

13:46:12

OCA PAD AMENDMENT - PROJECT HEADER INFORMATION

02/26/93

Active

Project #: E-18-534
Center # : 10/11-6-05044-5A0

Cost share #:
Center shr #:

Rev #: 5
OCA file #:
Work type : INST
Document : GRANT
Contract entity: GIT

Contract#: 5 T32 DE07053-15
Prime #:

Mod #: ADMIN 2/23/93

Subprojects ? : N
Main project #:

CFDA: 93.121
PE #: N/A

Project unit:
Project director(s):
HOCHMAN R F

MSE
MSE

Unit code: 02.010.112
(404)894-2879

Sponsor/division names: DHHS/PHS/NIH
Sponsor/division codes: 108

/ NATL INSTITUTES OF HEALTH
/ 001

Award period: 910701 to 930630 (performance) 930930 (reports)

Sponsor amount	New this change	Total to date
Contract value	0.00	97,054.00
Funded	0.00	97,054.00
Cost sharing amount		0.00

Does subcontracting plan apply ? : N

Title: POSTDOCTORAL-PREDOCTORAL TRAINING IN DENTAL MATERIALS SCIENCE (NIDR TRAINING)

PROJECT ADMINISTRATION DATA

OCA contact: Kathleen R. Ehlinger 894-4820

Sponsor technical contact

Sponsor issuing office

THOMAS M. VALEGA, PH.D.
(301)496-6324

SUSAN KAITLIN
(301)496-7437

SPECIAL ASST FOR MANPOWER DEVELOPMNT
NATL INST OF DENTAL RESEARCH
9000 ROCKVILLE PIKE
BETHESDA, MD 20892

GRANTS MANAGEMENT OFFICER
NATL INSTITUTE OF DENTAL RESEARCH
9000 ROCKVILLE PIKE
BETHESDA, MD 20892

Security class (U,C,S,TS) : U
Defense priority rating : N/A
Equipment title vests with: Sponsor

ONR resident rep. is ACO (Y/N): N
NIH supplemental sheet
GIT X

Administrative comments -
ISSUED TO REVISE DELIVERABLE SCHEDULE.

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

NOTICE OF PROJECT CLOSEOUT

Closeout Notice Date 07/17/95

Project No. E-18-534_____

Center No. 10/11-6-05044-5A0_

Project Director HOCHMAN R F_____

School/Lab MSE_____

Sponsor DHHS/PHS/NIH/NATL INSTITUTES OF HEALTH_____

Contract/Grant No. 5 T32 DE07053-15_____ Contract Entity GIT_

Prime Contract No. _____

Title POSTDOCTORAL-PREDOCTORAL TRAINING IN DENTAL MATERIALS SCIENCE (NIDR TRAIN

Effective Completion Date 930630 (Performance) 930930 (Reports)

Closeout Actions Required:	Y/N	Date Submitted
Final Invoice or Copy of Final Invoice	Y	940802
Final Report of Inventions and/or Subcontracts	Y	_____
Government Property Inventory & Related Certificate	N	_____
Classified Material Certificate	N	_____
Release and Assignment	N	_____
Other _____	N	_____

Comments _____

***NOTE** USE DHHS FORM FOR PATENT _____

Subproject Under Main Project No. _____

Continues Project No. E-18-520_____

Distribution Required:

Project Director	Y
Administrative Network Representative	Y
GTRI Accounting/Grants and Contracts	Y
Procurement/Supply Services	Y
Research Property Management	Y
Research Security Services	N
Reports Coordinator (OCA)	Y
GTRC	Y
Project File	Y
Other _____	N
_____	N

NOTE: Final Patent Questionnaire sent to PDPI.

June 25, 1993

Dr. Thomas M. Valega
Special Asst. for Manpower
Development & Training
Extramural Program
National Institute of Dental Research
Westwood Building, Room 510
Bethesda, Maryland 20892

Dear Dr. Valega:

Attached are some of the written accomplishments of the last few years by trainees on my training grant DE07053-15. Of major importance are the extensive contributions of Dr. Brian Love. Dr. Love has been offered a potential academic position to start this fall or winter.

Since there is approximately \$9,500 in residual funds from my last year's budget available, I am asking that a no cost extension be granted to support Dr. Love for three months to complete all his work before leaving and that the contract be terminated as of 12/31/93. This no cost extension will also allow me time to do a final summary report of the more than 30 years of training supported by the NIDR training grants. By last count these programs have produced 10 people presently in academic positions.

A budget for this carry-over program is attached. I am also copying this letter and budget without attachments to Theresa Ringler in Grants Management for her information.

Thank you and best regards.

Very cordially yours,


Robert F. Hochman

RFH:ew

Attachments

cc: Ms. Theresa Ringler
(w/o attachments)

PROPOSED CARRY-OVER BUDGET FOR
NO COST EXTENSION OF DE07053-15

For 6 months until 12/31/93

Postdoctoral		
Dr. Brian Love		
3 months at \$2,137/mo.	-	\$ 6,400
Supplemental Support		
1/2 Postdoctoral	-	1,250
Trainee Travel (1 man trip)	-	1,000
		<hr/>
Total Direct Costs	-	\$8,650
Overhead (8% of \$8,900)	-	692
		<hr/>
Total Requested Carry-over to 12/31/93	-	\$ 9,342*

*A balance of approximately \$9,500 will be available as of 6/30/93 on the present contract.

Kathryn Ehlinger
Contracting Officer
Contract Administration

~~Robert F. Hochman, PhD~~
~~Project Director, DE07053~~

Georgia Institute of Technology

A UNIT OF THE UNIVERSITY SYSTEM OF GEORGIA
SCHOOL OF MATERIALS SCIENCE AND ENGINEERING
ATLANTA, GEORGIA 30332-0245
USA

TO:

**Dr. Thomas M. Valega
Special Asst. for Manpower
Development & Training
Extramural Program
National Institute of Dental Research
Westwood Building, Room 510
Bethesda, Maryland 20892**

Sponsor: National Institute for Dental Research

Georgia Institute of Technology Training Grant

Area: Orthodontic Attachment Studies Covering the period from August 1988 to Present:

Support for:

Melisa Rathburn, D.D.S. (September 1988 to June 1990)

Mark Sanchez, D.D.S. (July 1990 to June 1991)

Brian Love, Ph.D. (January 1991 to present)

Kent Starling, D.D.S. (October 1991 to June 1992)

Scope of Effort: The NIDR support for this period was used to focus on the removal procedure for ceramic orthodontic appliances (Figure 1). To date, work has looked at the comparing the removal forces on various manufacturers of ceramic brackets, investigated alternative appliance removal mechanisms involving shear, and varying the removal strengths of bonded appliances through modifications of the adhesive by plasticizer additions. The development of this area has progressed with several presentations cited below.

Many new questions have arisen since the emergence of ceramic orthodontic appliances in treatment. While ceramic brackets are a major cosmetic improvement during treatment, some new problems have also emerged. For example, the removal mechanism is totally different for ceramic brackets than metal ones. The removal forces are also higher and instances of both bracket and tooth fracture have been recorded. Many factors could contribute to this including differences. Manufacturing designs, adhesives, tooth preparation, primers, and tooth curvature all lead to variations in performance. This approach was the crux of the efforts by both Rathburn and Sanchez.

They determined the force required to remove bonded brackets from standard substrates and also from teeth by both tensile shear forces and by torsion. The goal of this research was to determine whether some manufacturing designs and removal mechanisms were more easily removed than others. Table 1 represents a summary of removal force results by Rathburn on 5 different bracket designs using shear removal. Work by Sanchez is shown in Figure 2 documenting the removal force measurements on various brackets in torsion. A second goal was to determine whether any of these new removal methods would be better than those currently recommended by manufacturers. It appeared that torsional removal would require the least amount of force but was clinically too difficult to incorporate due to patient pain during removal.

New efforts under this project have approached this problem from a different angle. Since recorded removal forces are higher for ceramic brackets than for the stainless steel brackets everything else being the same, new efforts were undertaken to determine whether a

reduced modulus adhesive could offset the increase in force required when using ceramic brackets. By work of Starling and Love, this has been achieved through the use of plasticizers like DiEthyl Phthalate (DEP). DEP additions of about 10% of the adhesive weight have decreased the modulus and the torsional strength in bonding tests significantly and the reductions in modulus and torsional strength are shown as a function of plasticizer content in Figures 3 and 4.

There are additional concerns about whether the plasticized adhesive is affected through interactions with saliva. Recent tests indicated in Tables 2 and 3 have shown that the plasticized adhesive is relatively unaffected by the interaction with an artificial saliva solution both at room temperature and at elevated temperature. The initial reductions in the torsional removal force do not return to the virgin values without any plasticizer after exposure to the artificial saliva solution even at elevated temperature.

Several explanations are possible for this behavior. One is that there is no interaction between the plasticized adhesive and the saliva resulting in no increased stiffness of the adhesive due to salivary exposure. Another explanation for the lower torsional force is that the saliva has extracted the plasticizer leaving residual voids in the adhesive after exposure to saliva. A third explanation which cannot be ruled out is that the saliva has somehow replaced the plasticizer contributing to the lower stiffness. It is possible to elucidate this mechanism through some analytical characterization via gas chromatography and high pressure liquid chromatography on carefully fabricated samples.

Some Finite Element Analyses (FEA) have also been performed to determine the optimum level of plasticizer without causing problems during treatment. Remember, this is a stressed interface. In figure 1, the bracket is loaded through the use of a NiTi wire installed in the channel of the bracket. This is then loaded during treatment to move the teeth. The level of plasticizer should be set deliberately low such that the mechanical properties of the adhesive are not so low that creep or plastic deformation of the adhesive occurs during the loading requirements for treatment. When the mechanical properties of the 10% plasticized adhesive resin are entered into the model, the computed stresses in the FE model are low in comparison to their tensile stress limits (Figure 5). This suggests that 10% plasticization is an acceptable level for treatment. Note this model does not take into account the stresses placed on the bond during chewing. Other FEA have been done to model the removal mechanism and significantly lower inputted loads are required to cause failure within the adhesive region due to the reduced stiffness.

New Directions:

With all of the work already having been done to establish the plasticizing effect of phthalate esters and other organics on dental and orthodontic resins, our direction has shifted towards more synthetic chemistry to evaluate the plasticizing efficiency on some model compounds which look similar to the diethyl phthalate. With the concerns about salivary plasticizer leaching, structural modifications to the phthalate ester may make salivary leaching an even more remote possibility.

Diethyl phthalate is formed by the trans esterification reaction of phthalic acid or phthalic anhydride by ethanol (Reaction 1). The compound has a relatively high boiling point ($\sim 300^{\circ}\text{C}$) and is a liquid at room temperature. Similar compounds like dioctyl phthalate (boiling point = 384°C) and dibutyl phthalate (boiling point = 340°C) are made in similar ways. The interesting thing to note about these compounds is the relative lack of significant steric hinderance in the ester regions. The two ester groups in each molecule are thought to provide the attractive interactions with polymers like Poly methyl methacrylate. One interesting approach to investigating these interactions is to attach more steric hinderance to the esterification product.

In addition, there is some interesting theory behind this work. Solution mixing theory has worked well to describe the efficacy of most plasticizer systems. Conceptually, as a solvent is mixed with a polymer, entropic considerations would make intimately mixing polymer and solvent most disordered. Enthalpic considerations prevents the mixing if the solvent and polymer repulse one another. We will not get heavily involved in the theory here, but in the enthalpic term for this thermodynamic expression, there is a variable, z , called a lattice coordination number. It relates to how many polymer solvent interactions are possible per atom of solvent. By using plasticizers with a variable number of interaction points, we are really varying z , the coordination number in a very systematic way.

Some initial synthetic chemistry has already been done from work we have performed here at Georgia Tech. We have reacted phthalic anhydride with 1 phenyl - 3 propan-ol to form di 1 phenyl 3 - propyl phthalate (Reaction 2). This ester, like diethyl phthalate, has functionality two relating to two ester groups. Infrared spectroscopy has been used as a confirmation of the formation of the ester product as the shift in the carbonyl peak has occurred from 1712 cm^{-1} to 1704 cm^{-1} comparing phthalic anhydride with the liquid product in the reaction vessel. There is also accompanied by the absence of the hydroxyl peak at 3300 cm^{-1} in the liquid product that is present in the 1 phenyl - 3 propan-ol. This is a very rare compound and very little work has been done to evaluate its plasticizing effect in glassy polymer systems.

In addition, we have set out on a program to synthesize other moieties of the 1 phenyl 3 propan-ol with different functionalities, in order to test how the number of attractive sites on the plasticizer effects the mechanical properties of the compound. A variety of tests are required in order to complete this work. First, more infrared spectroscopy is needed to determine the formation products. Nuclear Magnetic Resonance (NMR) will also be used to confirm the formation of the phthalate ester compounds with attractive interactions ranging from 1-4.

Once these new plasticizers have been synthesized and characterized, these compounds will then be evaluated for their plasticizer efficacy. Similar testing on fabricated samples will evaluate the modulus as a function of temperature and torsional bond strength using functional brackets and the stainless steel bonding disk. We will further determine the efficacy of these plasticizers to modify the functional characteristics of the adhesives when used in this application.

Presentations:

M. A. Rathburn and R. F. Hochman, "Ceramic Brackets: A Comparative Study" presented at the A. A. O. Table Clinic, (Won the Table Clinic Award) (1990).

M. S. Sanchez and R. F. Hochman, "Torsional Shear Load Strengths of Chemically and Mechanically Retained Ceramic Brackets", presented at the A. A. O. Table Clinic, Seattle, WA (May 1991).

B. J. Love, R. F. Hochman and K. E. Starling, Jr., "Orthodontic Adhesive Modifications to Enhance After-Treatment Removal", presented to the Annual Adhesion Society Meeting, Hilton Head, SC (February 1992), and published in the proceedings.

K. E. Starling, B. J. Love, and R. F. Hochman, "Orthodontic Adhesive Modification to Enhance Post Treatment Debonding of Ceramic Brackets", presented at the A. A. O. Table Clinic, St. Louis, MO (May 1992).

S. Nasser, J. Slappey, B. J. Love, and W. C. Hutton, "Evaluation of the Sphercity and Surface Finish of Unimplanted and Retrieved Alloy, Ion-Treated Alloy and Ceramic Total Hip Arthoroplasty Femoral Heads", presented at the 23rd Annual Eastern Orthopaedic Society Meeting , Puerto Rico, October 1992.

B. J. Love, and K. E. Staling, Jr., "On Controlling the Adhesion of Orthodontic Appliances through Adjustment of the Interphase Mechanical Properties", presented at the 16th Annual and 4th International Symposium on Adhesion, Williamsburg, VA (February 1993).

Papers and Publications:

K. E. Starling, B. J. Love, and R. F. Hochman, "Orthodontic Adhesive Modifications to enhance Post-treatment Removal", accepted for publication, *J. Clinical Orthodontics*, (1993)

B. J. Love, and K. E. Staling, Jr., "Controlling the Adhesion of Orthodontic Appliances through Adjustment of the Interphase Mechanical Properties", accepted for publication, *The Journal of Adhesion*, (April 1993)

T. G. Wilson, and B. J. Love, Clinical Effectiveness of Fluoride Releasing Elastomers II: Enamel Hardness Levels, accepted for publication, *Pediatric Dentistry* (May 1993).

K. E. Starling, Jr., and B. J. Love, Softer Adhesive for orthodontic Applications using Ceramic Brackets, Submitted for *U. S. Patent*, February 1993.

