

Effect Of Cyclic Loading
On The Mechanical Properties
Of Filling Resins

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Submitted in partial fulfillment of the
requirements for a certificate of
pedodontics at the University of Oregon
Health Sciences Center, School of Dentistry

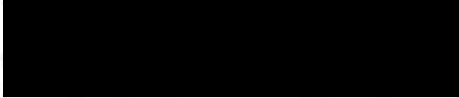
June, 1981

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ABSTRACT

The hypothesis of this study is that if a dental restorative resin is subjected to cyclic loading as experienced during mastication, the bond between the filler and the resin matrix could be broken (due to differing moduli of elasticity) and result in accelerated clinical wear. To test this hypothesis two microfill, two conventional, one combination microfill/conventional and two unfilled resins were subjected to cyclic loading and then tested in a transverse strength mode. Uncycled specimens served as a control. If the bond between filler particles and resin matrix is broken, the result will be manifest in a lower modulus of elasticity. Results of modulus of elasticity determination showed a range from 308,300 psi to 1,954,200 psi for the resins tested. No significant difference was found in the modulus of elasticity of the cycled specimens when compared to the uncycled control specimens. Transverse strength was also determined and a small but statistically significant higher strength was found for the pre-cycled specimens when compared to the uncycled specimens. For the conditions of these tests, cyclic loading of filling resins does not appear to affect the bond between filler particles and resin matrix.

ACKNOWLEDGMENTS

The author expresses his sincere appreciation and gratitude to Dr. D.B. Mahler, DR. J.C. Mitchem, and Mr. J.D. Adey for their guidance in this study.

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INTRODUCTION:

The single most important clinical mode of failure of composite resin dental restorations is loss of their anatomic form. Clinical studies of Philips, et al,¹ showed that class II composites tended to wear and lose their anatomic form faster than the adjacent enamel or amalgam restorations. No laboratory tests have been able to predict clinical wear due to the inability to determine the exact mechanism of the wearing of dental resins. Although, Eames et al,² noted the appearance of a textured surface, which they described as a "gross surface deterioration," for the composite dental restorations, they did not identify a mechanism that brought about this deterioration.

Many in-vitro studies have been performed on resins using wear or abrasion tests,^{3,4,5,6} but none have been done regarding the role played by mechanical factors such as the differences in elastic moduli between filler particles and the resin matrix. Although most of the composite systems use a BIS-GMA matrix, the physical and mechanical properties of each composite varies according to the composition, number, size and shape of the filler particles as well as the strength of the filler-matrix bond.⁷ Because of large differences in moduli, the resin matrix will deform more than the filler particles when it is under a stress.⁸ This could be part of the mechanism of composite resin wear in the mouth. After a period of repeated loading and unloading of occlusal stress, the bond between the filler and the matrix could break which could result in accelerated wear of the composites.

This hypothesis would also help to explain the clinical success of the microfilled resins over conventional composites in regards to wear. Jorgensen, et al,⁶ observed that an experimental microfilled resin showed significantly less wear than Adaptic. Jorgensen hypothesized that since the distance

between particles in the microfill does not exceed 0.1 μm , food abrasion of the resin matrix will be prevented. In the case of the microfilled resins, the extremely small size of the filler particles could resist debonding.

To test this hypothesis of debonding, transverse strength specimens will be subjected to the cyclic loading followed by a static transverse strength test. Modulus of elasticity in bending will be compared for both uncycled and cycled specimens. If debonding occurs, the modulus of elasticity should be reduced significantly in the cycled specimens.

MATERIALS AND METHODS:

Two microfill, two conventional, one combination microfill/conventional, and two unfilled resins were used in this study (fig. 1-4). The materials, their manufacturer and the material classification are listed in Table I.

Ten specimens of each material were prepared to a size of 1 mm x 4 mm x 12 mm. The specimens of each material were prepared from the same manufacturer's kit and according to the manufacturer's instructions. Each specimen was made in an individual glass mold. The mold consisted of glass microscope slides that were bonded together with cyano-acrylate cement, using a brass rectangular spacer as a guide (fig. 5). Inlay wax was used to block the ends of the glass molds. A Centrix syringe was used to place each material into the glass mold, except the pure Adaptic resin which was flowed into the mold. The material was covered by a cellulose matrix strip and then by a glass microscope slide. Hand pressure was applied on the microscope slide and glass mold, and excess material was extruded from the mold by use of a C-clamp. The mold was then placed in a 37° C water bath (fig. 6). The specimens were removed from the glass molds in accordance with the manufacturer's setting times. The specimens were trimmed to the approximate specimen size of 12 mm x 4 mm x 1 mm using a razor blade and 400 grit paper. The specimens were then replaced in the 37° water bath and stored for a period of seven days^{9,13,14} before being tested.

