groups as well as two carboxyl groups [6, 21]. Hytac contains BISMA, and F2000 contains dimethacrylates [4, 5]. Water-uptake by the resin component is expected to weaken these materials. The amount of hydrophilic resin present in the final product will also affect the water absorption of the material. Bis-GMA type resins are very

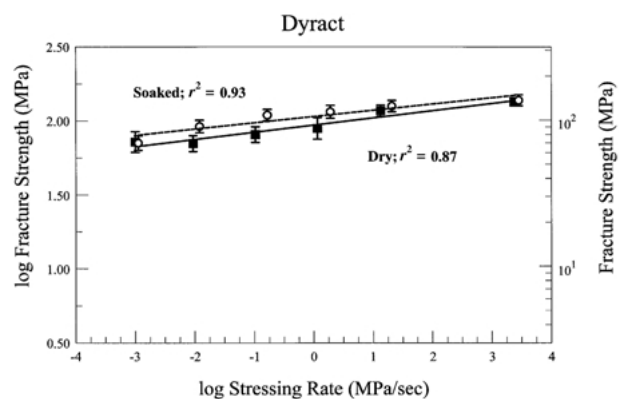


Figure 3 Dynamic fatigue plot for the highly viscous glass ionomer Ketac-Molar.

hydrophobic, while 2-hydroxy-ethyl-methacrylate (HEMA) is more hydrophilic.

Indeed, some resin-modified glass ionomer cements exhibit a decrease in mechanical properties following 24 h exposure to water [22]. However, for other resin-modified glass-ionomer cements exposed to water for three months, an increase in mechanical properties is observed due to a continued acid–base reaction [23]. The difference in response reported for similar materials is presumably due to differences in composition of the resin phase (with some materials having higher water sorption than others), differential degrees of glass particle dissolution, and method of mixing [24].

In terms of crack propagation, we found that soaking increased the rate of crack propagation for all materials. Following soaking, the three compomers Compoglass (Fig. 4), Hytac (Fig. 5) and Dyract (Fig. 6) were found to be similar to each other. Strength properties changed as a result of soaking, for several materials; the strength increased for F2000 (Fig. 7), and decreased for Compoglass and Hytac. Our findings for Dyract are similar to those of Nicholson and Alsarheed [25] who reported no change in the strength of Dyract and Compoglass following soaking in aqueous 0.9% NaCl solution, but do not reflect their findings concerning Compoglass. Gladys *et al.* [6] also found no increase in clamped fracture strength following soaking for Dyract. Kakaboura *et al.* [26] showed that Dyract undergoes a fast acid–base reaction at the outermost material surface. Testing Dyract and Compoglass, Eliades *et al.* [27] reported that polyacid-modified composite surfaces

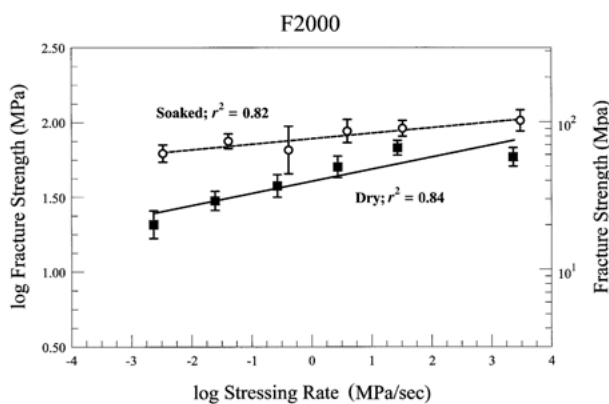


Figure 4 Dynamic fatigue plot for the light-cured compomer Compoglass.

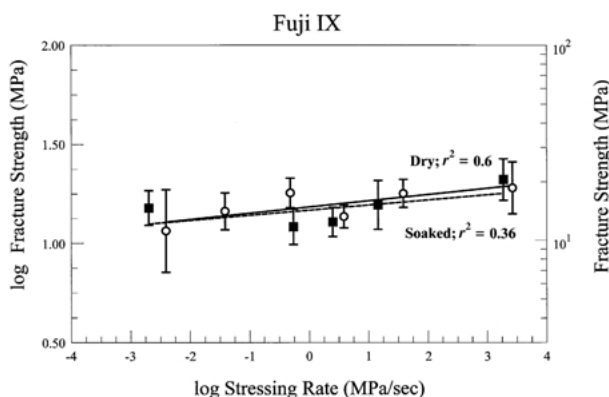


Figure 5 Dynamic fatigue plot for the light-cured compomer Hytac.

