REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE May 1995	3. REPORT TYPE AND DATES COVERED

4. TITLE AND SUBTITLE
An Investigation of THE Bond Strength of Dental Porcelain Fused TO TITANium

5. FUNDING NUMBERS

6. AUTHOR(S)
Jose M. Gutierrez III

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
AFIT Students Attending:
The University of TEXAS - Graduate School of Biomedical Sciences ; San Antonio TX

8. PERFORMING ORGANIZATION REPORT NUMBER
AFIT/CI/CIA
95-045

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
DEPARTNEMT OF THE AIR FORCE
AFIT/CI
2950 P STREET, BDLG 125
WRIGHT-PATTERSON AFB OH 45433-7765

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for Public Release IAW AFR 190-1
Distribution Unlimited
BRIAN D. GAUTHIER, MSgt, USAF
Chief Administration

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

DISTRIBUTION STATEMENT A
Approved for public release
Distribution Unlimited

DTIC
ELECTE
JUL 19 1995
B

DTIC QUALITY INSPECTED 1

14. SUBJECT TERMS	15. NUMBER OF PAGES 98
	16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18

AN INVESTIGATION OF THE BOND STRENGTH OF DENTAL PORCELAIN FUSED TO TITANIUM

A

THESIS

Presented to the Faculty of
The University of Texas Graduate School of Biomedical Sciences
at San Antonio
in Partial Fulfillment
of the Requirements
for the Degree of
MASTER OF SCIENCE

By

Jose M. Gutierrez, III, B.A., D.D.S.

San Antonio, Texas

May, 1995

AN INVESTIGATION OF THE BOND STRENGTH OF DENTAL PORCELAIN FUSED TO TITANIUM

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DEDICATION

This thesis is dedicated to my wife, Lisa R. Burkett-Gutierrez; and, to my parents, Jose M. Gutierrez, Jr. and Faye C. Gutierrez.

ACKNOWLEDGMENTS

I would like to express my sincere appreciation to Dr. Ronald B. Blackman for serving as my research committee chairman. His guidance, instruction, editorial support, and encouragement were invaluable for the successful completion of this research.

I would like to thank Dr. Ramon J. Baez, Dr. John O. Burgess, Dr. David A. Chance, and Dr. Ronald G. Verrett for serving on my research committee. Their many contributions to include acquisition of essential materials and supplies, editorial review of the thesis, and overall supervision and encouragement were also invaluable for completion of this research.

I would like to give special thanks to Dr. Waldemar G. de Rijk for his significant contributions to this research which included providing the testing methodology, guidance, support, editorial review of the proposal and thesis, and performing a Weibull statistical analysis of the data.

I would like to thank Mr. James Mathis, Mr. Dan South, TSgt Nancy Kujak, SSgt Michelle Henry, and the staff of the MacKown Dental Laboratory, Lackland AFB, TX, for their laboratory support and assistance throughout this research.

I would like to thank Ms. Kathleen McKay, Clinical Investigation Directorate, Lackland AFB, TX, for her expertise and assistance with the Instron testing machine.

I would like to thank SSgt Ralph Pena, Dental Investigation Service, Brooks AFB, TX, for his assistance in acquiring essential materials.

AN INVESTIGATION OF THE BOND STRENGTH OF DENTAL PORCELAIN TO TITANIUM

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Biological concerns over existing restorative metal alloys have led researchers to study titanium due to its biocompatibility. The objective of this study was to determine bond strengths of two dental porcelains fused to a 99.6% pure titanium. Testing was accomplished using four-point bending of porcelain-fused-to-titanium (PFT) composite beams. For comparison, a commonly-used porcelain-fused-to-metal (PFM) system was tested in the same manner. A total of forty-five composite beam samples were made. The samples were divided as follows: Group 1 consisted of twenty samples of Duceratin porcelain fused to Rematitan metal; Group 2 consisted of twenty samples of Vita Titankeramik porcelain fused to Rematitan metal; and Group 3 consisted of five samples of Vita VMK-68 porcelain fused to Olympia gold alloy (control). Individual samples were placed in a four-point bending fixture and loaded under compression using an Instron Universal Testing Machine until fracture of the porcelain was recorded. The mean bond strength for Group 1 was 46.4 MPa, Group 2 was 36.5 MPa, and

Group 3 was 28.6 MPa. After experimentation, the fractured samples were refired through the glaze cycle and retested in the same manner to evaluate the effects on bond strength. Mean bond strength values were not obtained for Groups 1 and 2 due to bond failure of the samples after the refiring process; however, the mean bond strength value for Group 3 was 30.1 MPa. A Weibull distribution statistical analysis (90% Confidence) was performed. Results indicate: (1) the bond between Duceratin and Rematitan was the highest of the systems investigated, (2) the difference between Duceratin and Vita Titankeramik was statistically significant, (3) the bond strength value for control (PFM) was the lowest, but comparable with the other reported values for metal-ceramic systems, (4) glaze cycle refiring did not adversely affect the bond strength of the control group, and (5) both porcelain fused to titanium systems exhibited bond failure after the glaze cycle refiring process. This may be due to oxygen or oxide contamination of the metal at high temperatures. <u>The bond strength values for porcelain fused to pure titanium systems are greater than the bond strength values for a porcelain fused to gold alloy system.</u>

TABLE OF CONTENTS

page

Title i

Approval ii

Dedication iii

Acknowledgments iv

Abstract v

Table of Contents vii

List of Tables x

List of Figures xi

List of Plates xiii

I. INTRODUCTION 1

II. LITERATURE REVIEW 4

A. Background Information On Titanium Metal 4

B. Physical and Mechanical Properties of Titanium 6

C. Biologic Characteristics of Titanium 9

D. Dental Porcelain 10

1. Definition, Composition, and Properties 10

2. Porcelain Bonding To Metal 11

3. Classification Of Bond Failure 12

E. Dental Casting of Titanium 13

1. Heat and Titanium Characteristics 13

2. Casting Machines 15

3. Investments 17

4. Castability 19

5. Titanium Dental Porcelain 20

F. Metal-Ceramic Bond Strength Tests And Studies 24

1. Pull-Shear and Push-Shear Tests 24

2. Flexure Tests 28

3. Planar Shear Tests 36

4. Tensile Tests 39

5. Conical Interface Shear Test 39

6. Tension-Shear Test 41

7. Oblique-Shear Test 41

8. Torsion Tests 41

III. MATERIALS AND METHODS 47

A. Experimental Plan 47

B. Samples 48

C. Fabrication of Wax Pattern 48

D. Spruing and Investing 48

E. Burnout 52

F. Casting 52

G. Metal Finishing 54

H. Measurement of Metal Casting 54

I. Porcelain Application 55

J. Finishing of Samples 60

K. Measurement of Samples 60

L. Experimental Testing of Samples 60

M. Refiring and Retesting of Samples 67

N. Statistical Analysis 67

IV. RESULTS 71

V. DISCUSSION 83

VI. SUMMARY 90

Literature Cited 91

Vita 98

LIST OF TABLES

page

Table 1. Physical and Mechanical Properties 5

Table 2. Chemical Composition of Titanium - Volume % 8

Table 3. Duceratin Porcelain Firing Chart 56

Table 4. Vita Titankeramik Porcelain Firing Chart 58

Table 5. Vita VMK-68 Porcelain Firing Chart 59

Table 6. Duceratin / Rematitan Fracture Load Data 62

Table 7. Vita Titankeramik / Rematitan Fracture Load Data 63

Table 8. Vita VMK-68 / Olympia Fracture Load Data 64

Table 9. *Retested* Duceratin / Rematitan Fracture Load Data 68

Table 10. *Retested* Vita Titankeramik / Rematitan Fracture Load Data 69

Table 11. *Retested* Vita VMK-68 / Olympia Fracture Load Data 70

Table 12. Duceratin / Rematitan Bond Strength Values 72

Table 13. Vita Titankeramik / Rematitan Bond Strength Values 73

Table 14. Vita VMK-68 / Olympia Bond Strength Values. 74

Table 15. *Retested* Duceratin / Rematitan Bond Strength Values 75

Table 16. *Retested* Vita Titankeramik / Rematitan Bond Strength Values 76

Table 17. *Retested* Vita VMK-68 / Olympia Bond Strength Values 77

Table 18. Mean Bond Strength Values: All Dental Porcelain / Metal Systems 79

Table 19. Confidence Limits for the Weibull Parameters and the Modulus of Rupture at 90% Confidence 81