Immediately prior to testing, each specimen was wiped dry and allowed to cool to room temperature. The specimen was then placed in a transverse loading device under cyclic loading. The load was cycled between 5025 psi tension to 5025 psi compression for 10⁵ cycles (fig. 7). If one were to assume an average chewing time of 30 minutes per day, then 10⁵ cycles would correspond to a service in the mouth for 1½ months.¹⁵ Following the cyclic loading, each specimen was measured at the center point (as indicated by an

inked line) using a micrometer, and the width and thickness of each specimen was recorded. Each specimen was then placed in an Instron testing device (fig. 8) and the load and deflection of each sample were recorded on a load-deflection plot up to and including the breaking point.

Transverse Strength Determination:

Transverse strength for each specimen was determined from the following formula:

$$S_t = \frac{3L}{2bh^2} \times P$$

S_t = Transverse strength of sample

L = Sample length between supports

b = Sample width

h = Sample thickness

P = Sample fracture load

The specimens were subjected to a crosshead loading speed of 0.02 in/minute. The length, width and thickness of each specimen having been previously recorded, the fracture load was obtained from the Instron load-deflection plot.

Modulus of Elasticity Determination:

The modulus of elasticity for each specimen was determined from the following formula:

$$E = \frac{FL^3}{4bh^3d}$$

E = Modulus of elasticity

F = Force of loading

L = Sample length between supports

b = Sample width

h = Sample thickness

d = Sample deflection

A straight line was drawn coincident with the linear portion of each load-deflection curve. The deflection per unit load was determined as the slope of this line. To provide a more accurate measurement, the line was extended over a five pound range.

Since the deflection on the Instron recorder measures loading head travel, the indentation occurring at the specimen-supporting rods and the 1/16" loading-ball are included in the total deflection and this needs to be corrected to get true specimen deflection. The deflection correction was made as follows. A load-deflection curve was run with a steel bar (12 x 4 x 1 $\frac{1}{2}$ mm) on top of the specimen, at the bottom of the specimen and by itself. In Table II, the calculation for determining the true deflection of the resin specimen is outlined. Three of the ten specimens made from each material were tested using this procedure, and the corrections averaged. The average correction was then applied to all ten specimens.

The transverse strength and the modulus of elasticity was determined for each material in both the stressed and unstressed state. The transverse strength is important when considering the edge strength of dental resins, especially when feather-edged margins are used. The modulus of elasticity will be used to determine if debonding has occurred in the stressed samples. Use of a bonded filler in composite resins will increase the modulus of elasticity,^{10,11} while the modulus of a resin with an unbonded filler does not differ from the modulus of elasticity of pure resin.¹² Therefore, if after the filled resin samples are cycled and we obtain the modulus of elasticity of the pure resin, then it appears that debonding has occurred, and our hypothesis appears to be true. If these same tests were performed on the microfilled resins, and there was no evidence of debonding (a decrease in the modulus), then one could assume that the microfills give improved clinical wear.

Preliminary Testing:

Preliminary testing was done on twenty specimens of Adaptic composite resin to determine the sample size, specimen preparation procedures and to establish the number of cycles each specimen could safely undergo without fracturing. Results of these preliminary tests indicated that 10^5 cycles was sufficient stress to cause a significant reduction in the modulus of elasticity of the stressed specimens as compared to the unstressed specimens. A significant difference was not found whether a specimen was cycled 10^5 or 5×10^5 times. All specimens fractured at lower than 10^6 cycles. As a result of this preliminary testing, a sample size of ten was selected for each group, specimen preparation procedures were refined, and 10^5 cycles was selected as the initial cyclic loading condition.