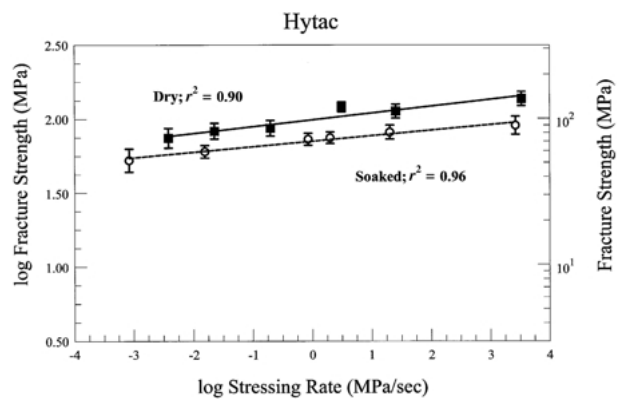


Figure 6 Dynamic fatigue plot for the light-cured compomer Dyract.

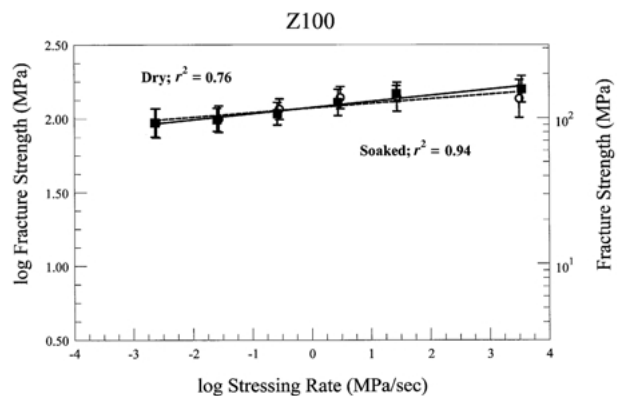


Figure 7 Dynamic fatigue plot for the light-cured compomer F2000.

stored in distilled water undergo a slow-rate, solid-state transformation to produce carboxylate salts and reach a saturation point approximately after four weeks. This carboxylate-rich topcoat apparently does not contribute significantly to strengthening of these materials.

Life prediction

Compomers are among the materials chosen as alternatives to dental amalgam. Although they are not recommended for placement in stress-bearing areas of permanent teeth, they are used on the occlusal surfaces of primary molars as well as in low stress-bearing areas of permanent teeth [28–30]. Cavity preparations for compomer restorations can be more conservative than amalgam preparations because of the ability of these materials to bond to tooth structure. Additionally, compomers have been shown to release fluoride, reducing recurrent decay on tooth structure adjacent to margins. If fatigue lifetimes approaching or surpassing amalgam can be demonstrated for compomers, then these materials could be preferred over amalgam for restoration of primary molars. These considerations motivated us to compute lifetimes for the compomer materials in this study. This approach also takes into account both the inherent strength of the materials as well as crack-propagation rates.

Following Ritter [11] it is assumed that under stress a crack of size “*a*” grows with time at a rate da/dt expressible as a power function of the stress intensity K_I :

$$da/dt = V = AK_I^N \quad (4)$$

where A is a constant, and N is the crack-growth or stress-corrosion exponent

Under a static tensile stress σ_a , Ritter [11] finds by integrating the crack-growth law that the time to failure t_f is:

$$t_f = B\sigma_i^{N-2}\sigma_a^{-N} \quad (5)$$

where B contains the terms A , the crack shape factor Y , and the material-dependent critical stress intensity factor K_{IC} . σ_i is the initial tensile strength of the specimen.

In applying this theory, we substitute the maximum calculated stress in a three-point bending specimen for σ_a and correspondingly base strength determinations (σ_i) on the measured value of the three point bending stress at fracture. The corresponding stresses at the cracks causing failure will be proportional in both cases.