LIST OF FIGURES

page

Figure 1. Classification of Porcelain Enamel Fractures According to Interfaces Formed (O'Brien, 1977) 14

Figure 2. Pull-Shear Bond Test Design (from Anusavice, 1983) 25

Figure 3. Normalized Porcelain Shear Stress Along Porcelain-Metal Interface in Pull-Shear and Push-Shear Bond Test Specimens (Anusavice, 1980) .. 26

Figure 4. Normalized Shear Stress in Porcelain along Porcelain-Metal Interface (A-B) in Three-Point Flexure Bond Test Specimen (from Anusavice, 1983) 29

Figure 5. Normalized Shear Stress in Porcelain along Porcelain-Metal Interface (A-B) in Four-Point Flexure Bond Test Specimen (from Anusavice, 1983) 31

Figure 6. Variation of Normalized Maximum Tensile Stress, δn, with Thickness of Porcelain, b, with Constant Bending Moment, M (Tesk et al., 1982) 33

Figure 7. Four-Point Bending of a Specimen Glued to Steel Extension Bars. (Derand and Hero, 1992) 34

Figure 8. Semicircular Arch Flexure Bond Test (Mackert et al., 1976) [from Anusavice, 1983] 35

Figure 9. Planar Interface Shear Bond Test with Circular Interfacial Area (Civjan et al., 1974) [from Anusavice, 1983] 37

Figure 10. Planar Interface Shear Bond Test with Rectangular Area (Susz et al., 1980) [from Anusavice, 1983] 38

Figure 11. Conical Interface Shear Test (British Standard Institute Specification No. BS 3366) [from Anusavice, 1983] 40

Figure 12. Tension-Shear Bond Test (Wight et al., 1977) [from Anusavice, 1983] .. 42

Figure 13. Oblique-Shear Bond Test (Anusavice and Fairhurst, 1977) [from Anusavice, 1983] 43

Figure 14. Torsion Bond Test (Carter, 1975) [from Anusavice, 1983] 44

Figure 15. Sample Dimensions 49

Figure 16. Four-Point Bending Fixture Dimensions 66

Figure 17. Bond Strength Values: All Porcelain / Metal Systems 78

Figure 18. Mean Bond Strength Values: All Dental Porcelain / Metal Systems 80

Figure 19. Cumulative Probability of Failure in Four-Point Bend 82

LIST OF PLATES

page

Plate 1. Aluminum Mold and Wax Pattern 50

Plate 2. Spruing and Investing Wax Pattern 51

Plate 3. “Castmatic” Casting Machine 53

Plate 4. Finished Sample 61

Plate 5. Experimental Testing of Sample Using Four-Point Bending 65

I. INTRODUCTION

In the early part of the 18th century, Fauchard "the father of dentistry", proposed the use of porcelain for making artificial teeth. Since that time, work has never ceased on this fascinating material; and has lead to the highly sophisticated ceramics that are being used today (McLean, 1991). Over the past 25 years, the development of metal ceramic technology has lead to almost universal acceptance of metal-ceramic systems for the fabrication of fixed partial dentures (Campbell, 1989).

Biological concerns over existing restorative metal alloys have led researchers to study titanium as an alternate due to its biocompatibility. Titanium is a key metal used by high technology industries, as well as dentistry. Titanium plays a major role in the replacement of the root portion of a tooth; but, also in the replacement of the coronal portion of the tooth (Chance, 1992).

The recent development of porcelain systems matching the thermal coefficient of expansion of titanium, made it possible to construct porcelain-fused-to-titanium (PFT) restorations. To do this, laboratories must invest in and master titanium forming techniques. Some benefits of PFT crowns and fixed partial dentures are: (1) biological compatibility, i.e., hypoallergenic; (2) low cost compared to noble and base metals; (3) PFT restorations provide all the physical and biological benefits of those made with noble alloys; (4) unique low heat transfer properties of titanium reduce thermal sensitivities; and, (5) galvanic reactions and patient "metallic taste" are eliminated with titanium restorations (Glidewell, 1990).

Of particular interest is the claim stated above that "Porcelain fused to titanium restorations provide all the physical ... benefits of those made with noble alloys." This claim generated the interest to investigate the bond strength of PFT.

Research Hypothesis

The bond strength of dental porcelain-fused-to-titanium is equal to or greater than that of a porcelain-fused-to-metal system.

Null Hypothesis

There is no difference between the bond strength of dental porcelain-fused-to-titanium and that of a porcelain-fused-to-metal system.

II. LITERATURE REVIEW

A. Background Information On Titanium Metal

Titanium is Number 22 in the Periodic Table of the Elements, has a relative atomic mass of 47.9, a coefficient of thermal expansion of 9.7 X 10^{-6}/°C at 20-540°C, and a very low density of 4.51 g/cm^3 (O'Brien, 1989; Lampman, 1990) (Table 1).

Titanium was first discovered by William Gregor, an English scientist, in 1789 (Voitek, 1991). Titanium is the ninth most abundant element in the earth's crust and the fourth most abundant structural metal after aluminum, iron, and magnesium. It is mined in the form of rutile, ilmenite, anatas and brookite. Australia produced 77% of the total known production of rutile; and, Australia (33%), Norway (24%), USA (17%), and the former USSR (12%) produced over 85% of the global production of ilmenite (WHO, 1982).

Most titanium is converted to titanium dioxide; and, is used in the paint and coatings industry. Only five to ten percent of titanium is actually converted to the pure metallic form. Titanium ore is heated in the presence of chlorine and carbon to produce $TiCl_4$. This compound is reduced with molten sodium to produce a titanium sponge, which is compressed and fused under vacuum or in an argon atmosphere into pure metallic titanium (Parr et al., 1985).

Table 1. Physical and Mechanical Properties.

Physical and Mechanical Properties

Material	Vickers Hardness Number (VHN) (kg/mm^2)	Yield Strength 0.2 % offset (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)	Modulus of Elasticity (MPa X 10^3)	Density (g/cm^3)	Fusion Temp (°C)
Grade 1 Ti	135[3]	170-310[1]	240[1]	24[1]	100[2]	4.51[4]	1760-1860
Grade 2 Ti	165[3]	275-410[1]	343[1]	20[1]	100[2]	4.51[4]	1760-1860
Grade 3 Ti	-	377-520[1]	440[1]	18[1]	100[2]	4.51[4]	1760-1860
Grade 4 Ti	215[3]	480[1]	550[1]	20[1]	100[2]	4.51[4]	1760-1860
Type III Gold (Softened)	120-150	200-240	400-450	30-40	100[4]	15.5	875-1000
Type III Gold (Hardened)	150-170	290-310	510-550	12-22	100[4]	15.5	875-1000
Type IV Gold (Softened)	150-170	275-310	470-520	30-35	99.3[4]	15.2	875-1000
Type IV Gold (Hardened)	220-250	480-510	750-790	5-7	99.3[4]	15.2	875-1000
Olympia™ (Au-Pd)	220[5]	550-575	700-730	8-12	116-117	13.5[5]	1271-1304[5]
Vitallium™ (CoCr)	380	495	640	1.5	228	8.5[2]	1400-1500
Ticonium™ (NiCr)	340	690	800	1.7	186	7.9[4]	1275

Craig, 1993

[1] Lampman, 1990

[2] Lautenschlager and Monaghan, 1993

[3] Dentaurum,1993

[4] O'Brien, 1989

[5] Olympia Data Sheet (Jelenko)

Dental applications of titanium include implants, crowns, fixed partial dentures, removable partial denture frameworks, orthodontic wire, posts, and pins (Branemark, 1983; Akagi et al., 1992; Craig, 1993; Goss et al., 1992; Burgess and Summitt, 1991).