RESULTS AND DISCUSSION:

TRANSVERSE STRENGTH:

The values for the transverse strength of both the unstressed and stressed specimens of each material are listed in Table III and represented in bar graph form in Table V. Analysis of variance was applied to the data and the results are listed in Table IV. The F values indicate a significant difference in materials and the stressed versus unstressed condition. Scheffe's contrast test was applied to the means to determine statistically significant differences. Horizontal lines between bars in Table V indicated no differences using Scheffe'.

The calculated value for each unstressed material is comparable to those reported in earlier studies.^{9,16,17} A study done by Valiaho and Forsten²⁵ obtained a value of 13,500 psi for Adaptic and a value of 13,743 psi was obtained for Adaptic in this current study. Miradapt (combination conventional/microfill composite) exhibited the highest mean transverse strength, but was not significantly greater than Adaptic. The mean transverse strength of Adaptic was significantly higher than the other conventional composite (Profile) used in this study. There was no significant difference in the mean transverse strength of the unfilled resins or the microfilled composites. The mean transverse strength of Miradapt and Adaptic was found to be significantly higher than any of the other materials studied. Bowen¹⁸ found that the use of submicron filler particles in a bimodal system required six to eight volume per cent less resin. This resulting composite had a higher modulus of elasticity and a lower coefficient of thermal expansion than a comparable conventional composite with normal filler particles. It appears that the transverse strength is also increased when a bimodal system is employed, but a qualitative analysis of the materials utilized in this study would be needed before acceptance of

this statement. The mean transverse strength of the Adaptic unfilled resin was the third highest and this illustrates the importance of the tensile element in transverse strength. Resistance of a material to tensile stress depends on the bond within the resin matrix. There appears to be an increase in the transverse strength of the stressed group as compared to the unstressed group for each material and this phenomenon will be discussed later in this paper.

MODULUS OF ELASTICITY:

The values for the modulus of elasticity of both the unstressed and stressed specimens of each material are listed in Table VI and represented in bar graph form in Table VIII. Analysis of variance was applied to the data and the results are listed in Table VII. The F values indicate a significant difference in materials. Scheffe's contrast test was applied to the means to determine statistically significant differences. A horizontal line between bars in Table VIII indicated no differences using Scheffe'.

The modulus of elasticity of the conventional composites and the combination conventional/microfill resin (Miradapt) were significantly higher than the moduli of microfilled or unfilled resins, although Miradapt was not significantly higher than the modulus of Adaptic. The microfilled resins showed a significantly higher modulus than the unfilled resins, but the modulus of the microfilled resins was less than fifty percent of the modulus of the conventional composite with the lowest modulus (Profile). The values obtained for the modulus of elasticity in this project are comparable to those obtained in previous studies.^{13,16,19,20,21} Craig¹⁹ found that the modulus of elasticity of human enamel and dentin is higher than that of dental composite resins. Nakayama²⁰ found that the modulus of elasticity of a dental restorative material should be as near to that of the adjacent

tooth material as possible, as this helps to maintain marginal integrity when the tooth is under stress. Modulus values of 1.8973×10^6 psi for Adaptic and 0.3083×10^6 psi for Sevriton were obtained from this current study, and these are very near those obtained by O'Brien and Ryge,¹⁷ of 2.41×10^6 psi for Adaptic and 0.340 for Sevriton. Although the modulus values of all materials were significantly lower than that of human enamel,¹⁷ this was a qualitative study and does not attempt to deal with the clinical significance of this.

De'rand and Ehrnford²⁴ found in a long-term evaluation of dental resin abrasion that conventional composite resins illustrated decreased abrasion resistance while no such effect was evident in the microfilled resin. This decreased abrasion resistance was thought to be due to a reduction of bond strength between the fillers and resin matrix. The results of this current study do not support this theory. There appears to be no significant difference in the modulus of elasticity of the unstressed specimens when compared with the stressed specimens of the same material. There does appear to be a significant increase in the transverse strengths of the unstressed specimens when compared to the stressed specimens. One explanation for this phenomenon is that the stressed specimens were inadvertently being selectively chosen. Each stressed specimen had to successfully complete 10^5 cycles in a transverse loading device under cyclic loading before it would be tested for deflection and fracture load in an Instron testing device. If the specimen fractured during the cycling and did not complete the 10^5 cycles, the specimen was replaced with a new specimen. Therefore, only the stressed specimens had to go through a selection process, and the weaker specimens (due to porosities or incomplete polymerization) were not eliminated from the unstressed group, as they had been from the stressed group. The importance of this attrition and its possible effect on the study results can be comprehended