CERAMIC BRACKET DEBONDING MODES

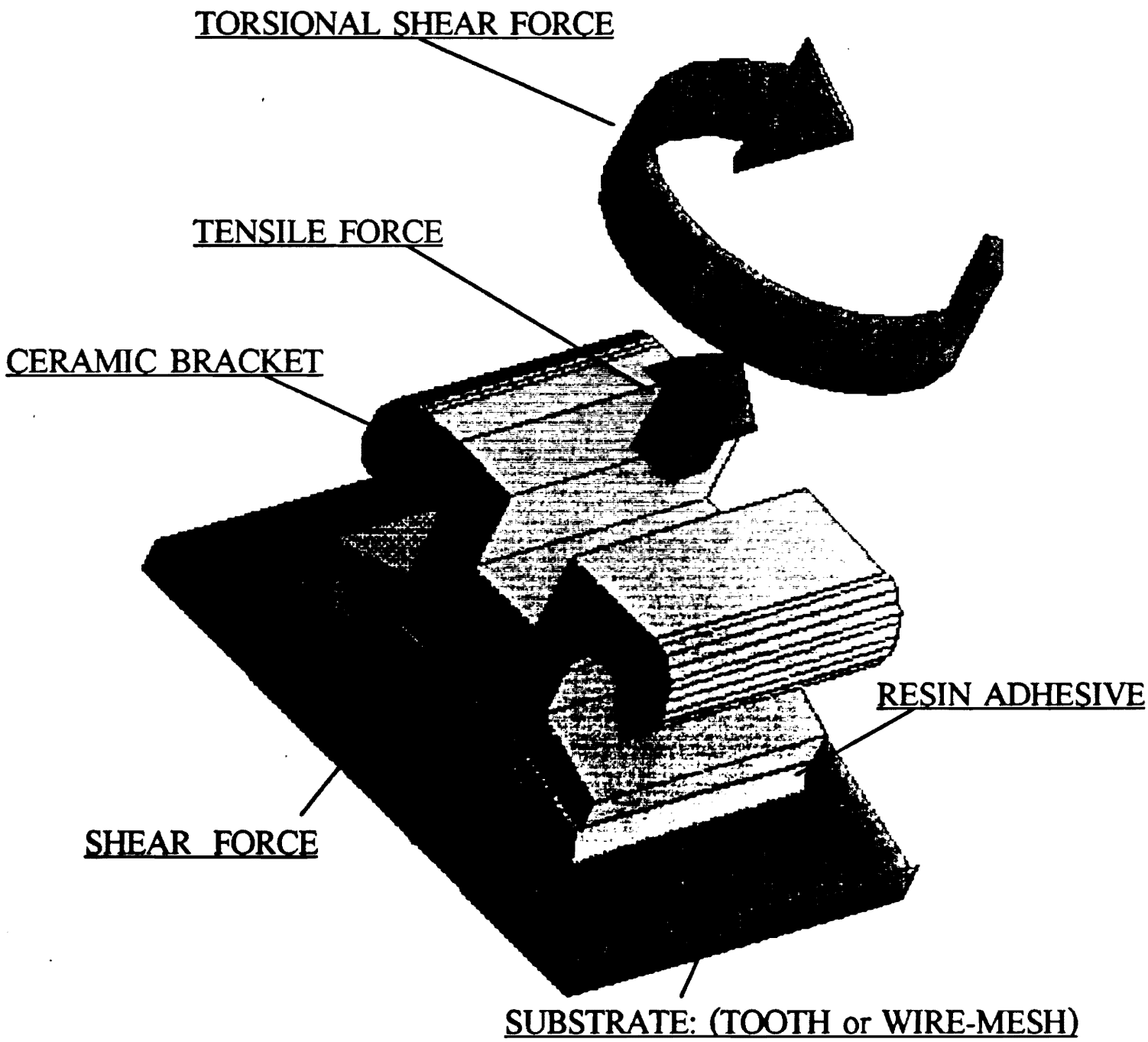


Figure 1

(Group I) Mean values of bond loads for each bracket. These values were calculated for all observations including those recorded for bracket fractures.

<i>Bracket</i>	<i>N</i>	<i>Mean Load Value (lbs)</i>	<i>Standard Deviation (lbs)</i>
Quasar	18	59.97	27.58
Illusion	19	91.11	52.31
GAC III	17	64.91	38.35
GAC IV	20	81.58	42.06
Transcend 2000	20	81.13	45.82

N represents the number of successful tests for each bracket including those recorded as bracket fractures.

(Group II) Mean values and standard deviations of bond loads for each bracket. These values were calculated for all observations **except** for those recorded as bracket fractures.

<i>Bracket</i>	<i>N</i>	<i>Mean Load Value (lbs)</i>	<i>Standard Deviation (lbs)</i>
Quasar	17	56.74	24.65
Illusion	14	76.61	48.00
GAC III	14	52.29	24.79
GAC IV	20	81.58	42.06
Transcend 2000	16	67.97	40.15

N represents the number of successful tests for each bracket excluding those recorded as bracket fractures.

Table 1: Peak removal loads for ceramics removed by a shearing force (from Rathburn)

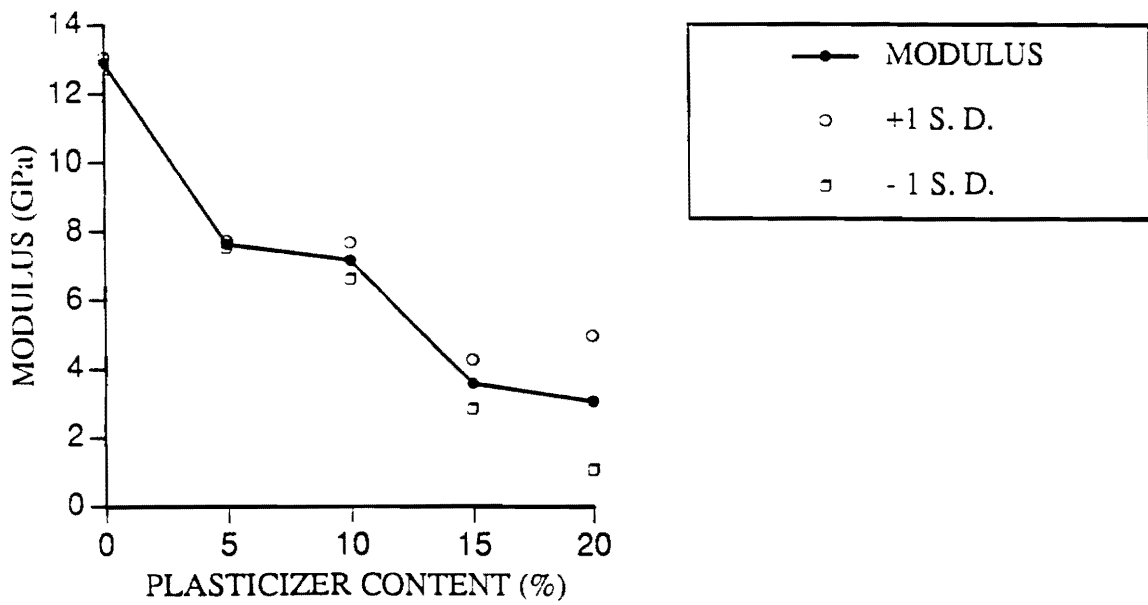


Figure 3. Modulus vs. plasticizer content at 30°C.

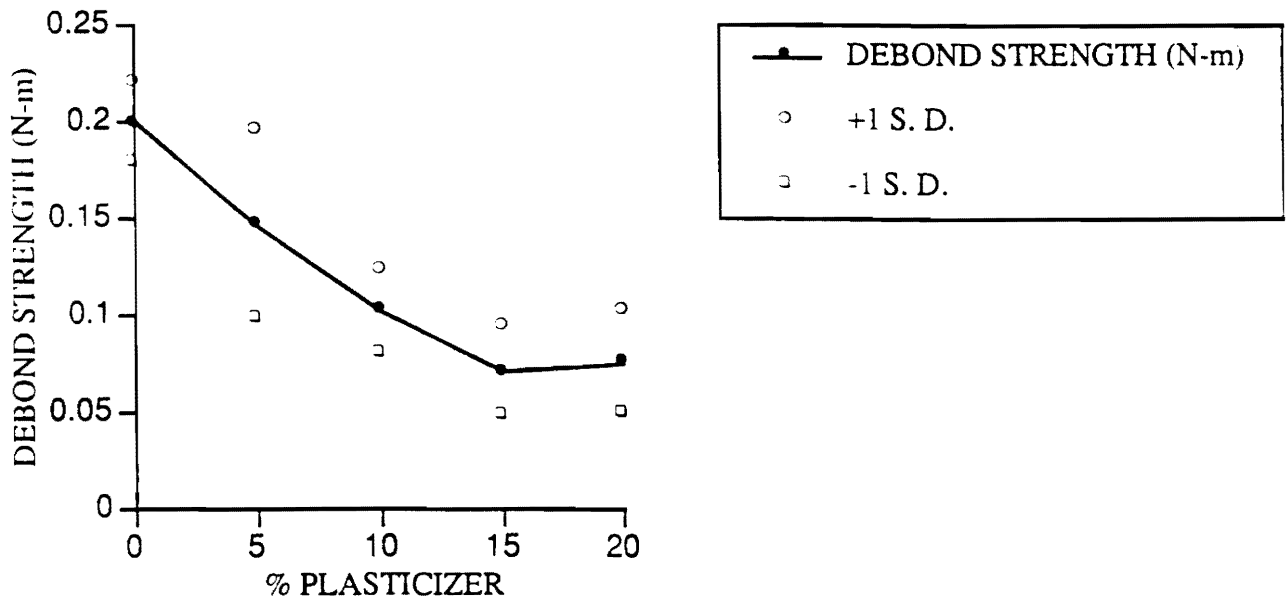


Figure 4. Torsional debond strength(N-m) vs. plasticizer content.

Torsional debond strength (N-m \pm 1S.D.)			
Days (at room temp.)	% Plasticizer		
	0	5	10
0	0.20 \pm 0.02	0.15 \pm 0.05	0.10 \pm 0.02
15	0.16 \pm 0.05	0.15 \pm 0.08	0.11 \pm 0.05
30	0.21 \pm 0.04	0.17 \pm 0.06	0.09 \pm 0.01

Table 2: Torsional Debond Strength as a function of time in the Synthetic Saliva Solution (Room Temperature)

Torsional debond strength (N-m \pm 1S.D.)			
Days (at 60°C)	% Plasticizer		
	0	5	10
0	0.20 \pm 0.02	0.15 \pm 0.05	0.10 \pm 0.02
15	0.13 \pm 0.03	0.13 \pm 0.03	0.09 \pm 0.01
20		0.16 \pm 0.03	0.08 \pm 0.02
25		0.13 \pm 0.05	0.08 \pm 0.01

Table 3: Torsional Debond Strength as a Function of Time in the Synthetic Saliva Solution (60°C)

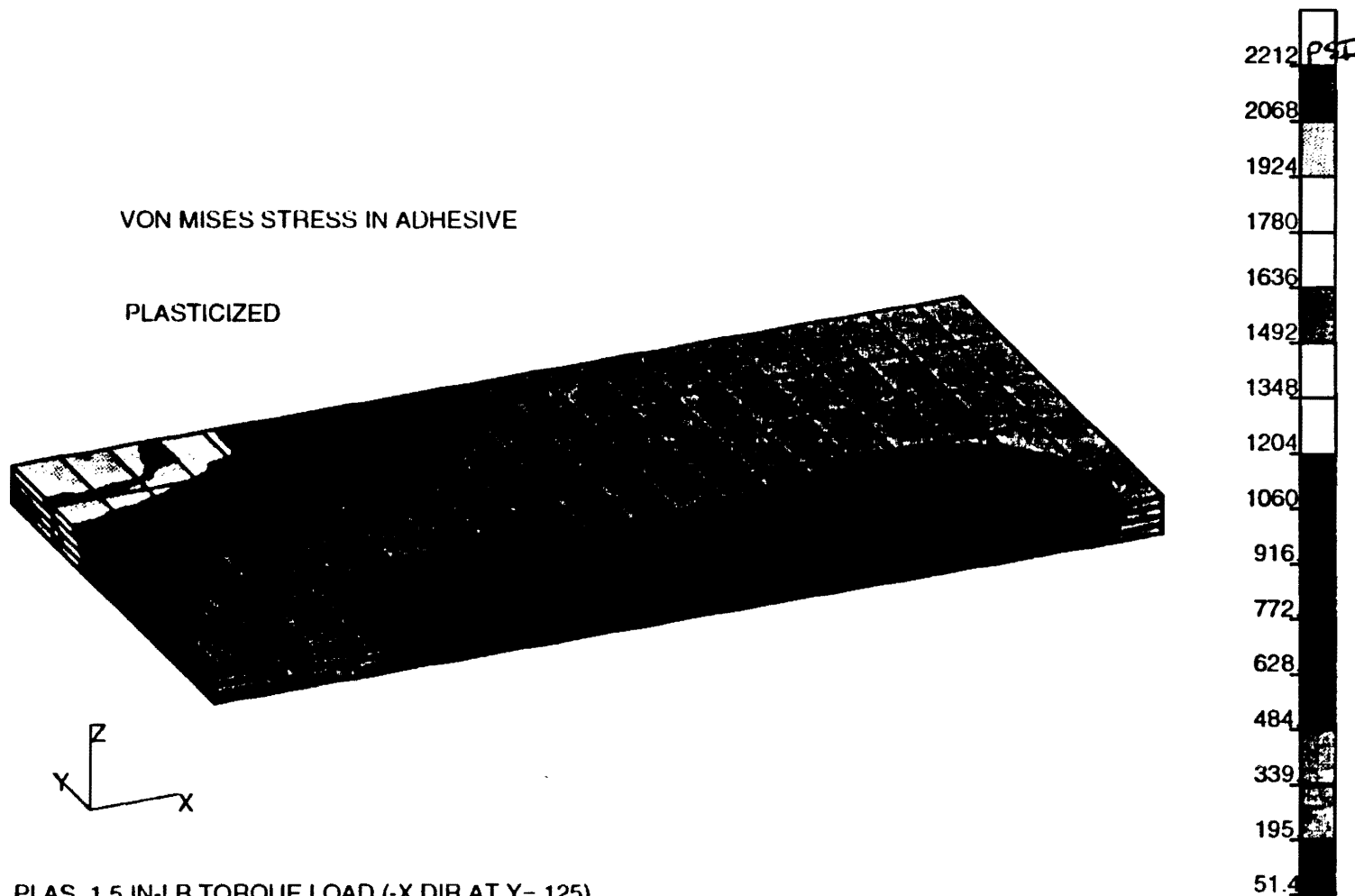
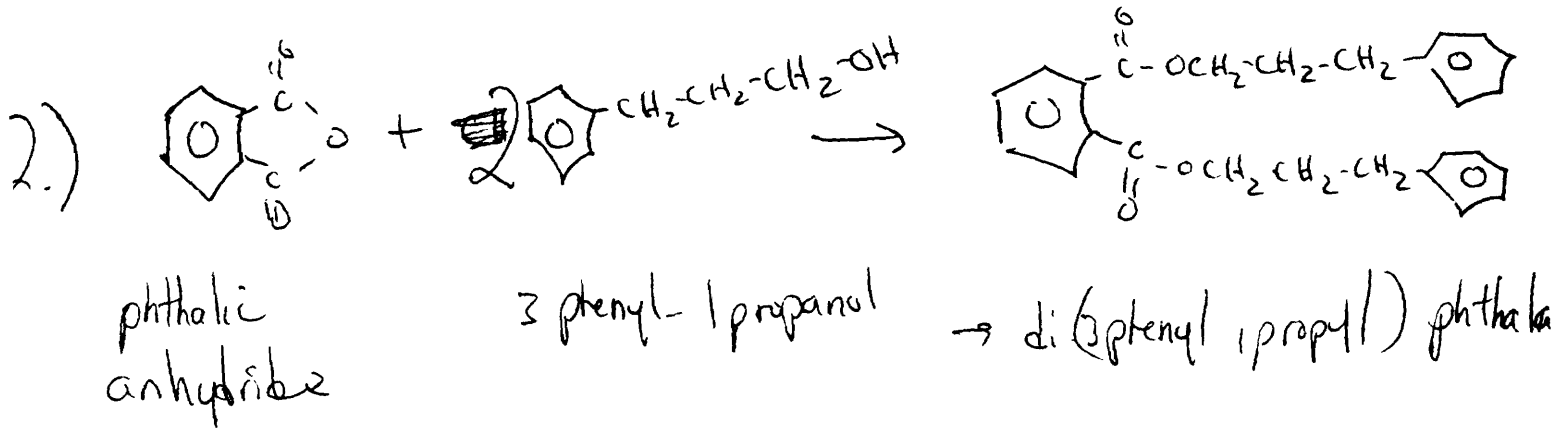
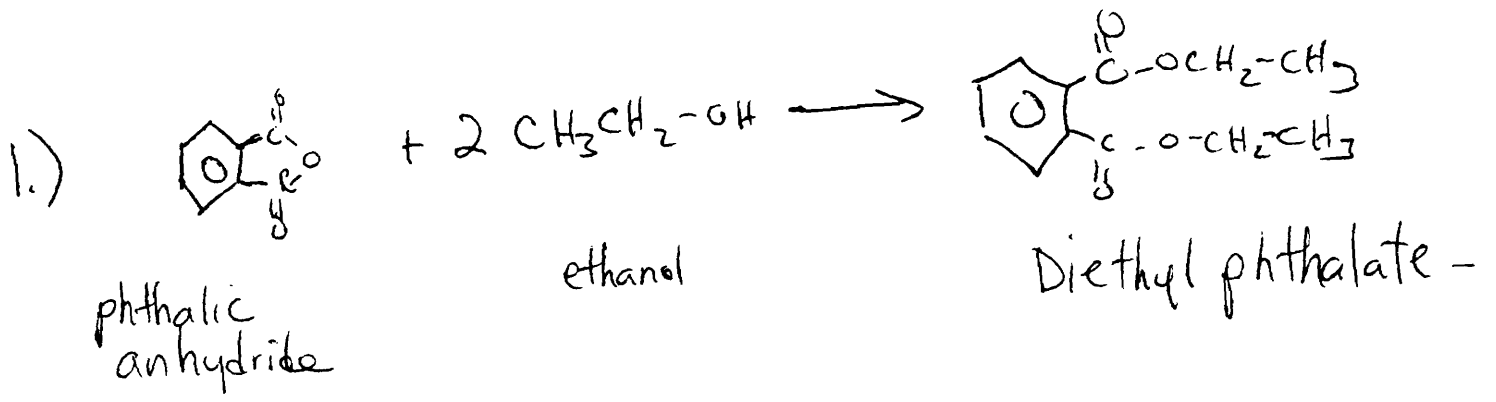