Titanium is widely used in industry for jet engines, air frames, chemical processing, nuclear waste containment, heat exchange units, ocean water desalinization, marine equipment, deep well drilling, and food processing equipment (Parr et al., 1985).

The aerospace industry uses three-fourths of the metallic titanium in the United States for defense applications. For decreased weight and decreased radar detection the F-15 fighter is one-third titanium and the B-1B bomber is one-fourth titanium. Modern submarines are made of titanium in order to avoid detection by magnetometers, and provides resistance to the corrosion produced by sea water. Also, titanium in the form of $TiCl_4$ is used to produce smoke screens. Today, the United States stockpiles 36,000 tons for defense applications (Chance, 1992).

B. Physical and Mechanical Properties of Titanium

Titanium is a lustrous white metal, soft and ductile when free of oxygen; it is nonmagnetic, burns in air, and is the only metal that will burn in a nitrogen environment. (Parr et al., 1985). The metallurgical properties of titanium are a high strength-to-weight ratio, low weight-to-volume ratio, excellent fatigue and corrosion resistance, low thermal conductivity, non-toxic; and very importantly it is non-allergenic (Voitek, 1991).

In November 1987, titanium was classified into four grades of purity by the American Society for Testing and Materials (ASTM). These grades are titanium 1 through 4. Slight changes in the amount of oxygen are responsible for significant differences in the physical properties of the alloys. As seen in Table 2 each grade has a different limit for the amount of oxygen which determines its classification. Titanium Grades 1 and 2 are recommended for fixed restorations, and titanium Grade 4 is recommended for removable metal frameworks (Dentaurum, 1990).

The Vickers hardness number (VHN) of a restorative dental material is determined by the Vickers hardness test in which a 136-degree diamond pyramid-shaped indenter is forced into a material with a definite load application. The diagonal made on the surface is measured and the number is used to calculate the VHN (Craig, 1993) . A comparison of Tables 1 and 2 reveals that as the oxygen content increases the VHN increases.

The Vickers Hardness Number (kg/mm2) of various dental alloys can be seen in Table 1. Titanium VHN falls between Type III and Type IV dental gold, metal alloys used mainly in fixed prosthodontics; and Vitallium and Ticonium, metals used to fabricate removable partial denture (RPD) frameworks. Titanium Grade 1 is approximately as hard as Soft Type III gold; whereas, Titanium Grade 2 is close to the hardness of Hard Type III and Soft Type IV gold. “Soft Gold” is the result of quenching after casting, and “Hard Gold” is the result of hardening heat treatment in which a casting is allowed to cool slowly to room temperature.

TABLE 2

TITANIUM GRADES 1 - 4
CHEMICAL COMPOSITION (Volume %)

American Society for Testing and Materials

Material	O	N	C	H	Fe	Ti
Grade 1 Ti	0.18	0.03	0.10	0.015*	0.20	Balance
Grade 2 Ti	0.25	0.03	0.10	0.015*	0.30	Balance
Grade 3 Ti	0.35	0.05	0.10	0.015*	0.30	Balance
Grade 4 Ti	0.40	0.05	0.10	0.015*	0.50	Balance

Lampman, 1990
*Lautenschlager and Monaghan, 1993

The yield strength describes the stress at which a material functions plastically and becomes deformed (Craig, 1993). Titanium Grade 1 has about the same yield strength as Type III gold, and softened Type IV gold (Table 1).

Ultimate tensile strength is defined as the maximum stress that a material can withstand before failure in tension; in other words, before it breaks (Craig, 1993). Titanium Grade 1 breaks at a value less than that of Soft Type III gold and Type IV gold (Table 1).

Elongation is the deformation that results from the application of a tensile force; and is a good indicator of burnishability (Craig, 1993). Soft Type III gold and Soft Type IV gold are very burnishable, however, titanium is not (Table 1).

The elastic modulus is the measure of stiffness of a material, and indicates whether a material is rigid or flexible (Craig, 1993). Titanium is about as rigid as Type III gold (Table 1).

C. Biologic Characteristics of Titanium

Titanium has become very popular in some dental applications because of its biocompatibility. The term biocompatible is defined in Dorland's Illustrated Medical Dictionary (1988) as: being harmonious with life; or, not having toxic or injurious effects on biological function.

Biocompatibility is the most important characteristic of titanium relative to its use in dentistry (Donley and Gillette, 1991). Biocompatibility of a material is determined by the surface characteristics that the material presents to the host's response (Kasemo and Lausmaa, 1988). The most important surface characteristic is the type and amount of the surface oxide layer (Albrektsson and Jacobsson, 1987). Other important properties are solubility of the material in host tissues, and surface roughness characteristic of the material (Kasemo, 1983; Brunette, 1988).

Pure titanium forms an oxide layer on the surface immediately when exposed to air which provides corrosion resistance. Several stable titanium oxides are produced such as TiO, TiO_2, Ti_2O_3, and Ti_3O_4, with TiO_2 being the most stable and predominantly used under physiologic conditions. Ten angstroms of oxide layer is formed in 1 millisecond; and, 50 - 100 angstroms is formed in 1 minute (Kasemo, 1983).

Passivity refers to the ability of the oxide layer to remain intact under physiologic conditions. A metal is passivated when an oxide layer is formed and maintained once in contact with the host's bioenvironment (Tengvall et al., 1989).

There are currently many different designs of endosseous dental implants; however, the most successful and widely studied dental implant is the one made of titanium that was originally described by Branemark (Branemark, 1983). Albrektsson et al. (1981) reported evidence of direct contact between titanium and bone on the microscopic level utilizing Branemark's implant criteria. Hansson et al. (1983) examined Branemark implants that were removed after as much as seven years service. They reported that implants of pure titanium may be integrated into both human and animal tissue.

D. Dental Porcelain

1. Definition, Composition, and Properties

Porcelain is defined in the Glossary of Prosthodontic Terms (1994) as a ceramic material formed of infusible elements joined by lower fusing materials. Most dental porcelains are close to glasses and are used in the fabrication of teeth for dentures, pontics and facings, metal ceramic restorations, crowns, inlays, onlays, and other restorations.

Dental porcelains are classified according to the following fusion temperatures:

High-fusing:	1,288° to 1,371°C (2,350° to 2,500°F)
Medium-fusing:	1,093° to 1,260°C (2,000 to 2,300°F)
Low-fusing:	871° to 1,066°C (1,600° to 1,950°F)

Common ingredients of porcelain used for porcelain-fused-to-metal restorations are (in %): SiO_2 (52.0-62.2), Al_2O_3 (11.65-16.30), CaO (0-2.01), K_2O (9.6-11.3), Na_2O (4.75-8.63), TiO_2 (0-3.01), ZrO_2 (0.16-5.16), SnO_2 (0-15.0), RbO_2 (0.04-0.10), BaO (0-3.52), ZnO (0-0.26), UO_3 (0-0.67), and B_2O_3, CO_2, and H_2O (3.24-9.58) (O'Brien, 1989).