when one realizes that fifty per cent of the stressed specimens for some materials (Sevriton and Silar) had to be replaced. Draughn²² found in his study that the response of restorative materials to cyclic stress can be important to understanding and enhancing their clinical wear. The definition of fatigue life is the number of cycles a material can resist without failing. Failure will occur within a few cycles at high stress, and the number of cycles a material can withstand increases as the maximum stress per cycle decreases. The fatigue or endurance limit is the stress that a material can be subjected to indefinitely without fracture. The values of fatigue life and limit depend not only on the material, but also on the frequency of the cyclic loading, the testing environment, and on the nature of the applied stress.

A breakdown of the bond at the interface of the filler particle and the resin matrix results in failure of the restorative material.¹⁶ The project hypothesis is if a dental restorative resin is subjected to cyclic loading, the bond between the filler and resin matrix could be broken (due to differing moduli of elasticity) and result in accelerated clinical wear. Preliminary testing demonstrated that stress resulting from 10^5 cycles was sufficient to cause a significant reduction in the modulus of elasticity of the stressed specimens as compared to the unstressed specimens. Results of the final testing revealed that the transverse strengths of the stressed specimens were actually increasing in value, and there was no significant reduction in the modulus of elasticity in the stressed specimens, contrary to the results of the preliminary tests.

Two factors that should also be considered when evaluating the failure of the hypothesis in this study are water absorption and adequate cycling. Whiting and Jacobsen¹¹ found that conditioning specimens in water reduced the moduli of elasticity of the samples. There are several mechanisms for water absorption by dental resins, including diffusion-controlled absorption by the resin,

porosities, and capillary action in voids at the filler-resin matrix interface. The water absorption of a dental composite resin is influenced by its resin content, as it is the resin which absorbs the water. Water absorption by dental composite resins can result in dimensional changes in restorations and alter marginal integrity.¹³ Wilson, et al,²³ found that conventional composites soften on immersion, and that Isopost (Microfill) showed a long term hardening effect due to water absorption after 7 days. It should be pointed out though, that microhardness tests of Isopost at 7 days showed it to be fifty per cent lower than conventional composites. Whiting and Jacobsen¹¹ also found that as storage time is increased in a water bath, composite dental resins absorbed greater amounts of water and their moduli of elasticity decreases proportionately. Future work on this subject may wish to extend the seven day storage time of the specimens in a water bath to twenty eight days prior to testing, in order to achieve a more complete water absorption by the specimens. As regards the cyclic loading, more investigation should be done concerning whether the test apparatus and conditions are adequate. Kollmannsperger¹⁵ found that the deformity of the sample increased as the loading due to chewing increased and that the modulus of elasticity decrease ranged from eight to twelve per cent. Future work should investigate utilizing a lower load of compression and tension during specimen cycling in order to increase the number of cycles a specimen would be subjected to and allow a more uniform cycling.

Summary and Conclusions:

Two microfill, two conventional, one combination microfill/conventional and two unfilled resins were subjected to cyclic loading corresponding to masticatory movement in order to evaluate masticatory force-dependent characteristics. The hypothesis of this study is that if a dental restorative resin is subjected to cyclic loading, the bond between the filler and the

resin matrix could be broken (due to differing moduli of elasticity) and result in accelerated clinical wear. Results of static transverse strength tests and modulus of elasticity in bending showed the transverse strength of the materials tested to range from 8,685 psi to 14,598 psi, and the modulus of elasticity to range from 308,300 psi to 1,954,200 psi. Evaluation of the results revealed there to be no significant difference in the modulus of elasticity of the unstressed specimens when compared to the stressed specimens, and a significant increase in the transverse strengths of the stressed specimens when compared to the unstressed specimens. Future work should consider modification of the cycling apparatus to extend the cycling period and also allow a more uniform cycling, as well as increasing the water storage time.