Figure 5: FEM Von Mises Stress Distribution for the plasticized region with a typical torque removal force is applied (Simulating Removal).



**CERAMIC BRACKETS:
A COMPARATIVE STUDY**

EMORY UNIVERSITY
SCHOOL OF POSTGRADUATE DENTISTRY
DEPARTMENT OF ORTHODONTICS

MELISA A. RATHBURN, D.D.S.

Torsional-shear load strengths of chemically and mechanically retained ceramic brackets

MARK S. SANCHEZ, D.D.S.

PURPOSE

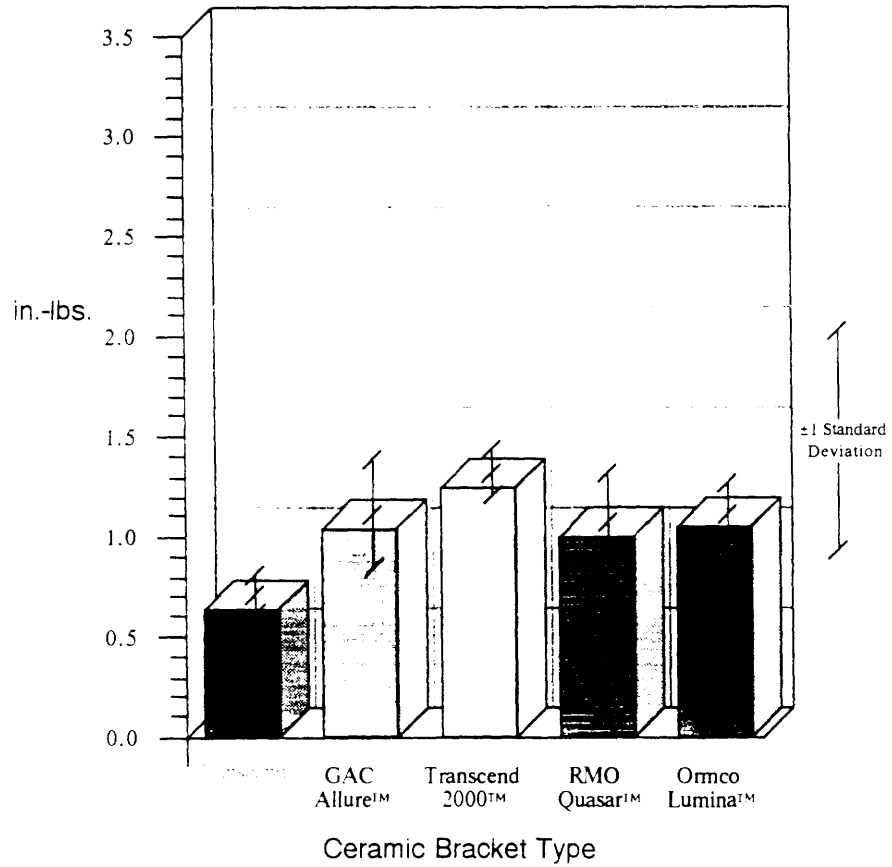
- TO DETERMINE THE AVERAGE PEAK DEBONDING LOAD OF SOME ORIGINAL SILANE-COATED CERAMIC BRACKETS USING SLOW TORSIONAL SHEAR
- TO DETERMINE IF NEWER BRACKET BASE DESIGNS HAVE A CLINICALLY SIGNIFICANT REDUCTION IN PEAK DEBONDING LOAD UNDER TORSIONAL SHEAR FORCES

MATERIALS & METHODS

- FIVE CERAMIC BRACKETS WERE TESTED:
FOUR COMMERCIALY AVAILABLE, AND ONE THAT HAS BEEN DISCONTINUED
- A 75%-FILLED DIMETHYLMETHACRYLATE RESIN SERVED AS THE ADHESIVE
- TWO SUBSTRATES WERE USED:
EXTRACTED HUMAN TEETH (ALL LOWER INCISORS TO GET AS FLAT A SURFACE AS POSSIBLE) AND A FIXED WIRE-MESH (TO SIMULATE ETCHED ENAMEL) LIKE THAT FOUND ON METAL BRACKET BASES
- TOOTH SAMPLES WERE CLEANED WITH NONFLUORIDE PUMICE AND ETCHED FOR 60 SECONDS
- MANUFACTURER'S DIRECTIONS WERE FOLLOWED FOR MIXING THE TWO-PASTE RESIN, AND THE BRACKETS WERE BONDED TO THE SUBSTRATES
- NINE TESTS WERE PERFORMED FOR EACH BRACKET TYPE ON THE HUMAN TOOTH SAMPLES (TOTAL: 45 TESTS)

RESULTS

AVERAGE PEAK LOAD
TORSIONAL SHEAR DEBONDING
FILLED RESIN ADHESIVE - WIRE/MESH SUBSTRATE



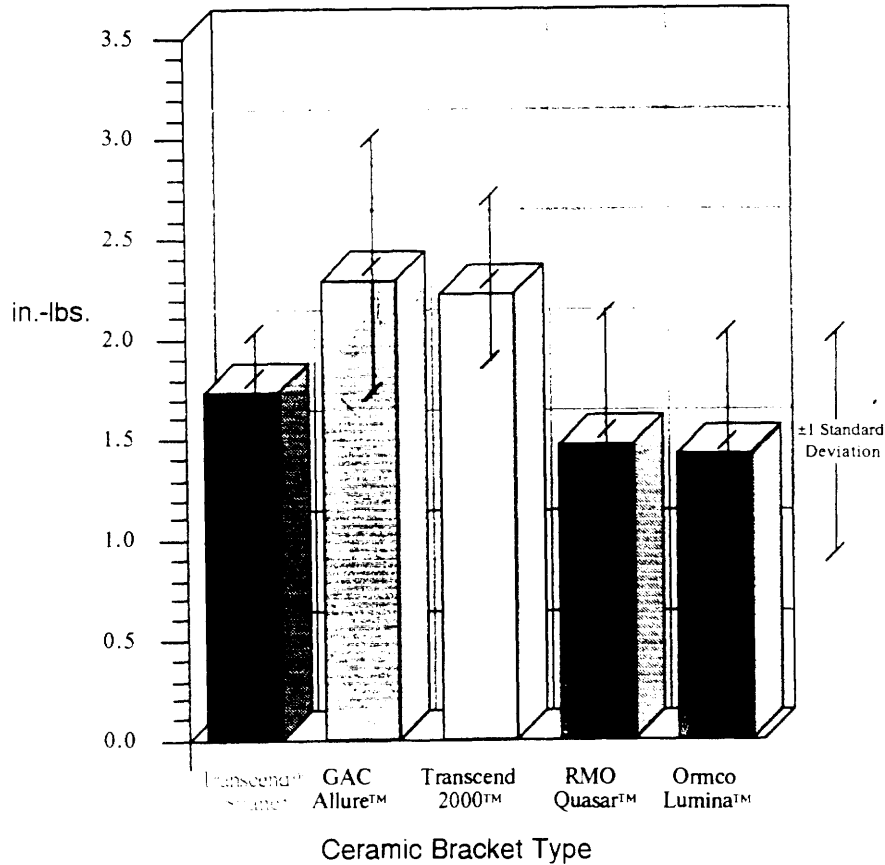
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RESULTS

CERAMIC BRACKET	BASE DESIGN	RETENTION MECHANISM	FRACTURE PLANE	BRACKET FAILURES
UNITEK TRANSCEND™‡	SMOOTH SURFACE	CHEMICAL	RESIN-BRACKET INTERFACE	0/9
GAC ALLURE IV™	DIMPLED SURFACE	CHEMICAL/MECHANICAL	RESIN-BRACKET INTERFACE	0/14
UNITEK TRANSCEND 2000™	ROUGH ANGULAR SURFACE	MECHANICAL	INTRARESIN	3/14
RMO QUASAR™	SMOOTH SURFACE	CHEMICAL	RESIN-BRACKET INTERFACE	4/14
ORMCO LUMINA™	SINTERED BEADS	MECHANICAL	BEAD-BRACKET INTERFACE	0/14

‡ NO LONGER MANUFACTURED NO ENAMEL FRACTURES IN ANY TEST

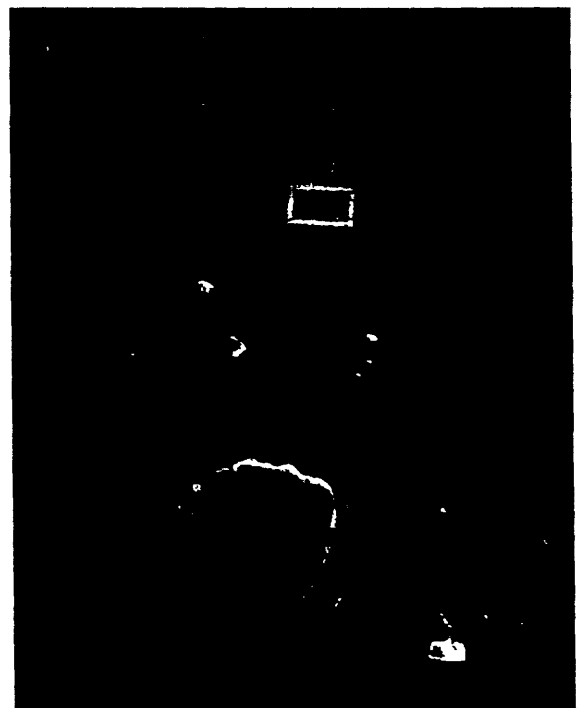
AVERAGE PEAK LOAD
TORSIONAL SHEAR DEBONDING
FILLED RESIN ADHESIVE - TOOTH SUBSTRATE



‡ No Longer Manufactured

CONCLUSIONS

- AT THE 95% CONFIDENCE LEVEL ($p < 0.05$), EVERY BRACKET DESIGN HAD A HIGHER AVERAGE DEBONDING LOAD THAN THE OLDER, SMOOTH-BASED, BRACKET WITH SILANE COUPLER WHEN TESTED ON A WIRE-MESH SUBSTRATE
- AT THE 95% CONFIDENCE LEVEL ($p < 0.05$), NO BRACKET DESIGN WAS STATISTICALLY DISTINGUISHABLE FROM THE OLDER SMOOTH-BASED BRACKET WITH SILANE COUPLER WHEN TESTED ON A TOOTH SUBSTRATE
- THE GAC AND UNITEK BRACKETS AVERAGED STATISTICALLY DISTINGUISHABLE ($p < 0.05$) HIGHER DEBONDING LOADS THAN THE RMO AND ORMCO BRACKETS
- POLYMER FRACTURE TOUGHNESS IS RATE DEPENDENT; THEREFORE THESE RESULTS ARE MINIMUMS (IMPACT TORQUING WILL CAUSE HIGHER LOADS DURING DEBONDING)
- NEWER BASE DESIGNS HAVE NOT SHOWN REDUCED DEBONDING LOADS AND SHOULD NOT BE TORQUED DURING DEBONDING



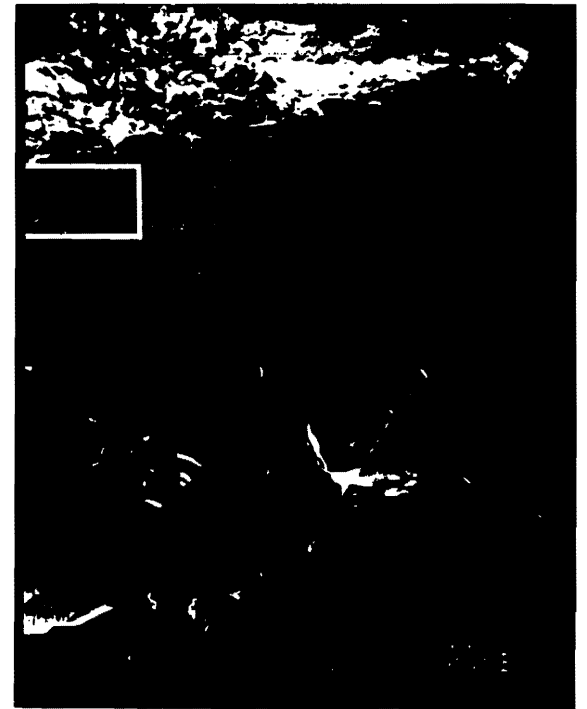
- FIVE TESTS WERE PERFORMED FOR EACH BRACKET TYPE ON THE WIRE-MESH SUBSTRATE (TOTAL: 25 TESTS)
- SLOW TORSIONAL SHEAR WAS APPLIED AT 5°/SECOND UNTIL THE BRACKET DEBONDED
- PEAK TORQUE FOR EACH TEST WAS RECORDED IN INCH-POUNDS
- FOUR ONE-WAY ANALYSES OF VARIANCE WERE PERFORMED FOR EACH BRACKET TYPE WITHIN EACH SUBSTRATE GROUP
- TWO-WAY ANALYSES OF VARIANCE COMPARED EACH SUBSTRATE
- FRACTURE PLANES WERE OBSERVED AND RECORDED USING SCANNING ELECTRON MICROSCOPY
- FRACTURES IN THE BRACKET BODY WERE RECORDED AS BRACKET FAILURES



TOOTH & RESIN
SMOOTH BASE: WITH SILANE



TOOTH & RESIN
GAC ALLURE IV™



UNITEK TRANSCEND 2000™

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Guide for 3-space
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EVALUATION OF THE SPHERICITY AND SURFACE FINISH OF UNIMPLANTED AND RETRIEVED ALLOY, ION-TREATED ALLOY, AND CERAMIC TOTAL HIP ARTHROPLASTY FEMORAL HEADS. Sam Nasser, M.D., 20 Linden Avenue, Atlanta, Georgia 30308; Joseph Slappey, M.D., Atlanta, Georgia; Brian Love, Ph.D., Atlanta, Georgia; William Hutton, D.Sc., Atlanta, Georgia. As the longevity of total joint arthroplasty increases, wear effects become more important. Identifying and using superior surfaces should decrease wear rates. The purpose of this study was to assess the physical characteristics of the surfaces of various commercially-available femoral heads, and to determine the effects of ion-hardening treatments on these characteristics.