2. Porcelain Bonding to Metal

The following developments have made metal-ceramic systems possible: (1) ceramics and alloys that form a strong bond, (2) ceramics and alloys with closely matched coefficients of thermal expansion, (3) low-fusing ceramic materials, (4) alloys that resist deformation at the ceramic fusing temperature. To achieve well fitting restorations, the ceramic must fuse and bond to the alloy without deformation of the metal. Upon cooling, the ceramic and metal must contract intimately so the ceramic will not crack or separate from the alloy (Craig, 1993).

Factors that promote good adhesion or bonding of a porcelain to a metal include:

wetting, adherent oxide, and mechanical retention. Good wetting is characterized by a low contact angle of molten porcelain when it is fired onto the metal. Wetting allows greater contact of the glass into the metallic surface irregularities. Good wetting is also indicative of chemical compatibility between the porcelain and the metal. An adherent oxide present on the metal surface that is wet by the porcelain promotes chemical bonding and is produced by diffusion of atoms from the metal and porcelain into this oxide. A nonadherent oxide can lead to bond failure. Mechanical retention can be created by the surface roughness of the metal, especially in the presence of undercuts (O'Brien, 1989).

Thermal expansion compatibility between the ceramic and metal is a necessary requirement for adhesion. If the two materials contract at different rates during cooling, porcelain may crack or separate from the metal due to strong residual stresses formed across the interface. To avoid these problems porcelains and metal alloys are formulated with closely matching coefficients of thermal expansion. In general, porcelains have coefficients of thermal expansion between 13.0 and 14.0 X 10^{-6}°C; and, the metals between 13.5 and 14.5 X 10^{-6}°C. After firing porcelain, the metal contracts a little more than the ceramic during cooling due to a difference of 0.5 X 10^{-6}°C in thermal expansion coefficients. This condition is desirable because it puts the ceramic under slight residual compression improving resistance to applied tensile forces (Craig, 1993).

3. Classification of Bond Failure

There are six types of porcelain-metal bond failures based on the location of failure (Figure 1). Type I bond failures occur between the metal and porcelain; Type II between the metal oxide and the porcelain; Type III is cohesive within the porcelain; Type IV between the metal and metal oxide; Type V between the metal oxide and metal oxide; and, Type VI is cohesive within the metal. Proper bonding is represented by Type III cohesive failure (O'Brien, 1989; Craig, 1993).

E. Dental Casting of Titanium

1. Heat and Titanium Characteristics

Commercially pure titanium at room temperature has an alpha (hexagonal close-packed) crystal structure, which transforms to a beta (body-centered cubic) structure at a temperature of about 885°C (1625°F). The type and amount of impurities or alloying additions can raise or lower the transformation temperature. It is the addition of alloying elements that divides the single temperature for equilibrium transformation into two temperatures. These are the alpha transus, below which the alloy is all-alpha, and the beta transus, above which the alloy is all-beta. Both alpha and beta are present between these temperatures. Typical transus temperatures (with an uncertainty of about ± 15°C, or ± 25°F) of commercially pure titanium are as follows:

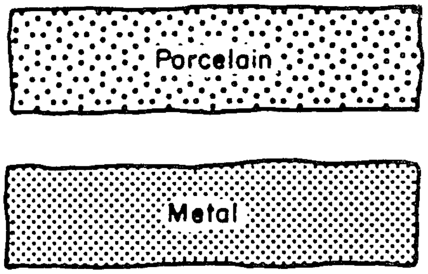
ASTM Grade 1 beta transus of 888°C (1630°F) and alpha transus of 880°C (1620°F),

ASTM Grade 2 beta transus of 913°C (1675°F) and alpha transus of 890°C (1635°F),

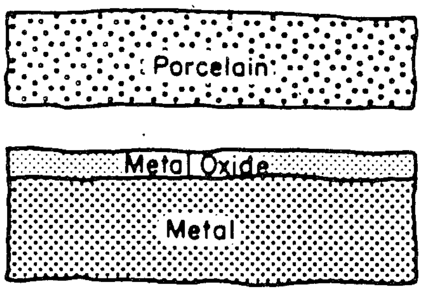
ASTM Grade 3 beta transus of 920°C (1685°F) and alpha transus of 900°C (1650°F),

ASTM Grade 4 beta transus of 950°C (1740°F) and alpha transus of 905°C (1660°F).

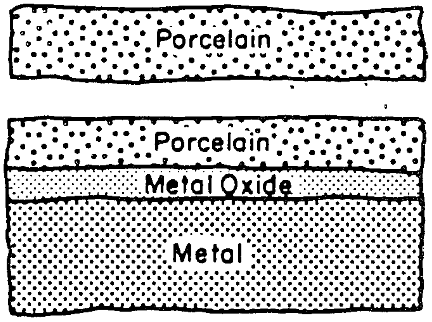
Figure 1. Classification of Porcelain Enamel Fractures According to Interfaces Formed (O'Brien, 1989; Craig, 1993).



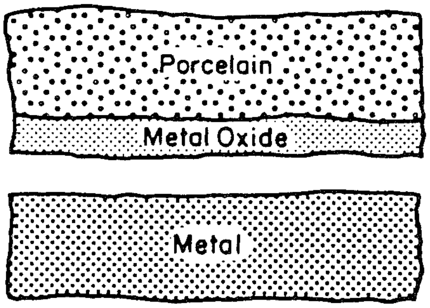
I. METAL - PORCELAIN



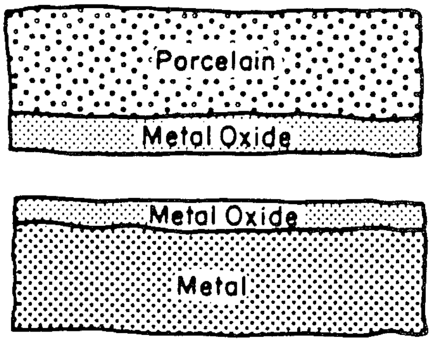
II. METAL OXIDE - PORCELAIN



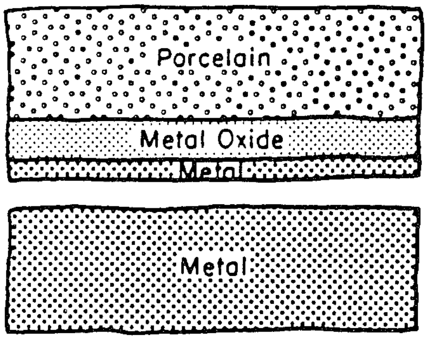
III. COHESIVE WITHIN PORCELAIN



IV. METAL - METAL OXIDE



V. METAL OXIDE - METAL OXIDE



VI. COHESIVE WITHIN METAL

Titanium is scaled and embrittled by oxygen-rich surface layers formed at temperatures higher than 540°C (1000°F); which, is commonly referred to as alpha case. A protective atmosphere such as argon should be considered for any heating above 540°C (1000°F) (Lampman, 1990).

Alpha phased titanium is stabilized primarily by the addition of aluminum; which, increases tensile strength, creep strength, and the elastic moduli. Aluminum, above 6%, promotes ordering and Ti_3Al (α_2) formation which is associated with embrittlement. Therefore, the aluminum content of all titanium alloys is normally below 7%. The Beta phase titanium is stabilized by two groups: beta isomorphous and beta eutectoid. The isomorphous group consists of elements that are completely miscible in the beta phase which include: molybdenum, vanadium, tantalum, and niobium. The eutectoid-forming group have eutectoid temperatures as much as 335°C (600°F) below the transformation temperature of unalloyed titanium and include: manganese, iron, chromium, cobalt, nickel, copper, and silicon (Lampman, 1990).