Figure 1 Conventional Resins

ADAPTIC

PROFILE



Figure 2 Microfill/Conventional Resin

MIRADAPT



Figure 3 Microfill Resins

SILAR
ISOPAST



Figure 4 Unfilled Resins

SEVRITON ADAPTIC
RESIN

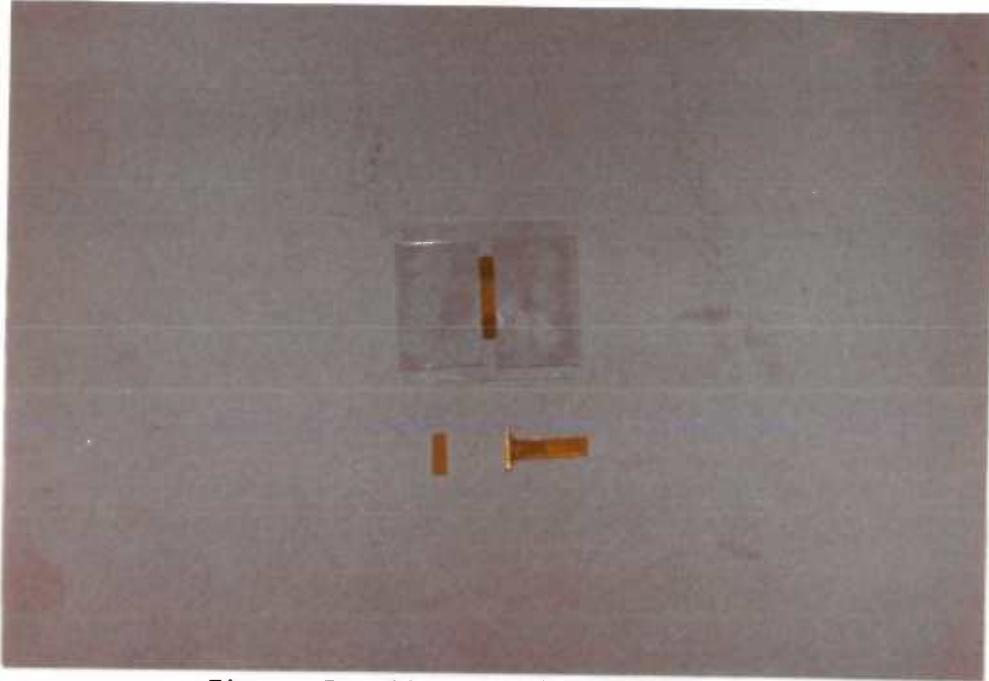


Figure 5 Glass Mold and Brass Spacer



Figure 6 Water Bath (37° C)