Femoral head prostheses fabricated of stainless steel (316L), ion implanted titanium alloy (Ti6Al4V), cast cobalt-chromium alloy, forged cobalt-chromium alloy, ion implanted cobalt-chromium alloy, alumina ceramic and zirconia ceramic were obtained from 10 different manufacturers for comparisons of unimplanted components. In addition, representative femoral heads of similar composition (where available), retrieved either at autopsy or revision (average time implanted, 8 years; range 4 mo.-18 yr.) were also analyzed. The unimplanted specimens were taken directly from the commercial packaging and were not further treated. The retrieved specimens were washed with a commercial soap solution in an ultrasonic bath, followed by rinsing in a sodium carbonate solution, followed by deionized water, isopropyl alcohol, then air drying in a dust-free chamber.

Physical measurements of sphericity were made using a pneumocentric roundness gauge utilizing a spherical stylus

with a radius of 1.6mm, held in a stationary position. Surface finish was evaluated using a diamond tipped stylus with a radius of 2 μm in a computer controlled system. Surface texture was also evaluated using laser interferometry with a Maxim noncontact profilometer. The ceramic balls were sputtered with gold to facilitate conduction, then all heads were viewed at 500X and 3000X using a Hitachi 5-800 scanning electron microscope with accelerating voltages between 15 and 20KeV.

Considerable variation was noted in the physical measurement of the sphericity and the surface finish of the heads. On visual inspection two different types of "blemishes" were noted: "pits" resulting from flaws in the fabrication process, and linear scratches produced during the polishing process. Ti6Al4V heads showed by far the poorest sphericity and surface finish. E.M. examination confirmed physical measurement, and dramatically demonstrated wear patterns on these specimens despite hardening procedures. Cast cobalt-chromium was superior to titanium in both sphericity and surface finish, but there was marked variation in both parameters from one manufacturer to another. Stainless steel was similar in character to the better cast cobalt-chromium alloy heads. Forged cobalt chromium alloy demonstrated better sphericity than the cast samples, as well as fewer pitting defects, but the pattern of linear striations was similar to cast heads. Ion treatments of the alloy heads did not appear to improve either the sphericity nor the surface finish of the test specimens. Comparison of new and retrieved stainless steel and cobalt-chromium heads showed few differences. Both types of ceramic were superior in sphericity to alloy heads, and physical measurement of surface finish showed these materials equal to or better than the best forged alloy heads. Alumina ceramic showed a more prominent pattern of linear

scratches, while zirconia showed more pitting defects. However, as with the alloy heads, there was considerable variation in surface finish among the various manufacturers. Conclusion: Sphericity and surface finish vary by material as well as by manufacturer. Surface hardening (ion bombardment, nitriding) does not improve finish or sphericity.

**Orthodontic Adhesive Modification to Enhance
Post-Treatment Debonding**

Kenneth E. Starling, Jr., D.D.S.

Department of Orthodontics

Emory University School of Post-Graduate Dentistry

and

Brian J. Love, Ph.D.

Materials Engineering Department

Georgia Institute of Technology

INTRODUCTION

The introduction of ceramic orthodontic brackets, made from single crystal or polycrystalline sapphire is seen as a major advancement in the esthetics of orthodontic treatment, compared to the existing stainless steel brackets.¹ Unfortunately, there are documented problems with the ceramic systems. The most serious of the drawbacks occurs during the bracket removal after orthodontic treatment is complete.

Several problems have been documented during the removal process. Higher forces are required to remove ceramic brackets than the metal brackets from the teeth.² This is attributed to the peeling mechanism used to remove the metal bracket that is not available for the ceramic bracket.³ In the ceramic system all three components (the bracket, the enamel, and the highly ceramic filled polymer resin) are strong and brittle. These higher forces have, at times, exceeded the strength of either the bracket itself, or more importantly the enamel to which the bracket is bonded. If the bracket fractures, diamond grinding of the residual ceramic is required for removal.³ If the enamel fractures, an expensive restorative dental procedure is required to repair the fractured region.³ Either procedure is time consuming and stressful for both the patient and dentist. "Enamel crazing" has been reported as an additional sign of the brittle removal of these brackets.⁴ While crazing does not lead to an immediate need for restorative care, it does indicate enamel damage. There is a real need to make the removal process easier and more predictable.

There have been an number of efforts aimed at facilitating the removal process.^{3,4,5,6} Bracket manufacturers have placed deliberate flaws within the brackets base to cause a lower strength failure within this region during removal.⁶ However, placing stable flaws within the ceramic is not trivial, or inexpensive. Other efforts have looked at different removal techniques (i.e. torsional and shear modes).⁷ Some promising work investigated the use of a heated tool to lower the modulus and tensile strength of the adhesive during removal, but considering the potential tooth pulp damage while heating the adhesive, widespread use of these instruments has not developed.^{3,5,8,9}

Our belief is that a more effective modification of the polymeric adhesive mechanical properties would be accomplished through interaction with plasticizers. Plasticization is a common technique in polymers.¹⁰ Our interest was to determine whether this would be an effective way to lower the removal forces.

METHODS AND MATERIALS

The adhesive that was modified was Phase II[®],* a two part BIS-GMA adhesive mixed with the appropriate amounts of an aliphatic pthalate as plasticizer. The brackets used during all phases of the experiments (unless otherwise noted) were Signature^{®†} lower incisor brackets with no silane treatment.

* Reliance Orthodontic Products, P.O. Box 678 Itasca, Ill. 60143

† RMO[®], Inc., P.O. Box 17085, Denver, CO 80217

Tensile strength samples of 0% and 10% plasticized adhesive were made from a mold into strips approximately 1.5 mm thick, 12 mm wide and 35 mm long. The samples were glued to retention blocks and centered in the load cell. Tensile strengths were measured on an Instron T/T testing machine with a 10,000 pound load cell, a calibrated 50 pound full scale load, at a testing rate of 0.5 inches per minute.

Samples of the adhesive and the plasticized adhesive were made approximately 0.2 mm thick and 6 mm wide and 25 mm long. These strips were placed into a Polymer Laboratories DMTA. The Shear Storage Modulus was measured as a function of temperature from 30°C to 200°C at a frequency of 10 Hz.

Metal mesh bonding disks were used to bond the brackets, to eliminate the variations that occur in enamel. The metal mesh was first sealed with the manufacturer's sealer to increase the wetting of the mesh. Ceramic brackets were then bonded to the metal mesh bonding disks with the adhesive mixed with the appropriate amount of plasticizer (0, 5, 10, 15, and 20% by weight). The excess adhesive was carefully removed from around the bracket base and the adhesive was allowed to set for 24 hours. Removal force measurements were taken in a torsional shear mode by a 5 inch-pound Sturtevant torque wrench, measuring peak torque forces.

For the adhesion to enamel tests, three teeth were pumiced and acid etched with 40% phosphoric acid for 30 seconds, then rinsed, dried, and sealed with the manufacturer's sealer. The adhesive was mixed as before and the brackets were bonded and allowed to cure for 24 hours. The brackets were

Allure®‡ brackets and the Signature® brackets. These were removed with the same Sturtevant torque wrench and the torsional debond strengths were recorded. The teeth and remaining adhesive were gold sputtered and scanning electron microscopy was performed, using a Hitachi S-800 microscope, to observe the fracture region.

Limited room temperature 15 and 30 day artificial saliva tests were performed with the brackets bonded to the metal mesh bonding disks in the same manner as the initial bracket debonding strengths. The adhesive was allowed to cure for 10 minutes and then placed in an artificial saliva at room temperature.¹¹ The brackets were debonded with the same Sturtevant torque wrench at 15 and 30 days. The torsional debond strengths were recorded.

RESULTS

Table 1 displays the tensile strength measurements of the samples at 0% and 10% plasticizer content. The results showed a significant decrease in the tensile strength from 6.2 ± 0.3 kpsi for the unaltered adhesive to 2.7 ± 0.5 kpsi for the 10% plasticized adhesive.

Figure 1 shows the shear storage modulus (GPa) vs. plasticizer content at 30°C. The unaltered adhesive mean modulus was 12.88 ± 17 GPa; 5% plasticized, 7.64 ± 0.1 GPa; 10% plasticized, 7.13 ± 0.52 GPa; 15% plasticized, 3.56 ± 0.72 GPa; and the 20% plasticized, 3.04 ± 1.95 GPa.

‡ GAC®, International, Inc., 185 Oval Drive, Central Islip, NY 11722

Figure 2 demonstrates the torsional debond strength (N-m) vs. plasticizer content for the ceramic brackets bonded to the metal mesh disks. The debonding strengths under torsion of the adhesive was lowered from a mean of 0.20 ± 0.02 N-m with out plasticizer to a significantly lower mean of 0.070 ± 0.02 N-m containing 15% plasticized adhesive and 0.075 ± 0.02 N-m containing 20% plasticized adhesive.

The scanning electron micrograph shown in Figures 3a is of the unaltered adhesive. The micrograph seen in Figure 3b is of the 20% plasticized adhesive and Figure 3c is the bracket base from the corresponding 20% plasticized adhesive.

The debonding shear strengths of the adhesive exposed to artificial saliva are reported in Figure 4. The adhesive shear strength did not change significantly over the thirty day test period in the room temperature artificial saliva.

DISCUSSION

The tensile strength measurements (Table 1) evaluate physical properties of the adhesive itself, by removing the variables of the bracket and the metal mesh disk. The decrease in the tensile strength from 6.2 ± 0.3 kpsi for the unaltered adhesive to 2.7 ± 0.5 kpsi for the 10% plasticized adhesive is due to the softening of the adhesive as the plasticizer increases.

Enamel cohesive strength (tensile strength) is reported to range between 10-35 N/mm².¹ The measured tensile strength of the unmodified adhesive is

45.5 N/mm². This compares to the tensile strength of the 10% plasticized adhesive which has a tensile strength of 18.3 N/mm². The tensile strength of the unmodified resin is significantly greater than the enamel cohesive strength. This is clinically important since the adhesive strength is sufficient to fracture enamel. The 10% plasticized adhesive has a tensile strength that is in the lower half of the range of the enamel cohesive strength and less than half that of the unplasticized adhesive.

The shear storage modulus measurements (Figure 1) showed a significant reduction in modulus with increasing plasticizer. The shear modulus is a measure of the polymer's resistance to flow during deformation. The lower shear modulus seen in the plasticized samples indicates greater molecular motion and increased plasticity of the sample, and hence lower resistance to flow.

The shear bond strengths measured (Figure 2) correlate with the other physical properties of the altered adhesive. The fracture mode changes to allow more plastic deformation within the adhesive (less brittle) with increasing plasticizer, due resin softening. These changes should be most evident at the corners of the brackets since the levels of torsional shear stress are the highest at these points.

The apparent leveling of the shear bond strengths with increased plasticizer needs further investigation. Our efforts have focused on modifying the adhesive mechanical properties only enough to move the bond failure location into the adhesive and away from the enamel and bracket interfaces.

The debond strength is controlled by adding controlled amounts of the plasticizer.

The scanning electron micrograph (Figure 3a) of the adhesive without plasticizer revealed evidence of a brittle fracture occurring at the bracket-adhesive interface. An area of the brittle fracture looks like the base of the bracket, an impression in the adhesive of the bracket base. This is due to the true "adhesive" failure. The sample of 20% plasticized adhesive (Figure 3b) shows more evidence of plastic deformation in the adhesive. Plastic deformation within the adhesive is evident near the corner of the original bracket-adhesive interface. Fracture within the BIS-GMA adhesive has occurred in this corner region, the area with the highest shear stress under torsion. The bracket base (Figure 3c) retains residual adhesive in the corner region of the "cohesive" fracture, within the adhesive. The sample of 10% plasticized adhesive (not shown) reveals similar but less obvious results. The plasticized resins both had "cohesive" fractures of the adhesive in the corner region, demonstrating increased plastic deformation and a lowering of the adhesive modulus.

The artificial saliva testing investigated the change, if any, of the adhesive when exposed to saliva. Leaching of plasticizer from the adhesive could potentially reduce the softening effect. Limited testing done to date revealed no significant change from 0 to 30 days in the room temperature bath (Figure 4). This is the expected result since the surface area of adhesive exposed is very small and the amount of plasticizer is also quite small. Future work will include elevated temperature exposure studies to accelerate the oral aging to reflect typical patient treatment periods.

As an adjunct to this research we have attempted computer finite element modeling of the debonding mechanism. The finite element modeling is in its early stages of development. However, the initial models have been quite revealing and informative. The goal of the finite element modeling is to predict the properties of a modified material on the computer to more efficiently study the plasticizer effect on the filled adhesive. Knowing the mechanical properties of the material and the stresses that are placed on the bracket, we should be able to better predict the material's reaction to the stress. This is valuable information and could prevent the reduction of the material strength below those that are required clinically.

CONCLUSIONS

Addition of increased plasticizer to this BIS-GMA adhesive substantially lowers the observed peak torque required for bracket removal from the standard substrate. The observations indicate that the increased plasticizer concentration makes cohesive, ductile fracture within the adhesive more likely. Given that the overall goal of this effort is to make bracket removal easier and more predictable; moving the fracture zone within the adhesive contributes to a safer removal of the ceramic bracket. While the bracket removal force is controllable, research needs to identify the minimum acceptable clinical force required for acceptable orthodontic treatment.

Of additional concern is the oral implication of this application of plasticizer; salival interaction with the adhesive needs to be further considered from a functional as well as a toxicological standpoint. Continued efforts will

investigate the link between salivary extracts of plasticizer and the adhesion of bonded brackets in this type of adhesive configuration.

From the completed research the following conclusions can be drawn:

1. The modulus of a heavily ceramic filled BIS-GMA adhesive can be tailored through the use of selected plasticizers.
2. Increases in plasticizer content within the adhesive lead to lower measured adhesion values when bonded to a standard substrate.
3. The plasticized adhesive may make removal of the more esthetic ceramic brackets easier and more predictable, through a ductile failure mechanism within the adhesive, rather than the current brittle mechanism.

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Table 1. Tensile strength measurements at 0 and 10 % plasticizer content.