2. Casting Machines

Ida et al. (1979) described the “CASTMATIC” casting machine he and his team developed at Iwatani Co. Ltd., Osaka, Japan. This machine melts an alloy placed on a metal or graphite crucible by utilizing a tungsten electrode and casts the molten alloy into a mold with a flow of argon with a combination of pressure from the upper side and of vacuum from the lower side (Ida et al., 1979). This machine consists of an upper melting chamber and a lower casting chamber. The investment mold is placed in the lower chamber against the opening to the upper chamber. A titanium ingot is placed on a copper crucible, which tilts after the ingot melts. Both chambers are evacuated and replaced with argon gas before melting. The arc ignites automatically after the optimum gas pressure is reached (Hamanaka, 1993).

The Ohara (Ohara Co., Ltd., Osaka, Japan) is a vertical centrifugal casting machine (Ohara, 1987). In previous models, arc melting was conducted under an argon gas flow without replacing the argon gas. In the new machine, the chamber is evacuated and replaced with an argon gas before melting, with greatly improved casting properties (Hamanaka, 1993).

Hamanaka et al. (1989) described a new casting machine his group developed for casting titanium and Ni-Ti alloys. This machine was based on the same principle as the "Castmatic." The main features developed were: (1) the melting and casting chambers were evacuated to a higher degree by means of an oil diffusion pump, (2) a heater was placed in the casting chamber to control the mold temperature, (3) two types of copper crucibles were developed: a split type and a tilting type, (4) a device for direct suction was placed at the bottom of the mold for improved castability, (5) the vacuum tank and compressed argon gas tank were set to operate more efficiently, (6) the capacity for melting alloy was about 100 g with the use of the water-cooled electrode and double D.C. electric sources, and (7) a new control system was developed.

The "Tycast 3000" is a titanium casting machine developed and being manufactured by the Jeneric/Pentron Corporation. This machine utilizes a tungsten electrode for generating an arc to melt the metal, and a powerful driving mechanism to provide sufficient centrifugal force to the molten metal (Prasad et al., 1994).

3. Investments

It is difficult to cast pure titanium or titanium alloys in the conventional dental casting mold due to titanium's high melting temperature and chemical reactivity with the ceramic crucible and investment material at high temperatures (Ida et al., 1982).

Ida et al. (1982) reported on the casting of pure titanium and titanium-based alloys into molds of phosphate-bonded silica or magnesia investments using the "Castmatic" casting machine. Their results were as follows: (1) the magnesia investment was much more suitable for casting of titanium than the phosphate-bonded silica investment; (2) pure titanium or titanium-based alloys should not be cast in conventional phosphate-bonded silica investments, because the interaction between titanium and the main component of the mold, i.e., silica, made the castings hard and brittle, (3) the use of magnesia as a coating material combined with phosphate-bonded silica investment had a little effect on the properties of titanium castings, (4) the thermal rate of magnesia investments at 800°C was not so different from that of phosphate-bonded investments, (5) pure titanium and titanium alloys seemed to be suitable for crowns, fixed partial dentures, and prostheses for dental implants, and (6) practical titanium castings such as crowns, fixed partial dentures, and blades for dental implants could be made successfully using magnesia investments.

Mueller et al. (1990) reported on casting pure titanium using two phosphate-bonded investments. One was Rema Exakt (Dentaurum) which is compatible with molten titanium; and, the other was Biovest (Dentsply) which reacts strongly with molten titanium. Castings made in Rema were ductile and non-porous; but, castings made in Biovest were porous and brittle. Thermal expansion data indicated Rema obtains its expansion primarily from quartz; whereas, Biovest obtains its expansion from cristobalite. It was concluded that the Biovest-Ti incompatibility may be due to depletion of silica (cristobalite), whereas the presence of quartz inhibits this reaction in Rema.

Takahashi et al. (1990) reported on casting pure titanium into five different phosphate-bonded SiO_2 investments (Rema Exakt, Ceramigold, Complete, Tai-Vest, and Full-Vest) at 350°C using the "Castmatic" casting machine. They examined the mesh castability, the fit of the MOD inlay castings, and the Knoop hardness and the micro-structure in cross-sections of castings. The setting and thermal expansion, the compressive strength, and the x-ray diffraction analysis of the investments were also measured. Their findings were: (1) the castability test by the mesh method showed that the castability in Rema Exact was significantly lower than that in Ceramigold, Complete, Tai-Vest, and Full-Vest, (2) when MOD inlay gaps were considered, the degrees of fit of castings made in Rema Exakt, Tai-Vest, and Full-Vest molds were better than those made in Ceramigold and Complete, (3) an investment with low total (setting plus thermal) expansion produced undersized castings, and when two investment materials had the same total expansion, the lower-compressive-strength material yielded the more undersized castings, (4) the casting shrinkage of pure titanium is lower than that of Rexillium III (Ni-Cr) and Vitallium (Co-Cr) alloys, (5) the castings made in Ceramigold and Complete molds had significantly harder surfaces than those made in the other molds, and (6) the castings made in Ceramigold had a significantly thicker reacted layer (450 μm), whereas the reacted layers of castings made in Rema Exact, Tai-Vest, and Full-Vest were thinner (200-250 μm) than the hardened layers reported by other investigators. Ida et al. (1982) reported that titanium cast in an 800°C phosphate-bonded investment mold had a hardened layer 500 μm thick. Taira et al. (1989) found that castings into a ZrO_2 coated phosphate-bonded investment mold at 850°C had a layer that exceeded 300 μm thick.

4. Castability

Blackman et al. (1991) measured the dimensional changes in nineteen of twenty RPD frameworks that were cast with 99.5% commercially pure titanium using the equipment, materials, and procedures of the Ohara Company. They reported a maximum mean horizontal cross arch shrinkage of 2.6%, a vertical expansion of 1.8%, and essentially a neutral mean of the measured changes in the horizontal anterioposterior direction. It was concluded that dimensional changes in cast titanium RPD frameworks can be expected to be similar to those found with commonly used RPD base metal alloys.

Bessing and Bergman (1992) reported on the castability of unalloyed titanium in three different casting machines. The machines used were the Castmatic-S (Iwatani Int. Corp., Osaka, Japan), the Cyclarc machine (J. Morita Europe GmbH, Frankfurt, Germany), and the Titaniumer machine (Ohara Co., Ltd., Osaka, Japan). The unalloyed titanium used for the castings were those recommended by the manufacturers' for their respective machines. Ten castings were made of each casting machine, and the castability test according to Australian Standard (AS 1620, 1985) was used to evaluate the castability. They found (1) only the Titaniumer casting machine failed to produce more than eight complete castings out of ten, (2) the three machines produced castings with almost similar mean values for the crown edge diameter, and (3) the best results were obtained with the Cyclarc machine.

Blackman et al. (1992) examined titanium crown margins having external angles of 45-degrees and 90-degrees. Forty copings of each group were cast of commercially pure titanium (99.5%) using the Ohara casting machine. The titanium copings were cemented onto their individual master dies, and sectioned longitudinally for microscopic evaluation of marginal fit. They found that horizontal measurements in the plane of the shoulder revealed 90-degree margins were within a 0.050 mm standard; however, 45-degree margins had a mean greater than 0.050 mm. It was also found that 45-degree margins had less surface roughness than 90-degree margins.

5. Titanium Dental Porcelain

When firing porcelain on metal, it is desirable for the metal to contract a little more than the ceramic during cooling because this puts the ceramic under slight residual compression improving resistance to applied tensile forces. The tolerable difference in coefficients of thermal expansion between the porcelain and the metal is approximately 0.5×10^{-6}°C (Craig, 1993).