When the applied stress is not static but increases at a rate

$$\dot{\sigma} = \frac{d\sigma}{dt} \quad (6)$$

it follows from Ritter's work that the average, or expected, value of the logarithm of the stress at fracture may be expressed as:

$$E[\ln(\sigma_f)] = \frac{1}{(N+1)} [\ln B + \ln(N+1) + (N-2)\mu] + \frac{1}{(N+1)} \ln \dot{\sigma} \quad (7)$$

where μ is the expected value of $\ln \sigma_f$ over the population of specimens. This is a linear function of the form

$$E(\ln \sigma_f) = \beta_0 + \beta_1 \ln(\dot{\sigma}) \quad (8)$$

where,

$$\beta_0 = \frac{1}{N+1} [\ln B + \ln(N+1) + (N-2)\mu] \quad (9)$$

and

$$\beta_1 = \frac{1}{N+1} \quad (10)$$

Regressing observed values of $\ln \sigma_f$ against the logarithm of the stress rate gives estimates of β_0 and β_1

We may then estimate N from Equation 10:

$$N = \frac{1}{\beta_1} - 1 \quad (11)$$

$\ln B$ may then be found from Equation 9:

$$\ln B = (N+1)\beta_1 - [\ln(N+1) + (N-2)\mu] \quad (12)$$

With $\ln B$ and N established in terms of the regression constants we return to Equation 5 and take logarithms and expected values to give:

$$E(\ln t_f) = \frac{\beta_0}{\beta_1} + \ln \beta_1 - \left(\frac{1}{\beta_1} - 1 \right) * \ln(\sigma_a) \quad (13)$$

The median age of replaced amalgam restorations in adults has been shown to be 60 months [31], and the five-year survival rate for amalgam restorations in primary molars was found to be 60% [32]. We therefore selected five years as a desirable lifetime for the materials studies here. For an expected life of five years we set

TABLE VI Life predictions of tested materials using analysis of the dynamic fatigue data

Material	Five-year stress (dry), MPa	Five-year stress (soaked), MPa
Compoglass	11.76	28.47
Dyract	35.21	50.05
Hytac	44.89	36.31
Z100	62.91	67.65
F2000	4.15	39.01
Ketac-Molar	4.22	4.77
Fuji 9	4.87	7.07

$t_f = 5 \text{ years} * 3.153E7 \text{ s/year}$ in Equation 13 and solve for the static stress σ_a . This gives:

$$\ln(\sigma_a) = \frac{\beta_1}{(1-\beta_1)} \left[\frac{\beta_0}{\beta_1} + \ln(\beta_1) - 18.876 \right] \quad (14)$$

Note that μ is not needed for this calculation. It is only required when it is necessary to calculate B . It is emphasized that these five-year stresses do not apply to the conditions met in dental restorations. They are computed only to afford a unified comparison among the materials used in this study. They reflect the combined effect of the inherent strength of the material and its subcritical crack growth rate. Results are shown in Table VI.

Summary

All compomer and resin composite materials tested exhibited subcritical crack growth as manifest by increasing strength with increasing stress application rate. Long-term soaking had a significant effect ($p > 0.05$) on the crack-propagation rate of Compoglass, Dyract and F2000. F2000 exhibited an increase in strength following long-term soaking while Hytac and Compoglass exhibited a decrease. Combining both crack-propagation rate and strength data in a lifetime prediction approach, we find that Z100, Dyract, and Hytac potentially offer the greatest longevity.

Acknowledgments

Thanks to the following companies for the generous supply of material: 3M, St. Paul, MN, USA; DeTrey/Dentsply, Konstanz, Germany; ESPE, Seefeld, Germany; GC Corporation, Tokyo, Japan; Ivoclar-Vivadent, Amherst, NY USA.

Supported by USPHS grant DE 09530.

References

1. A. WILSON and J. MCLEAN, in "Glass-Ionomer Cement" (Quintessence Pub. Co., Chicago, 1988).
2. K. ANUSAVICE, in "Phillips' Science of Dental Materials" 10th edn (W. B. Saunders Company, Philadelphia, 1996).
3. J. MCLEAN, J. NICHOLSON and A. WILSON, *Quint. Int.* **25** (1994) 587.
4. I. EL-KALLA and F. GARCIA-GODOY, *Oper. Dent.* **24** (1999) 2.
5. J.-M. MEYER, M. A. CATTANI-LORENTE and V. DUPUIS, *Biomaterials* **19** (1998) 529.