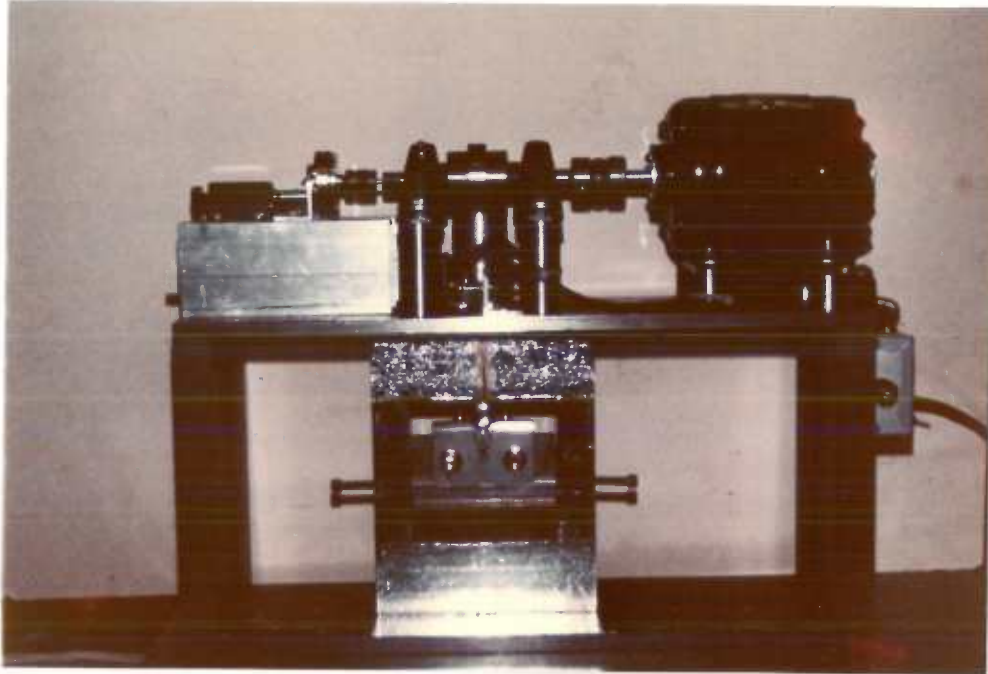


Figure 7 Transverse Loading Device

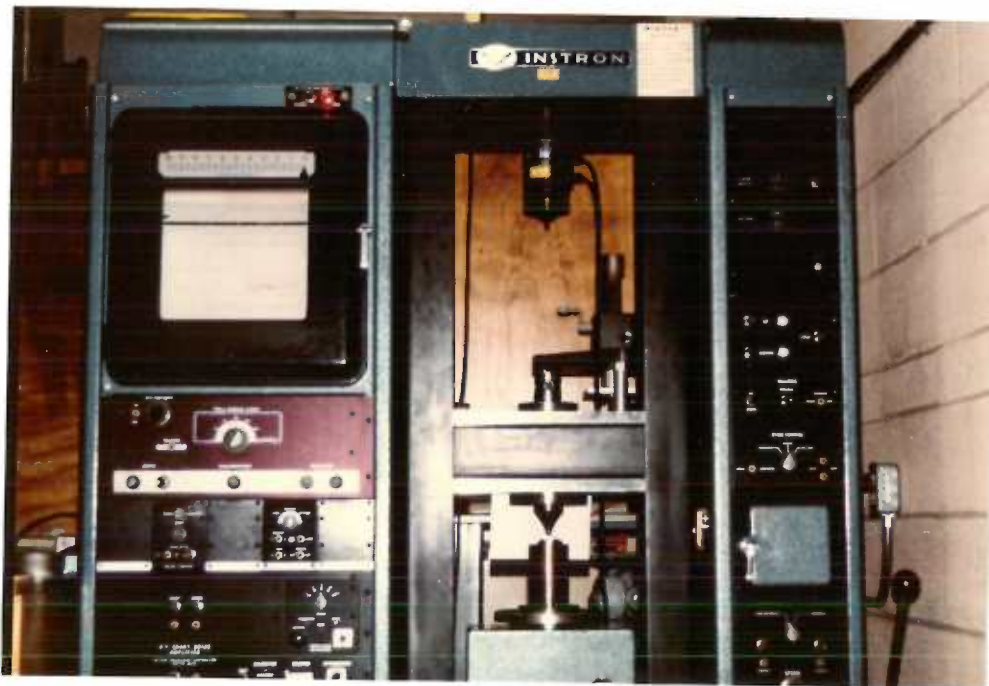
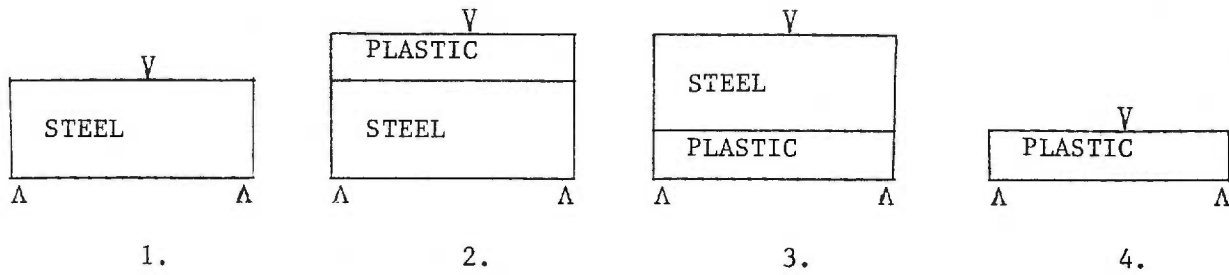


Figure 8 Instron Testing Device

Table 1

Code	Brand Name	Type	Manufacturer
AD	Adaptic	Conventional	Johnson & Johnson
PF	Profile	Conventional	S.S. White/Penwalt
MR	Miradapt	Microfill/ Conventional	Johnson & Johnson
IP	Isopast	Microfill	Vivadent (U.S.A.), Inc.
SL	Silar	Microfill	3M/Dental Products
SV	Sevriton	Unfilled Resin	Amalgamated Dental Products
AR	Adaptic Resin	Unfilled Resin	Johnson & Johnson