In order to achieve successful metal-ceramic bonding, low-fusing dental porcelains had to be developed that were compatible with titanium's low coefficient of thermal expansion (9.7 X 10^{-6}/°C at 20-540°C). Duceratin, for example, has a firing temperature of about 720-750°C (depending on the furnace used), with a coefficient of thermal expansion of 9.5 - 9.7 X 10^{-6}/°C at 25-500°C for the opaque porcelain, and 8.8 - 8.9 X 10^{-6}/°C at 25-500°C for the dentin porcelain. The composition of Duceratin opaque (O) and dentin (D) titanium dental porcelains are as follows (in %): SiO_2 (O=52.37, D=67.65)), Al_2O_3 (O=12.67, D=8.03), CaO (O=2.20, D=2.06), K_2O (O=6.06, D=9.14), Na_2O (O=8.27, D=8.28), SnO_2 (O=14.46, D=0.00), BaO (O=0.21, D=0.28), and B_2O_3 (O=2.46, D=2.10), Li_2O (O=0.70, D=0.64), F (O=0.42, D=1.82) (Ducera, 1991; Kononen and Kivilahti, 1994).

A significant problem with firing dental porcelain to titanium is the widespread dissolution of oxygen into the titanium lattice resulting in thick, oxygen-rich titanium layers. Above temperatures of about 750°C the oxygen dissolution rapidly increases, which damages the mechanical compatibility of the titanium-porcelain joint during a conventional fusing procedure. Another potential problem is the formation of reaction products during firing which may fracture under the influence of thermal stresses (Kononen and Kivilahti, 1994).

Riesgo et al. (1984) studied the bonding processes of dental porcelains with different titanium alloys (20% Co, 30% Co, 20% Cu, 30% Pd, 50% Pd, and 70% Pd). The porcelains were fired using two methods: low and high vacuum. The low firing of opaque porcelain was unsatisfactory; whereas, high vacuum rendered good results. The body porcelain was low vacuum fired over the opaque and then air autoglazed successfully. Studies of the metal-ceramic interface showed that the compatibility changed according to each couple tested, especially the Ti-30% Pd alloy. Results of this research indicate (1) it is feasible to fire dental porcelain over titanium alloys using high vacuum, and (2) the study of the metal-ceramic interface showed Ti-30% Pd to be the most successful.

Menis et al. (1986) studied the development of porcelains compatible with titanium castings. Titanium's low expansion coefficients and oxidation characteristics require porcelains of lower fusion temperature and expansion. Three experimental porcelains were examined and their coefficients of thermal expansion ($X10^{-6}$ / °C) on heating from 25 to 400 °C were found to be 6.3, 7.0, and 6.9; and, on cooling they were 7.1, 7.8, and 7.0.

Razzoog et al. (1994) reported on the effects of accelerated aging on the color stability of a conventional high-fusing dental porcelain (Ceramco) and a low-fusing titanium dental porcelain (Procera). Twenty porcelain-metal discs (four shades) of each metal-ceramic system were exposed to accelerated aging in a Weather-O-Meter 25-WR for periods of 100, 300, 600, and 900 hours; where, 300 hours of accelerated aging is equivalent to one year of exposure in an outdoor environment. They found (1) the greatest color change occurred during the first 100 hours of accelerated aging for both porcelains, (3) in three of the four shades tested the Procera porcelain had statistically greater color change than the Ceramco porcelain, and (4) both the Ceramco and Procera porcelains, in all shades, showed a "slight" color change after 900 hours of accelerated aging.

Kononen and Kivilahti (1994) investigated the microstructures and compositions of the interfacial regions (joints) created when firing a low-fusing porcelain (Duceratin) to commercially pure grade 1 titanium in an ordinary dental furnace and also in a high-vacuum furnace. Twenty-three samples were made; of which, 10 were sandblasted with 50 μm alumina particles at 2 atmosphere, and 13 were electrolytically polished. The interfacial regions between the ceramic coatings and titanium were analyzed using the following methods: (1) scanning acoustic microscopy (C-SAM) and scanning electron microscopy (SEM) techniques, (2) the formation sequence of reaction products was studied at 750°C thermodynamically and observed experimentally both at 750 and 800°C, and (3) the magnitudes of thermal stresses of the joints were evaluated with a multilayer finite-element model based on elastic strain analysis. They found (1) the C-SAM and SEM showed the integrity of the porcelain-titanium joints are better in the sandblasted samples than in the electropolished ones which contained larger defects, (2) due to the more continuous reaction layers in the electropolished samples, cracks propagated more readily during the cooling procedure, (3) the rough surface of sandblasted samples hindered the formation of continuous planar reaction layers whereby only short cracks were formed, (4) thermodynamic calculations and experimental chemical analyses strongly indicate that the cause for the cracking of the reaction zone is due to both an oxygen-containing solid titanium solution and a thin layer of Ti(oxo)silicide.

F. Metal-Ceramic Bond Strength Tests And Studies

Over the past thirty years various tests have been used to evaluate the bond strength of metal-ceramic systems. Anusavice (1983) presented a literature review of various bond strength tests and described them as (1) Pull-Shear and Push-Shear Tests, (2) Flexure Tests, (3) Planar Shear Tests, (4) Tensile Tests, (5) Conical Interface Shear Test, (6) Tension-Shear Test, (7) Oblique-Shear Test, and (8) Torsion Tests.

1. Pull-Shear and Push-Shear Tests

The bond strength tests that are most widely accepted (DIN 13927 and ISO 9693) are the pull-shear and push-shear tests (Anusavice, 1983; Ducera, 1991) (Figures 2 and 3). Shear tests involve bonding two strips in lap fashion and then separating them by pulling or pushing.

Shell and Nielsen (1962) reported on a pull-shear test in which the specimen assembly was designed such that (1) no thermal stresses due to different expansion coefficients were present, (2) no stresses normal to the bond interface developed during testing, and (3) no cohesive porcelain failure occurred on shearing the bond.

Moffa et al. (1973) reported on one of the first studies of base metal porcelain-alloy bond strength using a pull-shear bond test.

Lubovich and Goodkind (1977) reported on using the pull-shear test to investigate the bond strength of one precious alloy (S.M.G. III), one semiprecious alloy (S.M.G. W), and three nonprecious alloys (Nobil-Ceram, Permabond, and Victory), with two porcelains Ceramco and Vita. From this study it was concluded that (1) there were

Figure 2. Pull-Shear Bond Test Design (from Anusavice, 1983).