<u>% Plasticizer</u>	<u>0%</u>	<u>10%</u>
Adhesive Tensile Strength (kpsi)	6.3 ±0.3*	2.7 ±0.5*
Adhesive Tensile Strength (N/mm ²)	46	18
Enamel Tensile Strength (N/mm ²) ¹	10-35	

*1 Standard Deviation

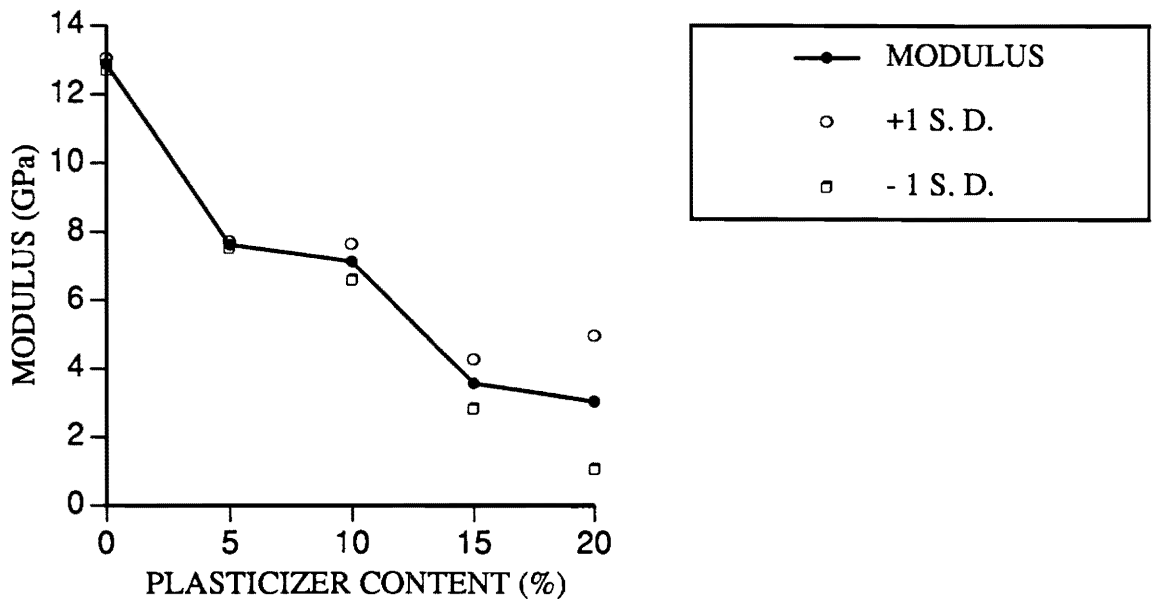


Figure 1. Modulus vs. plasticizer content at 30°C.

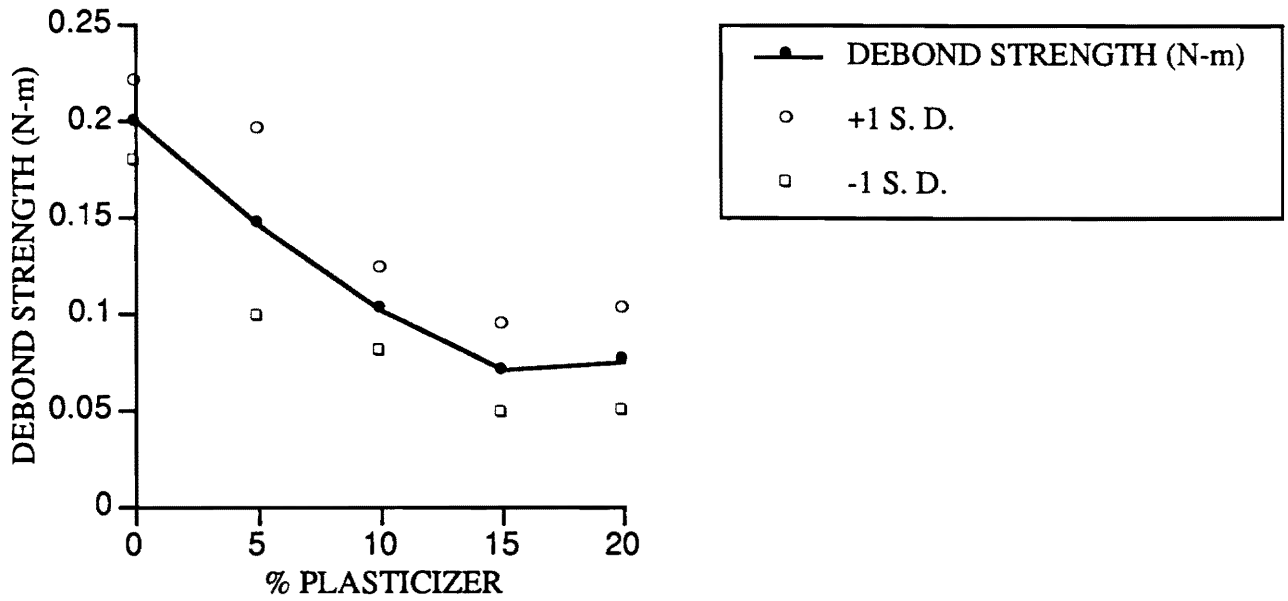


Figure 2. Torsional debond strength(N-m) vs. plasticizer content.

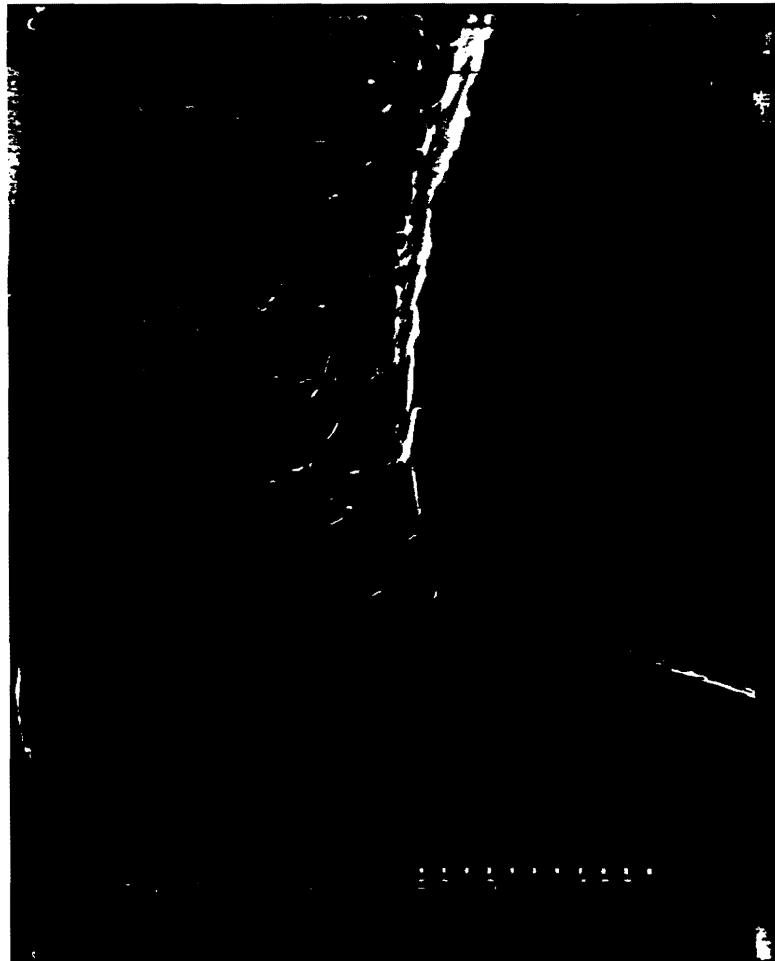


Figure 3a: Scanning Electron Micrograph of the Tooth Region of the Unaltered Adhesive After Fracture. There is a Complete Adhesive Fracture Between the Bracket and the Adhesive

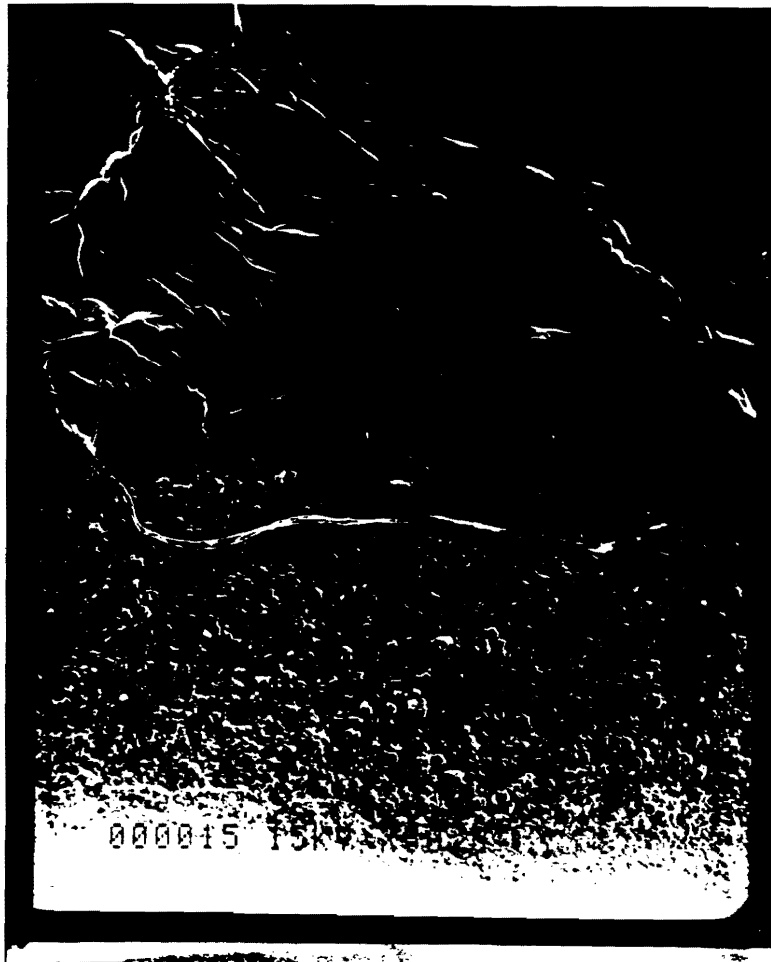


Figure 3b: Scanning Electron Micrograph of Tooth Region of the 20% Plasticized Adhesive. There is a Cohesive Fracture Region near the Corner of the Bracket. This is the Highest Stress Region.

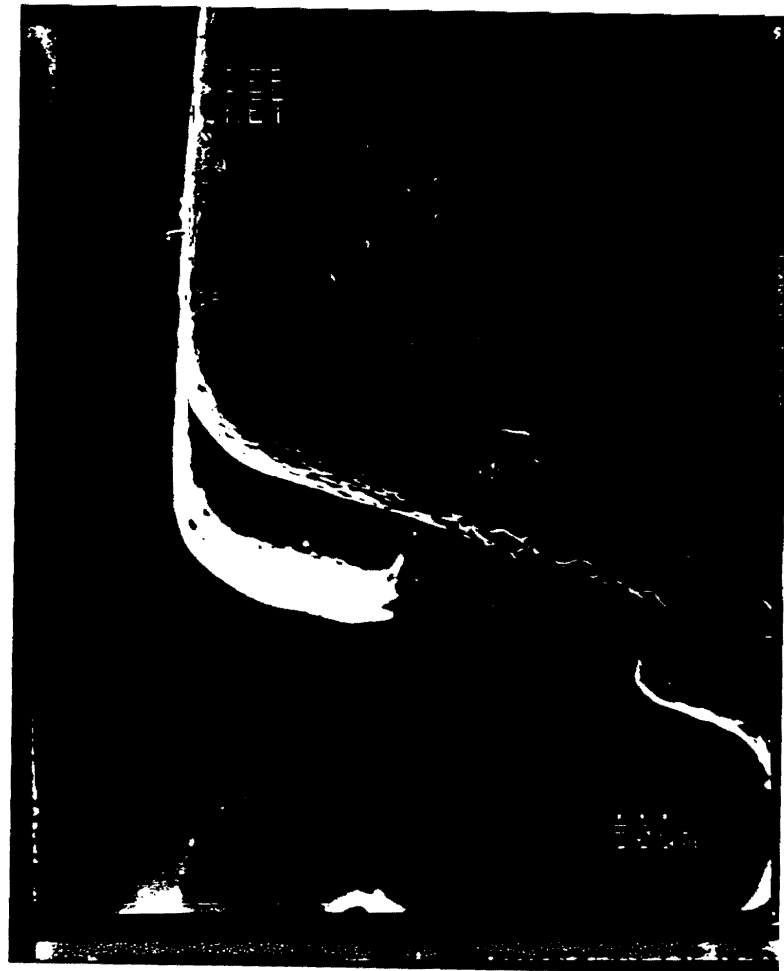


Figure 3c: Scanning Electron Micrograph of Bracket Region after Fracture. Note the Residual Adhesive left on the Bracket after Fracture Further Confirming the Cohesive Fracture Mechanism.

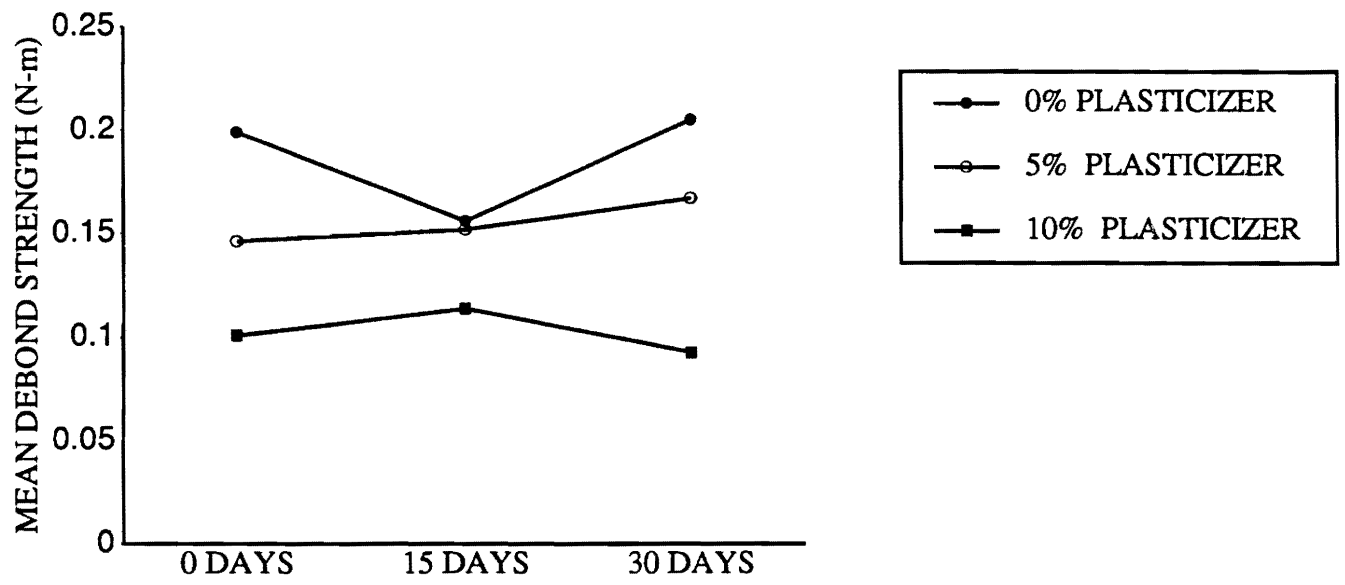


Figure 4. Debonding strengths vs. days in saliva bath.