Table II



d_s = steel deflection/pound

d_p = plastic deflection

d_b = ball indentation

d_r = rod indentation

d_{ps} = plastic and steel deflection

$$d_1 = d_s \quad d_2 = d_b + d_{ps} \quad d_3 = d_{ps} + d_r \quad d_4 = d_b + d_r + d_p$$

CALCULATION OF TRUE DEFLECTION

$$d_p = d_4 - (d_b + d_r)$$

$$d_2 + d_3 = 2d_{ps} + d_b + d_r$$

$$d_p = d_4 - (d_2 + d_3 - 2d_{ps})$$

$$\text{Assume } d_{ps} = d_s = d_1$$

$$d_p = d_4 - (d_2 + d_3 - 2d_1)$$

THEREFORE:

$$d_p = d_4 + 2d_1 - d_2 - d_3$$

Table III

Code	Unstressed Transverse Strength (PSI)	Stressed Transverse Strength (PSI)
SL	6,947	10,422
IP	9,551	10,797
SV	10,577	11,046
PF	10,007	11,682
AR	10,667	13,641
AD	13,743	14,955
MR	13,072	16,126

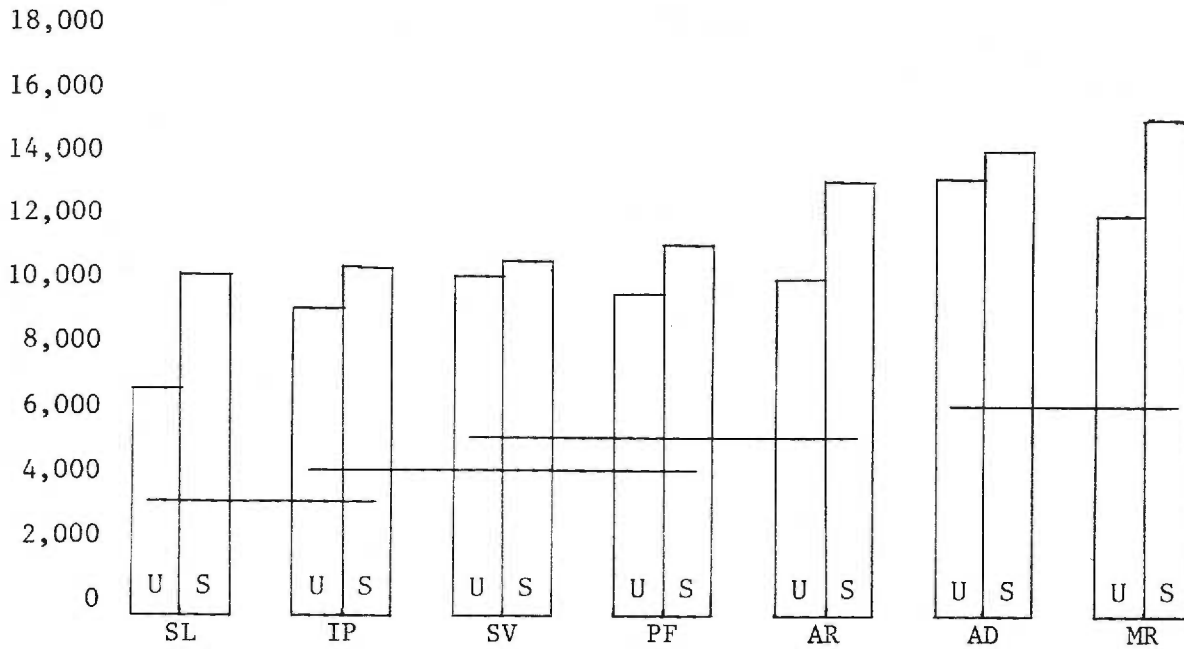
Table IV

ANOVA Transverse Strengths

	SS ($\times 10^6$)	DF	MS ($\times 10^6$)	F
Between Groups				
1. Materials	571.118	6	95.186	30.668*
2. Stressed vs. Unstressed	142.118	1	142.118	45.790*
3. Materials/ Stressed vs. Unstressed	39.369	6	6.561	2.114
Within Groups	391.064	126	3.103	1.000

* Significant F values

Table V
Scheffe's Contrast
Transverse Strength



Scheffe's Contrast 1,977

Bars Denote No Significant Difference ($\alpha=0.05$)