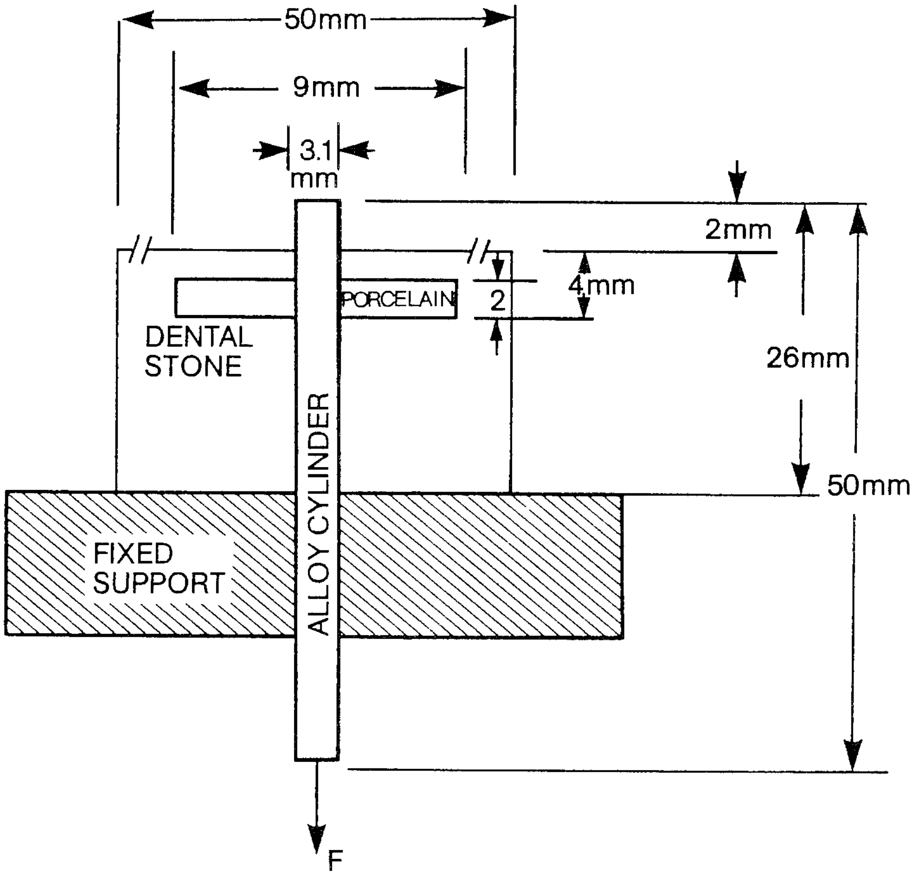
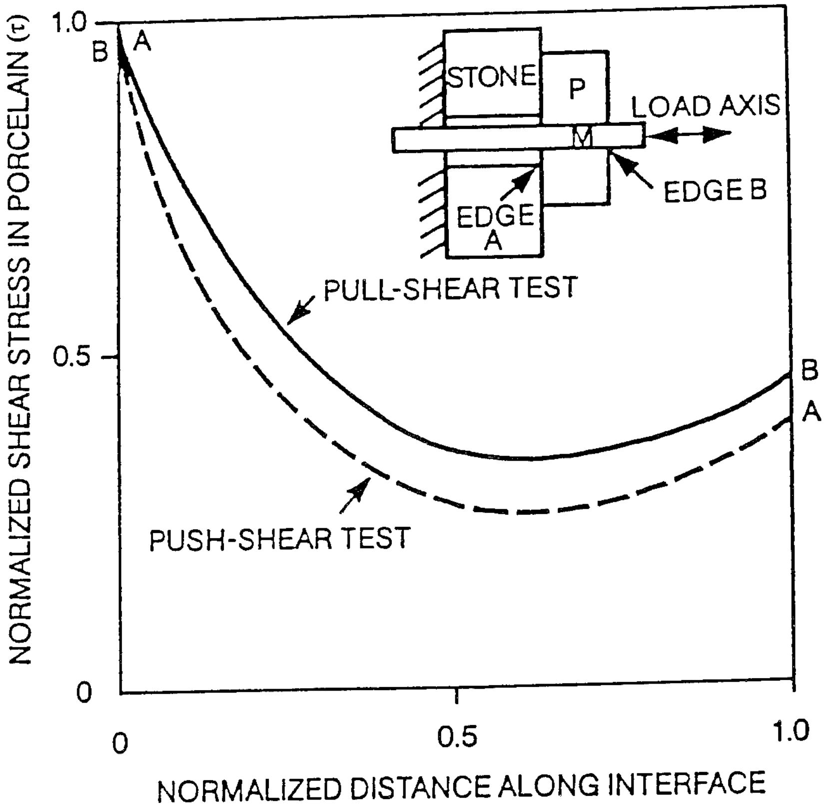

50mm
9mm
3.1 mm
2mm
4mm
2
PORCELAIN
DENTAL STONE
26mm
50mm
ALLOY CYLINDER
FIXED SUPPORT
F

Figure 3. Normalized Porcelain Shear Stress Along Porcelain-Metal Interface in Pull-Shear and Push-Shear Bond Test Specimens (Anusavice, 1980).



NORMALIZED SHEAR STRESS IN PORCELAIN (τ)
1.0
0.5
0
B
A
STONE
P
M
LOAD AXIS
EDGE B
EDGE A
PULL-SHEAR TEST
PUSH-SHEAR TEST
B
A
0
0.5
1.0
NORMALIZED DISTANCE ALONG INTERFACE

no significant differences in bond strength between Ceramco and Vita porcelains, (2) the various alloys tested demonstrated significantly different bond strengths, (3) nonprecious alloys performed better with Ceramco porcelain than with Vita porcelain, (4) surface roughness appeared to be the one common factor in nonprecious alloys that relates to large differences in bond strength, and (5) the cylindrical pull-through shear test is a reliable, reproducible method of testing metal-ceramic bond shear strength.

Carpenter and Goodkind (1979) reported on testing 120 samples using a pull-shear and four-point flexure test to investigate the effects of varied surface textures created by aluminum oxide air abrasion upon a gold-palladium-silver ceramic alloy, Cameo, and a nonprecious nickel- chrome ceramic alloy, N/P II. The objectives were (1) to determine how these variations in surface texture affect the bond strength of the two metal-ceramic systems studied and (2) to evaluate the resultant surface area and re-entrant angles created by air abrasion within the limited scope of available instrumentation. The pull-shear test employed a layer of platinum foil above and below the porcelain attachment area and a felt pad for load distribution. It was found that (1) overall bond strength was not significantly increased in the Cameo and N/P II ceramic alloy systems using the four-point bend test, (2) the bond strength was not significantly increased with Cameo alloy but was significantly increased for the rough-textured N/P II ceramic-alloy tested using the pull-rod test, and (3) test result means suggested that surface texture other than a smooth surface may enhance the bond resistance slightly against induced shear stresses, especially for the nonprecious metal-ceramic systems investigated.

Asgar and Giday (1978) found in their study that the push-type test method results in values significantly higher than those found with the pull-type test method.

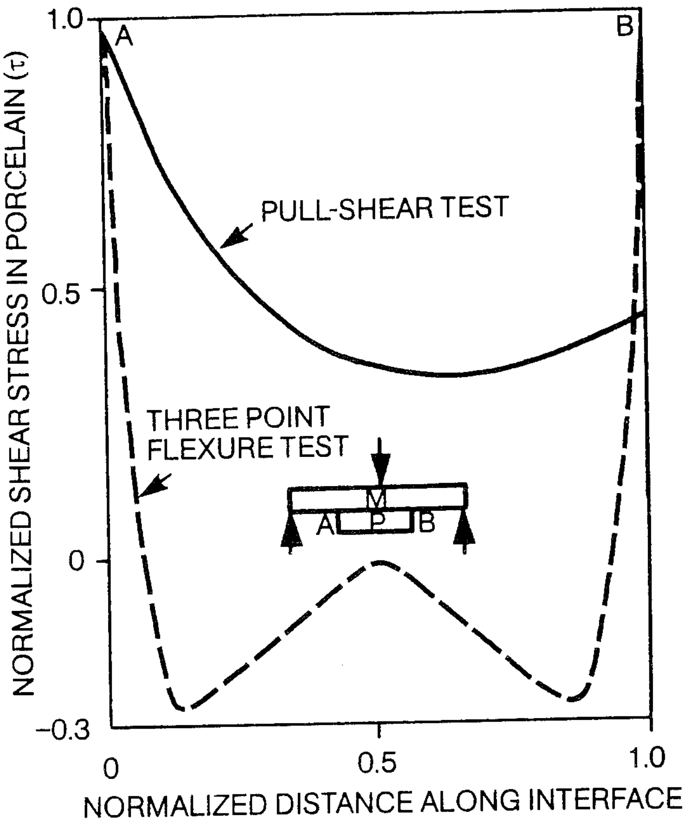
Malhotra and Maickel (1980) evaluated the effect of the pull-shear versus push-shear test on bond strengths for four gold-based and one palladium-based alloy veneered with three commercial products. They reported that the separator used on the metal rods prior to investing the porcelain part of specimens in the dental stone eliminated the undesirable bond between the dental stone and the metal rod. The shear bond strengths obtained were believed to be true shear bond strengths. Secondly, push-test shear bond strengths were always higher than pull-test shear bond strengths. Both the push-test and pull-test seemed to be equally valid for evaluating bond strengths in porcelain-metal composites.