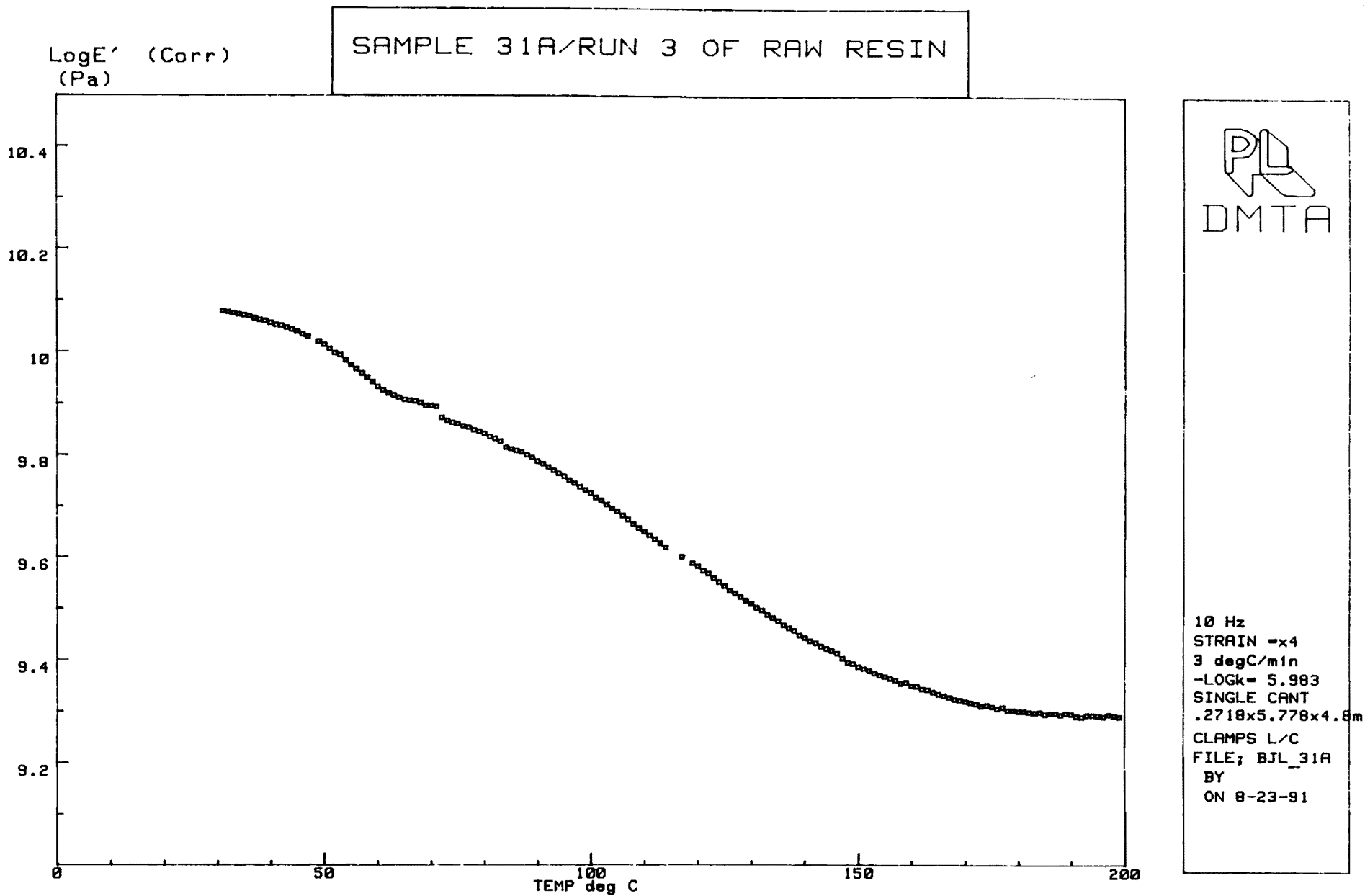


Figure 5. Typical shear storage modulus graph for the unmodified adhesive from 30 to 200°C at 10 Hz.

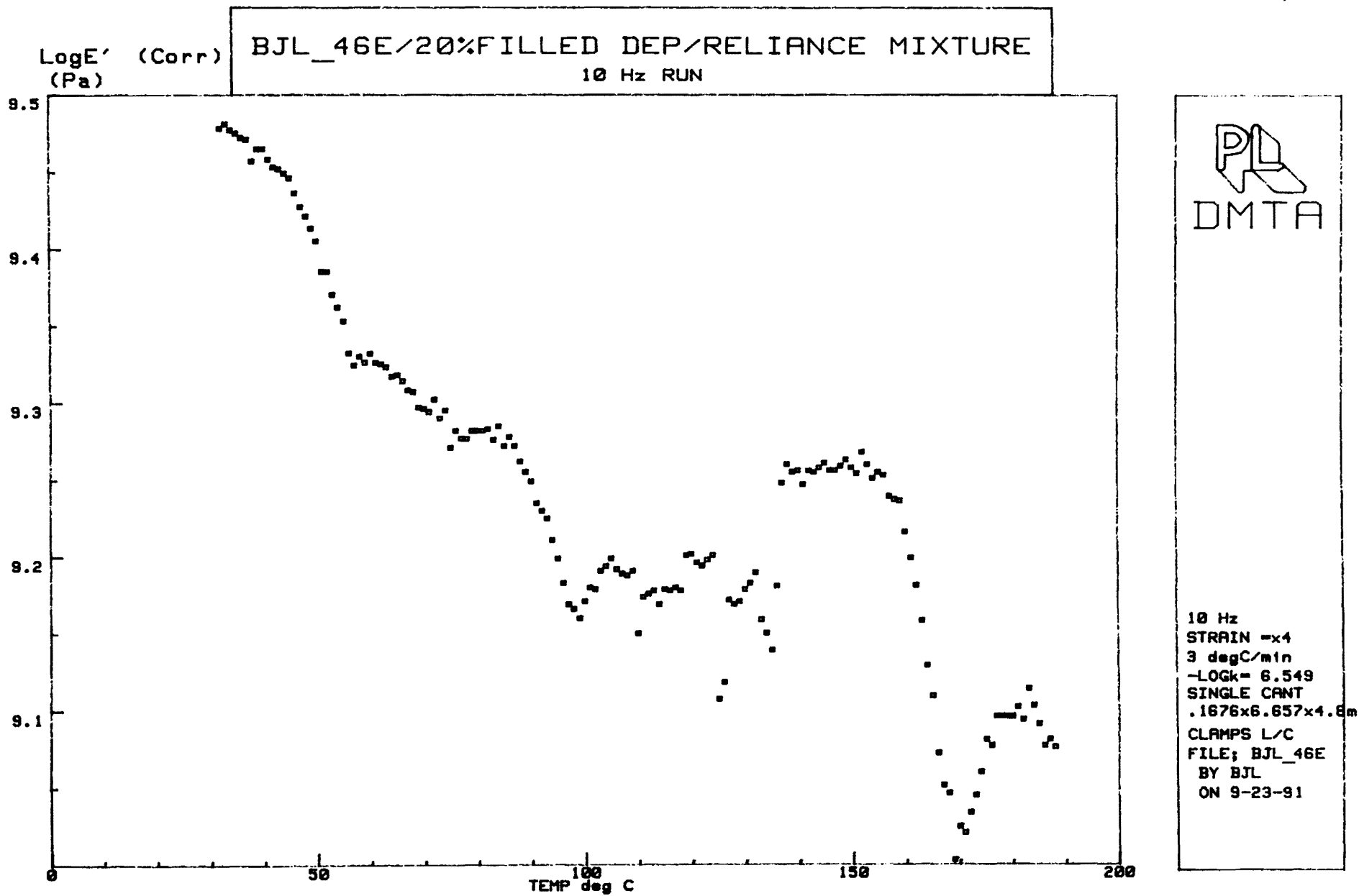


Figure 6. Typical shear storage modulus graph for the 20% plasticized adhesive from 30 to 200°C at 10 Hz.

CLINICAL EFFECTIVENESS OF FLUORIDE-RELEASING ELASTOMERS
II: Enamel microhardness levels

Thomas G. Wilson, D.D.S.
Resident, Department of Orthodontics
Emory University
School of Postgraduate Dentistry

Brian Love, Ph.D.
School of Materials Engineering
Georgia Institute of Technology

Charles H. Smith, D.D.S.
Chairman, Department of Orthodontics
Emory University
School of Postgraduate Dentistry

To whom all correspondence should be addressed:
Dr. Thomas G. Wilson
8515 Douglas
Omega Place, Suite 26
Des Moines, Iowa 50322
(515) 278-2333

RUNNING HEAD: Effects of Fluoride Elastomers II

ABSTRACT

The purpose of this study was to examine the effect of fluoride-releasing elastomers on enamel microhardness levels. Sixteen teeth from four patients scheduled to have premolars extracted as part of their orthodontic treatment were examined in this study. Orthodontic brackets were bonded to the buccal surface of the test teeth with a non-fluoridated adhesive. Two of the patients had fluoride-releasing elastomers placed on the right upper and lower brackets and conventional elastomers placed on the left side. This sequence was reversed for the remaining two patients. After one calendar month, the experimental teeth were extracted, sectioned and embedded in acrylic. Microhardness tests were performed 50 to 75 μ m cervical to the bracket. Indentations were taken at the surface and continued in 20 μ m increments to a depth of 200 μ m. Results showed the enamel was significantly harder ($p \leq 0.05$) in the fluoride group at the 20 μ m depth compared to the control group. No other microhardness readings showed a statistically significant difference.

INTRODUCTION

Part I of this study reviewed the etiology of decalcification surrounding fixed orthodontic appliances.¹ It also investigated the effect fluoride releasing elastomers had on *Streptococcus mutans* numbers. Part II of this study will evaluate the demineralization of enamel surrounding orthodontic brackets which contained fluoride releasing elastomers.

Scanning electron microscopy has demonstrated that bacterial accumulation around orthodontic bands with an open margin showed a localized demineralization of the enamel under the plaque after only one week.² With increased bacterial exposure more decalcification was noted.³

There are many studies suggesting that small areas of surface enamel demineralization may be remineralized or rehardened.⁴⁻⁶ Another investigation has shown that small quantities of fluoride can increase the degree of remineralization by reacting with hydroxyapatite crystals in enamel.⁷ The fluoride ions substitute themselves for the hydroxyl ions, forming fluorapatite. This chemical substitution forms a surface layer which is more resistant to acid dissolution by promoting remineralization, increasing crystallinity and decreasing solubility of the enamel. The role of fluoride as a suppressor of demineralization has been investigated.⁸ It has been shown that the presence of fluoride in solution at the time of acidic attack on the enamel may minimize the rate of demineralization. This study examined the microhardness of enamel surrounding orthodontic brackets with conventional and fluoride releasing elastomers.

MATERIALS AND METHODS

Sixteen teeth from four patients scheduled to have premolars extracted as part of their orthodontic treatment were examined in this study. Individual teeth rather than individual patients were selected for the sampling unit, based on previous research on the variability of enamel microhardness. Purdell-Lewis⁹ found that hardness of sound enamel can vary from buccal to lingual and occlusal to gingival on the same tooth. The experimental teeth were examined clinically to verify that they were free of white spot lesions or significant enamel defects. The teeth were then cleaned with a pumice slurry using a rubber cup on a slow speed handpiece. Orthodontic brackets were bonded to the buccal surfaces at a distance of 4mm from the cusp tip to the middle of the bracket slot. A non-fluoridated chemical curing acid-etch adhesive system was used to bond the brackets to the teeth. Reasonable care was taken during the etching procedure to ensure that only the area where the bracket would be placed would be etched and sealed. This was done to avoid the influence of the etching procedure on the enamel adjacent to the bracket.¹⁰ Two of the patients had fluoride-releasing elastomers placed on the right upper and lower brackets and conventional elastomers placed on the left side. The remaining two patients had fluoride-releasing elastomers placed on their left side and conventional elastomers on their right side. No attempts were made to alter the patient's current oral hygiene regimen or to standardize the patients for right or left handedness.

After one calendar month, the teeth for this study were extracted. Care was taken during extraction to ensure that the brackets were not removed from the teeth. Teeth were then sectioned buccolingually through the brackets with a diamond disk. Samples were then embedded in self-curing acrylic[¥] with cut faces exposed and sequentially polished.

Research by O'Reilly and Featherstone¹¹ found that the area of greatest demineralization occurs 50 to 75 μ m cervical to the bracket. With this in mind, microhardness tests were performed perpendicular to the cut face of the enamel, 50 to 75 μ m cervical to the bracket. Indentations were made using a Leitz microhardness tester[§] according to the method of Purdell-Lewis, Groeneveld and Arends.⁹ Indentations were taken at the surface and again in 20 μ m increments to a depth of 200 μ m. At each depth the mean Knoop hardness number (KHN) for the teeth with fluoride-releasing elastomers was compared to that for the teeth with conventional elastomers using the Student's T test.

[¥] Dentsply International Inc., Milford DE.

[§] Leitz Inc., New York, NY.

RESULTS

Table 1 shows the mean enamel microhardness from the control and fluoride groups. The enamel was significantly harder ($p \leq 0.05$) in the fluoride group at the 20 μ m depth compared to the control group. No other microhardness readings showed a statistically significant difference. However, there was a non-significant trend towards increased enamel hardness at increasing depths into the enamel for both groups.

DISCUSSION

The change in microhardness found on the surface of the fluoride group may be related to the findings in part 1 of this study.¹ This earlier work found that patients with fluoride-releasing elastomers had a temporary decrease in *Streptococcus mutans* levels. The cariogenic and demineralizing properties of *Strep mutans* are well known. It would follow that a reduction in this bacterium would lead to a decrease in the demineralization surrounding orthodontic brackets.

Another possible explanation for the lack of demineralization in the fluoride group is that the fluoride elastomer functioned as a fluoride reservoir. Recent research has shown that fluoride ions diffuse from areas of high concentration into areas of demineralization¹² This area of hyper-fluorosed enamel may also form a barrier during acid attacks.¹³

An interesting finding from this study was the non-significant trend towards decreased microhardness in the control group at subsurface depths. Previous research has shown that natural caries demonstrates a demineralized subsurface area.¹⁴⁻¹⁶ This trend toward softer enamel may have indicated an early subsurface lesion.

Further research is needed to gauge how long the increased microhardness surrounding each fluoride releasing elastomer lasts. Whether this protection will result in fewer clinically visible white spot lesions following orthodontic treatment remains unknown.

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Reprint requests to:
Dr. Thomas G. Wilson
8515 Douglas
Omega Place, Suite 26
Des Moines, IA 50322

FIGURE LEGENDS

Fig. 1. Comparison of mean enamel microhardness levels from the 8 control and 8 experimental teeth.

Fig. 2. Appearance under scanning electron microscope of test tooth embedded in acrylic. Indentations at 20 μ m intervals starting at the surface and continuing to a depth of 200 μ m are visible.

Table 1. Difference in mean enamel Knoop microhardness levels of control and experimental teeth in Kg/mm².

Table 1.

Depth	Fluoride Group	Control Group	Significance of difference between fluoride group and control group (p < 0.05)
20 μm	296.8 \pm 29.8	262.9 \pm 26.4	p < 0.05
40 μm	306.7 \pm 26.7	290.3 \pm 28.5	NS
60 μm	315.7 \pm 26.7	288.3 \pm 25.6	NS
80 μm	319.3 \pm 25.5	310.1 \pm 24.8	NS
100 μm	319.0 \pm 12.9	313.0 \pm 23.5	NS
120 μm	323.1 \pm 41.6	317.3 \pm 18.8	NS
140 μm	325.3 \pm 31.4	319.4 \pm 25.4	NS
160 μm	327.5 \pm 27.4	327.1 \pm 40.2	NS
180 μm	329.2 \pm 41.6	324.7 \pm 36.6	NS
200 μm	325.1 \pm 36.3	326.7 \pm 36.4	NS

Mean values \pm SD are shown

Figure 2.