U= Unstressed Specimens

S= Stressed Specimens

Table VI

Code	Unstressed Modulus of Elasticity ($\times 10^6$ PSI)	Stressed Modulus of Elasticity ($\times 10^6$ PSI)
SV	0.3209	0.2957
AR	0.4134	0.4428
IP	0.4260	0.6008
SL	0.7763	0.8012
PF	1.5886	1.8108
AD	1.8633	1.9314
MR	2.1191	1.7893

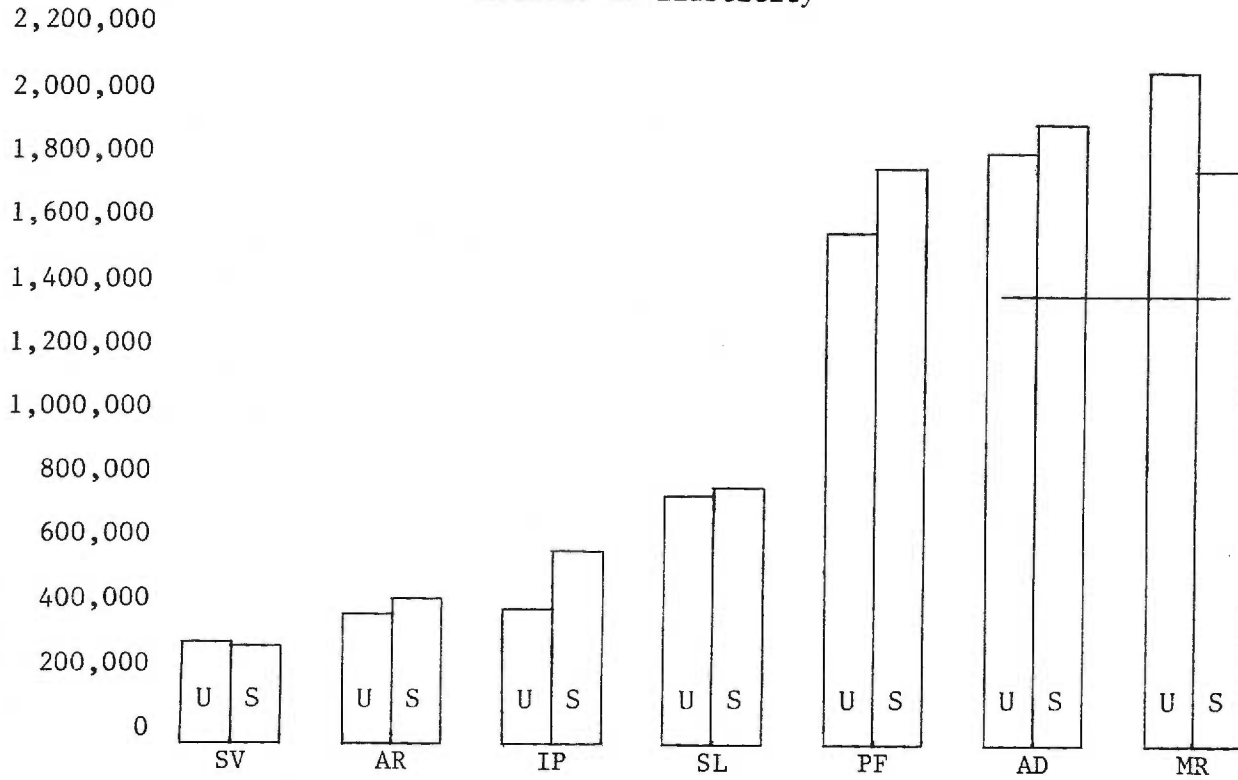
Table VII

ANOVA Moduli of Elasticity

	SS ($\times 10^6$)	DF	MS ($\times 10^6$)	F
Between Groups				
1. Materials	64.850	6	10.808	2521.118*
2. Stressed vs. Unstressed	0.019	1	0.019	4.501
3. Materials/ Stressed vs. Unstressed	0.957	6	0.159	37.239*
Within Groups	0.540	126	0.004	1.000

* Significant F values.

Table VIII

Scheffe's Contrast
Modulus of Elasticity

Scheffe's Contrast 73,496

Bar Denotes No Significant Difference(=0.05)

U= Unstressed Specimens

S= Stressed Specimens

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