6. S. GLADYS, M. BRAEM, P. LAMBRECHTS and G. VANHERLE, *J. Dent. Res.* **76** (1997) 883.
7. J. MCLEAN, in "Proceedings of the 2nd International Symposium on Glass Ionomers, Philadelphia, June 1994", edited by P. R. Hunt (International Symposia in Dentistry PC, Philadelphia, 1994) p. 61.
8. S. MITRA, in "Proceedings of the 2nd International Symposium on Glass Ionomers, June, 1994", edited by P. R. Hunt (International Symposia in Dentistry PC, Philadelphia, 1994) p. 13.
9. A. PEUTZFELDT, F. GARCIA-GODOY and E. ASMUSSEN, *Am. J. Dent.* **10** (1997) 15.
10. A. G. EVANS, *Int. J. of Fract.* **10** (1974) 251.
11. J. E. RITTER, in "Fracture Mechanics of Ceramics: Crack Growth and Microstructure", edited by R. C. Bradt, D. P. Hasselman and F. F. Lange (Plenum Press, New York, 1978) p. 667.
12. C. W. FAIRHURST, P. E. LOCKWOOD, R. D. RINGLE and S. W. TWIGGS, *Dent. Mat.* **9** (1993) 269.
13. J. E. RITTER, J. M. STEVENS and K. JAKUS, *J. Mater. Sci.* **14** (1979) 2446.
14. J. I. MCCOOL, *IEEE Trans. on Rel.* **R24** (1975) 186.
15. J. I. MCCOOL, *U.S.A.F. Res. Lab. Rep. ARLTR 74-018* (1974).
16. M. FREUND and E. MUNKSGAARD, *Scand. J. Dent.* **98** (1990) 351.
17. K. SODERHOLM, *J. Biomed. Mater. Res.* **18** (1984) 271.
18. J. FERRACANE, H. BERGE and J. CONDON. *ibid.* **42** (1998) 465.
19. E. A. WASSON and J. W. NICHOLSON, *J. Dent. Res.* **72** (1993) 481.
20. S. MATSUYA, T. MAEDA and M. OHTA, *ibid.* **73** (1994) Abstr. 2467.
21. N. MARTIN and N. JEDYNAKIEWICZ, *Biomaterials* **19** (1998) 77.
22. M. A. CATTANI-LORENTE, V. DUPUIS, F. MOYA, J. PAYAN and J.-M. MEYER, *Dent. Mat.* **15** (1999) 21.
23. Y. MOMOI, K. HIOSAKI, A. KOHNO and J. F. MCCABE, *Dent. Mat. J.* **14** (1995) 109.
24. A. AKASHI, Y. MATSUYA, M. UNEMORI and A. AKAMINE, *Biomaterials* **20** (1999) 1573.
25. J. W. NICHOLSON and M. ALSARHEED, *J. Oral Rehab.* **25** (1998) 616.
26. A. KAKABOURA, G. ELIADES and G. PALAGHIAS, *J. Dent. Res.* **75** (1996) 1218.
27. G. ELIADES, A. KAKABOURA and G. PALAGHIAS, *Dent. Mater.* **14** (1998) 57.
28. K. M. HSE and S. H. WEI, *J. Am. Dent. Ass.* **128** (1997) 1088.
29. L. PAPAGIANNOULIS, A. KAKABOURA, F. PANTALEON and K. KAVVADIA, *Pediatr. Dent.* **21** (1999) 231.
30. L. A. MARKS, K. L. WEERHEIJM, W. E. VAN AMERONGEN, H. J. GROEN and L. C. MARTENS, *Caries Res.* **33** (1999) 387.
31. K. H. FRIEDL, K. A. HILLER and G. SCHMALZ, *Oper. Dent.* **19** (1994) 228.
32. A. G. PAPATHANASIOU, M. E. CURZON and C. G. FAIRPO, *Ped. Dent.* **16** (1994) 282.

*Received 26 July 2000
and accepted 25 September 2001*