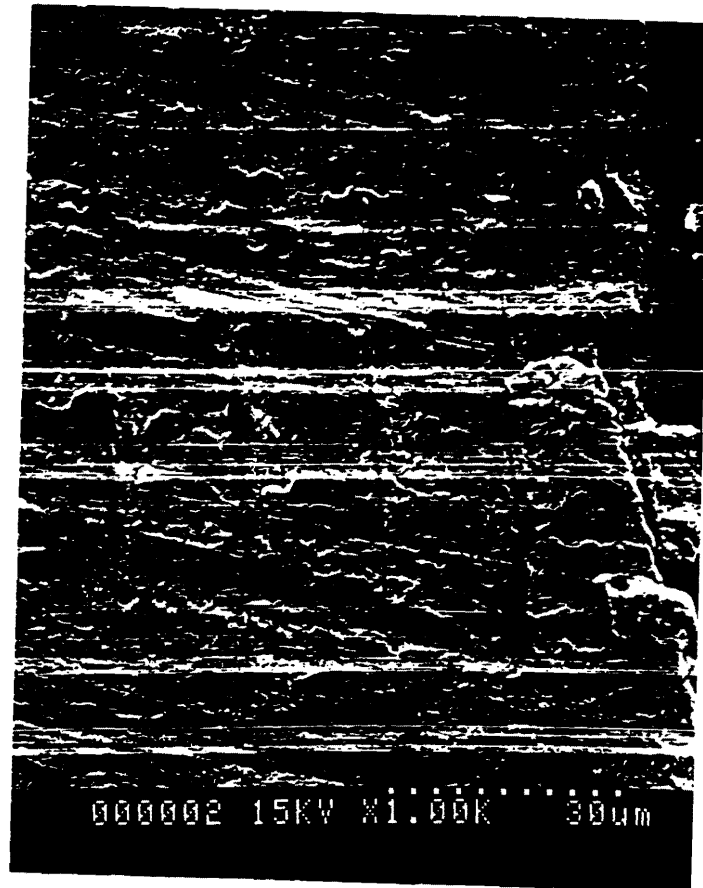
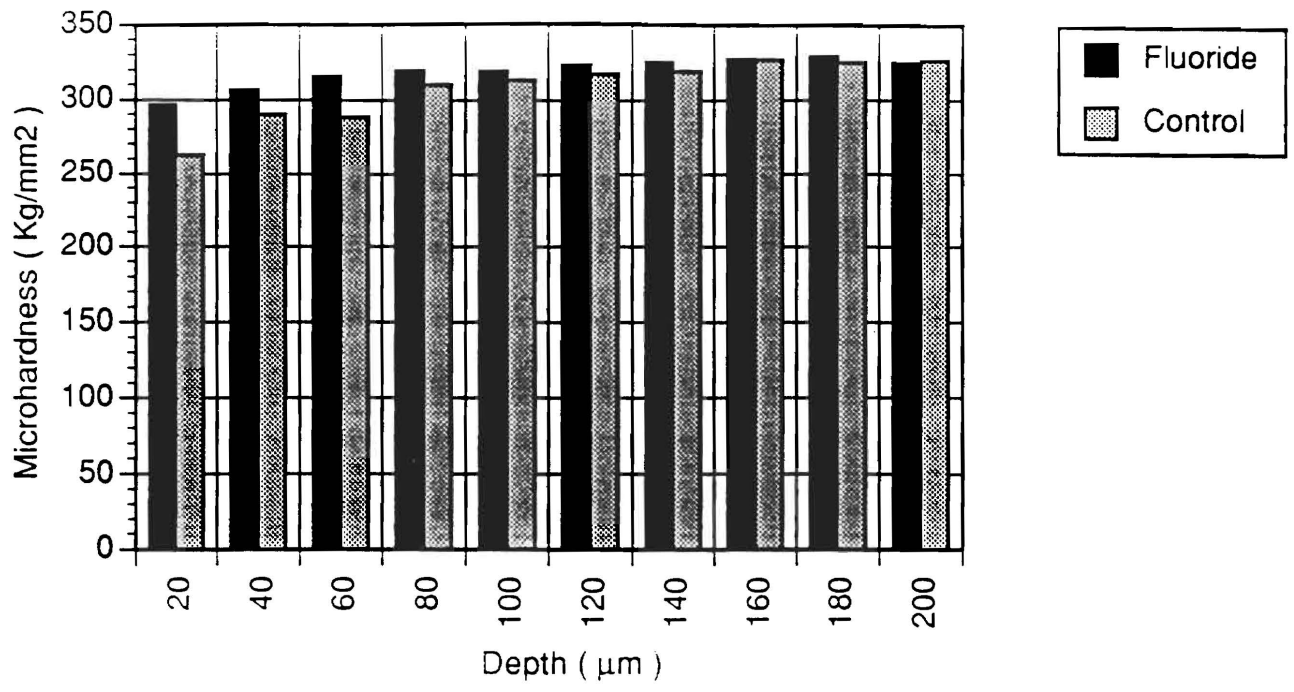


Figure 1.



Controlling Adhesion of Orthodontic Adhesives Through
Adjustment of The Interphase Mechanical Properties

B. J. Love*

School of Materials Science and Engineering
Georgia Institute of Technology, Atlanta, GA 30332-0245

K. E. Starling, Jr.

Department of Orthodontics
Emory University, Atlanta, GA 30033

T. V. Baughn

Texas Instruments
Defense Systems and Electronics Group
P. O. Box 655474 MS 213, Dallas, TX 75265

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Abstract:

Adhesives for ceramic orthodontic brackets are so strong that instances of enamel fracture and bracket fracture have occurred during removal. Our approach minimize the potential damage to the enamel was to adjust the mechanical properties of the adhesive, a BIS GMA-silica composite, by use of diethyl phthalate which is a common plasticizer. The plasticizer used in amounts up to 20% of the adhesive weight significantly decreases the adhesive modulus and tensile strength. One objective of this research is to evaluate plasticizer stability in the adhesive via functional testing in a simulated oral environment. A second objective was to simulate clinical loading conditions during orthodontic treatment and removal by use of finite element analysis. The finite element analysis determined the changes in computed stresses due to plasticization. After 25 days in an artificial saliva solution held at 60°C, the bracket removal torque was lower in the 10% plasticized adhesive group than that for the non-plasticized group. The 3-D linear elastic finite element analysis found that plasticization should not lead to premature failure when typical treatment loadings were applied. The torsional loading conditions simulating bracket removal reported peak stresses in excess of the plasticized adhesive tensile strength in the corner regions. Thus, modelling of the adhesive as a layer with distinct mechanical properties appears reasonable.

* To whom correspondences should be sent

Key Word Index: Dental Materials, BIS GMA, Diethyl Phthalate (DEP), Modulus, Torsional Adhesion, Plasticizer, Orthodontics, Ceramic Brackets, Bonding

Introduction

Single crystal and polycrystalline ceramic orthodontic brackets have improved the aesthetics during treatment¹. While ceramic brackets have not completely replaced stainless steel brackets, they are becoming widely used. When ceramic brackets are bonded to brittle tooth enamel using heavily ceramic filled adhesives, the joint is extremely strong and brittle which is desirable during treatment but leads to problems at the end of the treatment period. Among the biggest problems has been the difficulty in removing ceramic brackets after the treatment period is complete¹⁻³. Clinically measured increases in the removal force has been documented over that required to remove stainless steel brackets². As a result, clinical problems have included both bracket and enamel fractures.

There have been a series of published efforts recently to make bracket removal easier and more predictable. They have included heating the resin⁴⁻⁷, removing the silica reinforcement⁸ and introducing modifiers like plasticizers^{9,10}. All of these have shown varying degrees of success and also have their own unique drawbacks.

For example, heating the bis glycidyl methacrylate [BIS GMA with a glass transition temperature above 100°C] resin will definitely reduce the stiffness. Unfortunately, the resin retains mechanical rigidity well above 60°C, considered to be an upper limit before tooth pulp damage occurs. Removal of ceramic filler will also reduce the modulus of the adhesive, but clinically, orthodontists find that without the filler, the resin is more difficult

to use. Our efforts to reduce the stiffness of the adhesive have been to treat the adhesive as a separate layer with its own distinct mechanical properties between the adherends and reduce its stiffness by adding plasticizers. Significant reductions in elastic modulus and torsional adhesion have been shown^{9,10}. However, questions remain whether leaching of plasticizer by saliva will reduce the benefit of the plasticizer additions and how much plasticizer can be added to improve the removal characteristics without compromising the adhesive strength of the bonded bracket during treatment.

In previous work^{9,10}, we have demonstrated statistically significant reductions in the elastic modulus of a 75% silica filled BIS-GMA composite [from 12.0 GPa without any plasticizer to below 3.0 GPa adding up to 20% of the adhesive weight with diethyl phthalate (DEP) as measured at 10 Hz with dynamic mechanical spectroscopy]. In addition, when brackets were bonded to a standard stainless steel wire mesh substrate [made from Rocky Mountain Orthodontics, (RMO) Denver CO], statistically significant reductions in the torsional strength were observed due to the plasticizer additions. Maximum torsion values ranged from 0.20 N-m (1.76 in-lbs) for the non-plasticized adhesive to 0.07 N-m (0.66 in-lbs) when additions of up to 20% DEP by weight were made.

Conceptually, the mechanical properties of a particulate reinforced composite can be described by rule of mixtures as for example the elastic modulus¹¹,

$$E^n_{\text{composite}} = (X) E^n_{\text{polymer}} + (1-X) E^n_{\text{reinforcement}} \quad 1$$

where X is the weight fraction of polymer, E_{polymer} and $E_{\text{reinforcement}}$ are the elastic moduli of the polymer and reinforcement materials, and n is an exponent ranging from -1 to 1 between the isostrain and the isostress cases respectively. The exponent, n , is usually near 0 for particulate composites¹¹. Plasticizers can reduce the elastic modulus of the polymer, E_{polymer} . Therefore, if the polymer in a reinforced composite adhesive resin can be plasticized, the reductions in the adhesive modulus, $E_{\text{composite}}$, will be due to polymer-plasticizer interactions. E_{polymer} is temperature dependent due to the free volume dependence on temperature. Plasticizers should not interact with the ceramic reinforcement.

This paper summarizes our efforts to modify the mechanical properties of the bulk adhesive and to alter the resulting adhesion. In particular, the question of how much the adhesive stiffness can be reduced while remaining viable as an adhesive during orthodontic treatment and allowing for easier removal is considered. The FE model treats the adhesive zone as a separate layer with its own distinct properties. The results of salivary exposure studies are presented to determine the effectiveness of plasticization by exposure to an artificial saliva solution outlined by Marek et al¹² at two temperatures.

Experimental:

Methods and Materials:

The adhesive samples were based on a commercial orthodontic resin system (Reliance

Orthodontics Phase II, Itasca, IL). This is a two part, silica filled acrylic resin using a peroxide curing agent and a tertiary amine activator. The filler content is nominally 75% of the adhesive by weight. Samples for mechanical property testing were fabricated by mixing the prescribed amount of plasticizer with equal parts of the two part adhesive system. The mixture was then deposited into casting plates to make cured samples of the modified adhesive resin for dynamic mechanical property and tensile strength testing. Modulus measurements were taken at various temperatures but reported at 30°C using a Polymer Labs DMTA equipped with a shear head in a single cantilever mode at 10 Hz while applying the appropriate end correction factor. The modulus measurements were taken as baseline values for the finite element modelling and analysis. The tensile strength measurements were taken using an Instron TT Machine set at 0.127 cm/minute and were used for comparison to the stresses computed in the finite element model. At least 4 samples were used for the tensile strength measurements and at least 3 samples were used for the modulus measurements at each condition. Standard deviations were computed based on a student t statistical analysis. Adhesion tests were performed using the procedure below.

Groups of at least 5 orthodontic brackets for each plasticizer condition (RMO Denver, "Signature" Brackets) were bonded to a standard stainless steel wire mesh bonding disk made by RMO and used generally throughout the industry. The disk was prepared by first applying a sealant layer of unfilled resin to the disk. The thin sealant layer was a two part acrylic resin with no filler. After applying the sealant layer set (5-10 minutes), the 2 part silica filled adhesive with desired amounts of DEP were mixed together by hand and a small amount was

applied to each bracket, then the bracket was affixed to the bonding substrate. Again, all of the adhesives were modified from the original commercially available orthodontic resin. Care was taken to remove excess adhesive from around the edges of the bracket without dislodging the bracket as the adhesive set. This reasonably approximates what is performed by the clinician.

After the brackets were fixtured to each disk, salivary exposure studies were performed using an artificial saliva solution to determine how bond strength is affected by the interaction with saliva. The saliva solution was based on a composition originally outlined by Marek *et al.*¹² and exposures were performed at both room temperature and at 60°C. After periodic exposure times, sample disks were removed, gross water removed, and adhesion tested in torque using a Sturtevant 0.569 N-m (5 in-lb) measurement device. Measurements of the maximum torque required for removal were recorded. Again, standard deviations were computed based on a student t distribution. Ideally, measurements of the plasticizer extracted by the saliva solution would have been made, but there was such a small amount of plasticizer used for the bond tests, extraction was not a viable option. Only an indirect approach like measuring adhesion before and after exposure was thought to be a reasonable approach.

Finite Element Modelling:

A 3-D linear elastic finite element model was constructed for the orthodontic bracket

and adhesive. The bracket was modelled to provide a mechanism to introduce load into the adhesive. The stiffness of the sapphire bracket is large compared to that of the adhesive and the adhesive will encounter the most deformation. The curvature and the microporous topography of the tooth were not modelled. Although the bracket and adhesive layer have geometric symmetry, a full model of the bracket and adhesive were assembled in anticipation of introducing non-symmetric tooth curvature in future studies. The interface between the tooth and the adhesive was represented as a rigid interface. The mechanical properties introduced into the model were generated by the measurements done in the first part of this effort.

A boundary representation of the finite element model and the loading conditions for the two cases are shown in Figure 1. The adhesive pad is 2.65 mm wide and 3.54 mm long with a uniform adhesive thickness of 0.25 mm. The lower section of the model is the adhesive section and the upper section is the bracket. All materials were modelled with 20 node brick elements which are not shown. The bracket has 1200 elements total with 2 layers through the thickness. All loads were applied to the bracket section.

The stresses were computed using the finite element program ABAQUS¹³. Two distinct load cases were examined. The top illustration in Figure 1 shows the shear loading of 3.92 N applied as a uniform force across the surface of the bracket. The shear load is applied in the +Y direction and simulates the insertion and loading of the nickel/titanium wire in the bracket groove. The shear force is indicative of forces typically used in

orthodontic treatment. The second loading condition shown in the lower illustration of Figure 1 represents a 0.169 N-m torque acting about the global Z axis. The torque value is typical of bracket removal torques⁹. The von Mises stress in the adhesive was determined for both loading conditions in the unplasticized and 10% plasticized condition.

Results and Discussion:

Mechanical Property Measurements:

A summary of the in phase dynamic mechanical modulus measurements, E' , and the tensile strengths is shown in Table 1. The mechanical properties of the non-plasticized and 10% plasticized adhesive resins were used in comparing the finite element model. Significant reductions in both modulus and tensile strength occur with increasing plasticization. There should be a significant swelling effect due to polymer solvent interaction since DEP has nearly the same solubility parameter as methyl methacrylate, the main monomer segment. The plasticized polymer should also have decreased resistance to segmental motion and hence a lower modulus. One important result of this work is that the mechanical properties of the composite resin can be affected through plasticizer interactions with one of its constituents.

The modulus measurements from the dynamic mechanical measurements were used in the modelling work at two conditions, 0% plasticizer and 10% diethyl phthalate by weight.

One major concern is how long the decreased mechanical properties are retained. This aspect is addressed by the exposure studies in the artificial saliva solution.

Salivary Exposure Results:

The salivary exposure results are shown at room temperature in Table 2 and at elevated temperature in Table 3. All bond failures occurred either at the bracket adhesive interface or within the adhesive. At room temperature, the torsional removal force using the non-plasticized adhesive samples remains statistically higher than for the 10% plasticized sample group at 0 and at 30 days. While the general trend for all conditions is toward lower torsional strength at increased plasticizer content, the results for the 5% plasticized adhesive group were not considered to be statistically lower than for the non-plasticized group. There is no statistically significant increase over time in the torsional strength either for the 5% or for the 10% plasticized groups. An increase would have been indicative of salivary leaching of plasticizer.

At 60°C, more salivary leaching of the plasticizer from the adhesive would be expected. The torsional adhesion results for the 5% and 10% plasticized samples indicate no statistically significant change over time in the artificial saliva solution. The torsional adhesion results for the plasticized adhesives are not statistically different from their corresponding room temperature exposure values in the artificial saliva solution. There is an interaction between the non-plasticized adhesive and the saliva as measured by a

reduction in the torsional adhesion with time in the artificial saliva solution. This result is consistent with work by Beatty *et al.* who found moisture absorption in unfilled resin systems which reduced the overall hardness¹⁵. We were surprised to not see a similar effect with the plasticized adhesives.

There are several potential mechanisms for the lower adhesion at long times in the artificial saliva solution. The first is that the plasticizer interacts with the polymer to increase free volume, decrease the elastic modulus, and reduce the resultant adhesion. This is the case before the samples are exposed to the artificial saliva solution⁹. After salivary exposure, three mechanisms of reduced adhesion are possible. If there is no interaction between saliva and the plasticized adhesive, a continued reduction in adhesion is expected. Of course this indirect approach does not rule out the possibility of an exchange of saliva for plasticizer given that they may both have some affinity for the resin. There is also a chance that plasticizer extraction by saliva would create voids where extraction occurred in the bulk of the adhesive, lowering the total bond area and resulting in continued lower adhesion even though some plasticizer is extracted.

Nevertheless, lower adhesion from plasticization is more of a permanent effect even after exposure to an artificial saliva solution. Over the time of our experimentation, there was no change in adhesion using the plasticized adhesives that could be attributed to leached plasticizer as a result of exposure to the artificial saliva solution.

Finite Element Results:

Results from the FEA indicate that treatment of the adhesive as a separate and distinct layer with its own mechanical properties is satisfactory for describing the strength behavior of these bracket interfaces. The results of the simulation with 3.92 N orthodontic "treatment force" corresponds to very low computed stress values within the adhesive layer. The peaks of maximum stress are 0.84 N/mm² for the 10% plasticized adhesive and 0.93 N/mm² for the non-plasticized adhesive. A grey scale color contour plot for the computed stress state of the plasticized adhesive under this simulated loading condition is shown in Figure 2. Conceptually, the results are similar for the unplasticized case. These results indicate that a 10% DEP plasticized adhesive should survive the orthodontic loadings and should not result in premature failure during treatment.

Even more interesting are the modelling results when simulated torque loads nearly equal to the experimental failure torques are entered into the model. These results are shown in the grey scale contour plot in Figure 3 for the plasticized adhesive. The computed peak stress generated at the corners of the adhesive from the application of a 0.169 N-m (1.5 in-lb) loading is 22.8 N/mm² (3300 PSI) for the non-plasticized adhesive case and 20.3 N/mm² (2950 PSI) for the 10% plasticized adhesive case. Noting the reported tensile strengths for each adhesive (ultimate tensile strength = 18.6 N/mm² for 10% plasticized, 42.9 N/mm² for non-plasticized⁹), the modelling predicts cohesive failure within the adhesive when the torsion conditions are applied to the 10% plasticized adhesive. If the goal of adhesive modification

is to make bracket removal easier/more predictable, then lowering the adhesive tensile strength to stress levels that are achievable during removal appears reasonable. Treatment of the adhesive as an interphase region with its own distinct and separate mechanical properties is reasonable from the finite element modelling and stress analyses.

Other factors need consideration. Tooth curvature and bracket curvature have not been included in our analytical model. In addition, the sealant layer is modelled as filled resin. Thus, the true adhesive zone will not be completely reinforced throughout the adhesive layer as has been modelled. In addition, the elastic modulus and tensile strength values for the adhesive would be more accurate using samples which were exposed to an oral environment. Also, there may be stress concentrators in our adhesion construction due to the wires in the wire mesh bonding disk which could be initiator points for failure. Finally, concerns about plastic deformation suggests that the 3-D linear elastic model might be inappropriate given the computed stresses. Nevertheless, the results are very much in line with what is experimentally observed.

Conclusions:

The following conclusions can be made as a result of these efforts:

- 1.) The modulus and tensile strength of these adhesive resins are significantly affected by the addition of DEP. These properties may only require slight modification since the adhesive

is still required to withstand loading conditions during treatment and normal chewing forces. Thus, the highest amounts of plasticization may be undesirable for clinical use.

2.) Salivary leaching leading to higher observed removal forces after treatment is not a major concern regarding this adhesive. Torsional adhesive strength measurements on the 10% plasticized samples are significantly lower than for the unplasticized samples even after exposure in the artificial saliva for 25 days at 60°C. More scatter is apparent in the 5% plasticized samples, however it appears that plasticization leading to lower adhesive strength is a permanent effect.

3.) The finite element analysis has shown that typical stresses calculated for the simulated treatment conditions should not lead to premature debonding with the 10% DEP plasticized resins. The computed treatment stresses by our finite element model are between 0.8 and 1.0 N/mm², far below the measured tensile strength for either the non-plasticized or 10% plasticized adhesives.

4.) The treatment of the adhesive layer as an interphase region with mechanical properties of its own is reasonable. If the mechanical properties of the 10% plasticized adhesive are inserted into the FE model for the simulated torsional removal conditions, the computed stresses in the corners exceed the adhesive tensile strength where cohesive fracture was experimentally found. The peak stresses under the same conditions for the unplasticized adhesive are still well below the unplasticized adhesive tensile strength.

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Table 1: Mechanical Property Measurements vs. Plasticizer Content

Sample (% DEP)	In Phase Dynamic Modulus, E' (GPa)	1 SD (GPa)	Tensile Strength N/mm ² (KSI)
0*	12.9	0.2	42.9 (6.2)
5	7.6	0.1	
10*	7.1	0.5	18.6 (2.7)
15	3.6	0.7	
20	3.0	1.9	

*: Conditions inputted into the ABAQUS FEM Program

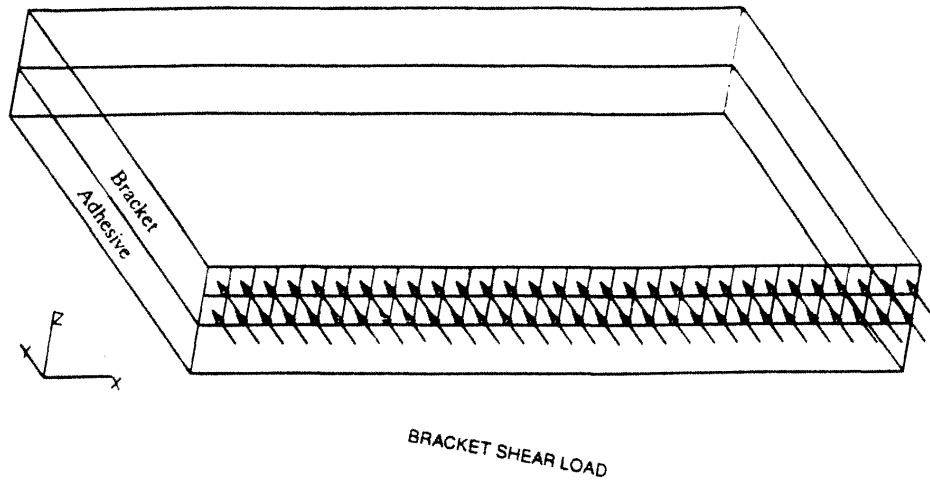
Torsional debond strength (N-m \pm 1S.D.)			
Days (at room temp.)	% Plasticizer		
	0	5	10
0	0.20 \pm 0.02	0.15 \pm 0.05	0.10 \pm 0.02
15	0.16 \pm 0.05	0.15 \pm 0.08	0.11 \pm 0.05
30	0.21 \pm 0.04	0.17 \pm 0.06	0.09 \pm 0.01

Table 2: Torsional Debond Strength as a function of time in the Synthetic Saliva Solution (Room Temperature)

Torsional debond strength (N-m \pm 1S.D.)			
Days (at 60°C)	% Plasticizer		
	0	5	10
0	0.20 \pm 0.02	0.15 \pm 0.05	0.10 \pm 0.02
15	0.13 \pm 0.03	0.13 \pm 0.03	0.09 \pm 0.01
20		0.16 \pm 0.03	0.08 \pm 0.02
25		0.13 \pm 0.05	0.08 \pm 0.01

Table 3: Torsional Debond Strength as a Function of Time in the Synthetic Saliva Solution (60°C)

a)



b)

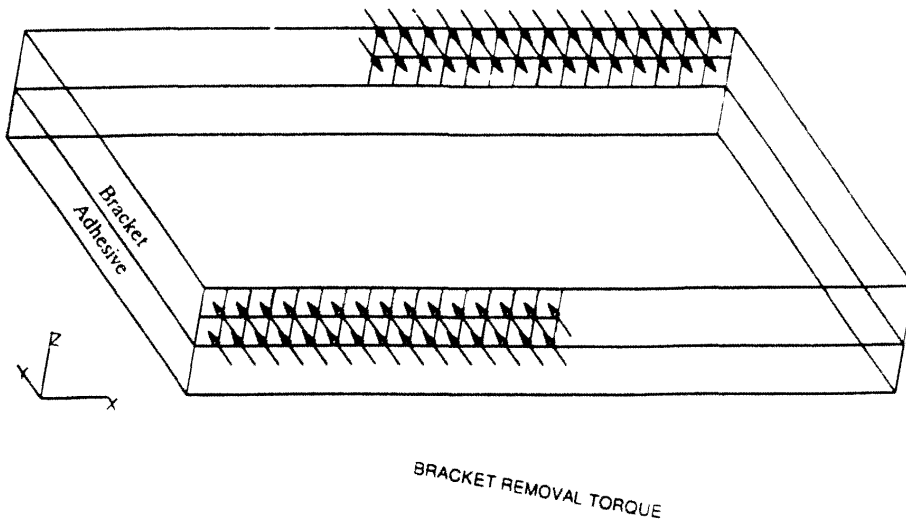
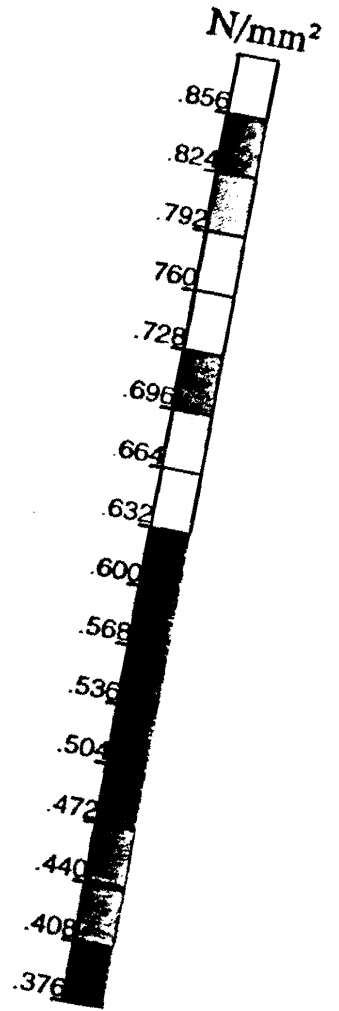
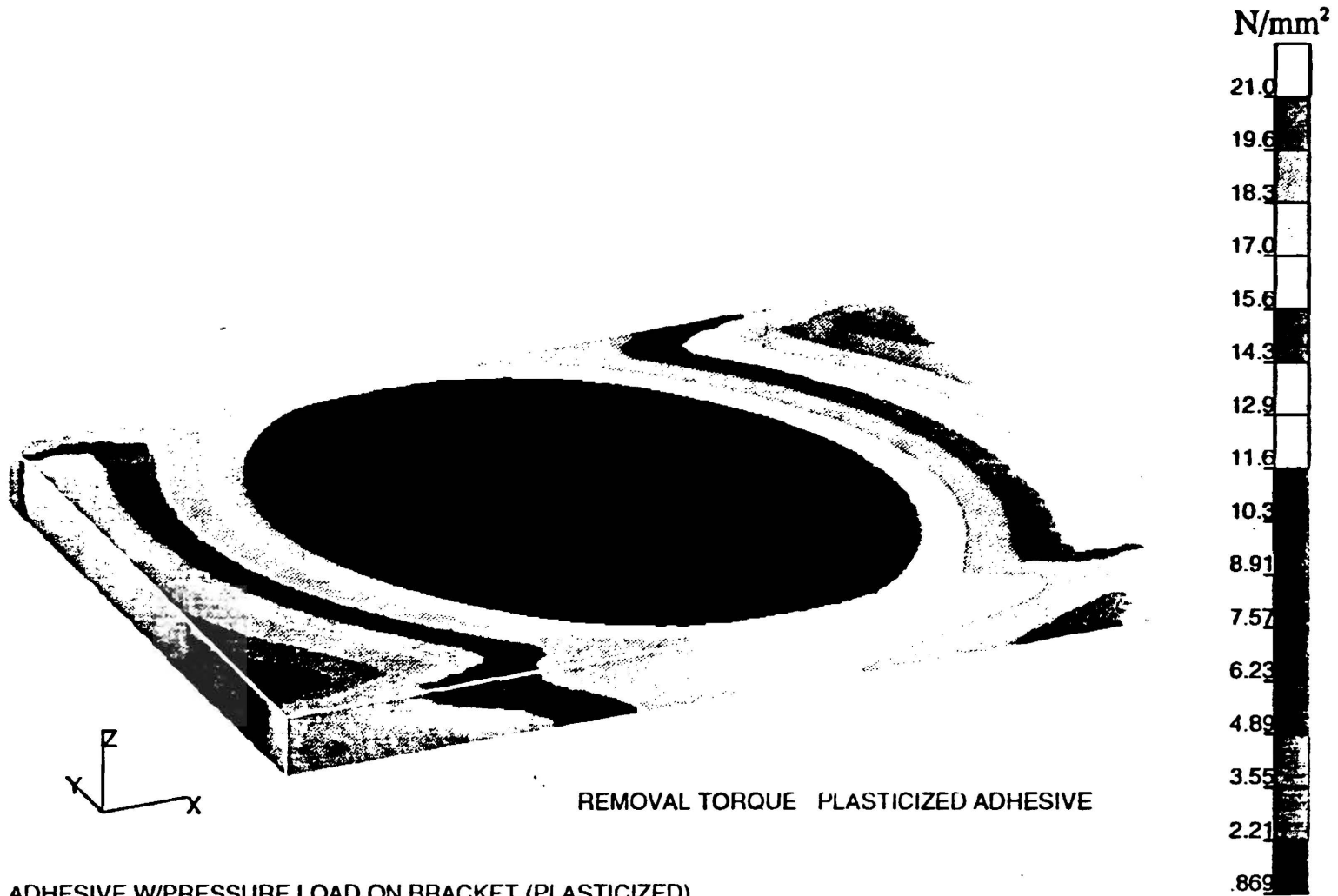


Figura 1



DENTAL ADHESIVE W/PRESSURE LOAD ON BRACKET (PLASTICIZED)
 ABAQUS V4.9-1 1-APR-93 07:33:22 3600 17213
 PROCEDURE 2 TIME STEP 1 INCREMENT 1
 SHEAR LOAD PLASTICIZED ADHESIVE



DENTAL ADHESIVE W/PRESSURE LOAD ON BRACKET (PLASTICIZED)
 ABAQUS V4-9-1 1-APR-93 07:33:22 3600 17213
 PROCEDURE 2 TIME STEP 2 INCREMENT 1

REPASSIVATION KINETICS OF DENTAL AMALGAMS

APPROVED:


Dr. Miroslav Marek, Chairman


Dr. Robert Hochman


Dr. W. Brent Carter

Date Approved by Chairperson 11-18-92

Repassivation Kinetics of Dental Amalgam

**A THESIS
Presented to
The Academic Faculty**

by

Christine Marie Lance

**In Partial Fulfillment
of the Requirements for the Degree
Master of Science in Metallurgy**

**Georgia Institute of Technology
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CHAPTER I

INTRODUCTION

Dental amalgam is a widely used material for restoring dental structure lost due to caries. Dental amalgam is a direct filling material; it is easily prepared, relatively cheap, and shows an adequate service life. However, because dental amalgam contains mercury, there is concern regarding mercury release into the human body.

When exposed to air or oral fluids, dental amalgam becomes covered with a protective oxide film; this film has been reported to inhibit mercury release (1-4). When this protective film is abraded by brushing or chewing, dissolution of mercury is significantly faster on the bare metal surface than on the film-covered surface (5). When the abrasive action ceases the amalgam surface repassivates. Therefore, repassivation kinetics may determine how much corrosion and mercury release occurs *in vivo* both during and after abrasion of the amalgam surface.

In this study, repassivation kinetics were determined for two major types of dental amalgams. The film-free surface was generated either by electrochemical depassivation, or by fracture of amalgam specimens. The kinetics of surface changes were examined using electrochemical techniques.