2. Flexure Tests

Flexure tests are comprised of the (1) Three-Point Flexure Test, (2) Four-Point Flexure Test, and the (3) Stress Analysis of a Semicircular Arch. The three-point flexure test (Figure 4) can be described as one in which a porcelain tab is bonded to the center of a metal strip; and, then a vertical force is applied to the center of the test specimen from above with the metal strip being supported below near both ends. This test method places the specimen surface with porcelain in a state of tension.

Lavine and Custer (1966) used the three-point flexure test to investigate the variables that would provide an optimal porcelain-fused-to-metal bond strength such as

Figure 4. Normalized Shear Stress in Porcelain along Porcelain-Metal Interface (A-B) in Three-Point Flexure Bond Test Specimen (from Anusavice, 1983).


1.0
0.5
0
−0.3
A
B
PULL-SHEAR TEST
THREE POINT
FLEXURE TEST
M
A
P
B
0
0.5
1.0
NORMALIZED SHEAR STRESS IN PORCELAIN (τ)
NORMALIZED DISTANCE ALONG INTERFACE

(1) preheating the metal to 1,800° F. before the addition of porcelain, as compared to eliminating the preheating, (2) stone-roughened surface compared to cast texture, (3) application of a metal conditioner to the castings fired at two different temperatures, and (4) vacuum and non-vacuum firing of the porcelain.

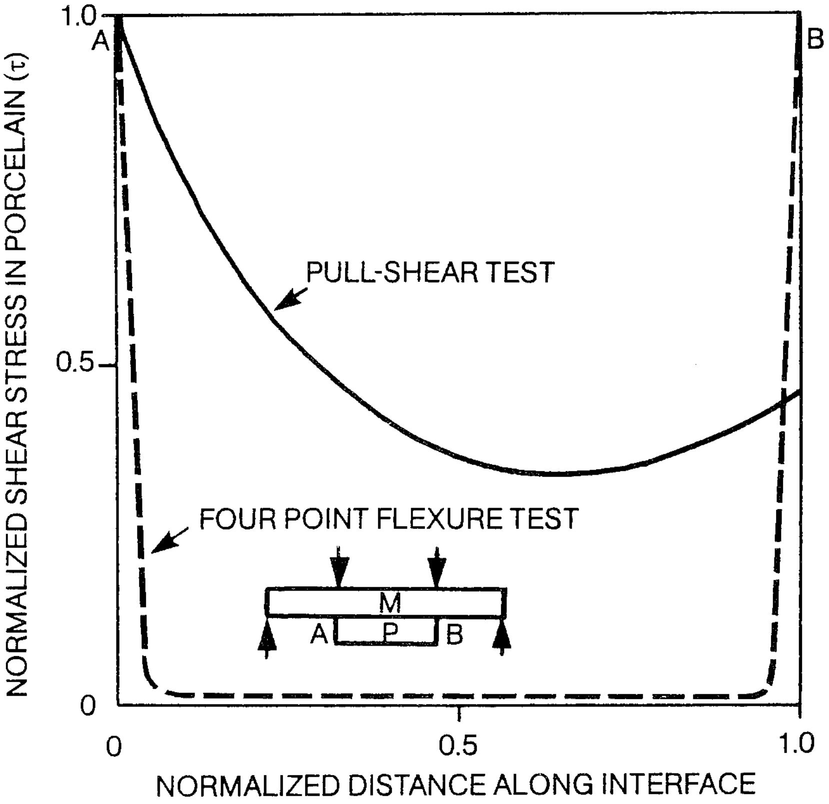
The four-point flexure test (Figure 5) can be described as one in which a porcelain tab is bonded to or onto the center of a metal strip; and, two vertical forces are applied to the test specimen on either side of the porcelain tab with the metal strip being supported underneath near both ends.

Caputo, Dunn, and Reisbick (1977) used four-point bending to evaluate bond strengths of specimens in which porcelain was bonded to the central tension side of a metal beam. The test produced failures at predictable locations and was able to differentiate between metal surface treatments.

DeHoff et al. (1980) evaluated the four-point flexural test and concluded that (1) the initial mode of failure is likely to occur in porcelain as a result of tensile stress and (2) bond strength data are highly sensitive to specimen geometry. Thus, the universal use of this flexure test may not yield comparable data.

DeRijk et al. (1982) and Tesk et al. (1982) described a four-point flexure test of a composite beam that determines the strength under pure tensile stress. The theoretical analysis for this test considered both simple beam bending and finite element stress analysis. The composite beam was designed such that it (1) evaluates the system in terms of brittle failure, i.e. in tension, (2) does not require absolutely precise alignment in the test fixture, (3) provides a direct evaluation of strength of the system, (4) avoids

Figure 5. Normalized Shear Stress in Porcelain along Porcelain-Metal Interface (A-B) in Four-Point Flexure Bond Test Specimen (from Anusavice, 1983).


1.0
0.5
0
A
B
NORMALIZED SHEAR STRESS IN PORCELAIN (τ)
PULL-SHEAR TEST
FOUR POINT FLEXURE TEST
M
A
P
B
0
0.5
1.0
NORMALIZED DISTANCE ALONG INTERFACE

significant stress concentration effects due to wettability and spreadability, (5) minimizes stress concentrations due to loading and provides a uniform stress along the sample, through the PFM bond interface, (6) has relatively easy sample preparation, with porcelain opaque and body layers applied in the same manner as used in the dental laboratory, (7) has a thickness and mass of metal which approximates those found at some locations on a clinical crown or fixed partial denture, (8) for efficiency provides more than one test result per sample, and (9) the normal tensile stress is essentially the same as the surface stress, when the ratio of the metal thickness underneath the porcelain component (c) to total thickness of the metal and porcelain component (t) is between 0.2 and 0.5 inclusive, i.e., $0.2 \leq c/t \leq 0.5$ (Figure 6).

Derand and Hero (1992) reported on using the four-point flexure test to investigate the bond strength of porcelain on cast vs. wrought titanium (Figure 7). The test specimens they used in their experiment were porcelain fused to titanium plates, that were then glued to two steel bars.

Mackert et al. (1976) introduced the stress analysis of a semicircular arch flexure test (Figure 8). This design is a flexure-shear test which consists of a cast metal semicircular hoop to which porcelain is bonded on the convex surface. The fired specimen is then deformed radially in tension and, as a result of this semicircular specimen design, tensile stresses are produced in the metal, compressive stresses in the porcelain, and longitudinal shear stress at the interface. The apparent advantages of this test design are (1) porcelain application on a convex surface simulates clinical design, (2) the stress application is analogous to clinical conditions, (3) compressive

Figure 6. Variation of Normalized Maximum Tensile Stress, δ_n, with Thickness of Porcelain, b, with Constant Bending Moment, M (Tesk et al., 1982).

VARIATION OF NORMALIZED MAXIMUM TENSILE STRESS, δ_n, WITH THICKNESS OF PORCELAIN, b, WITH CONSTANT BENDING MOMENT, M

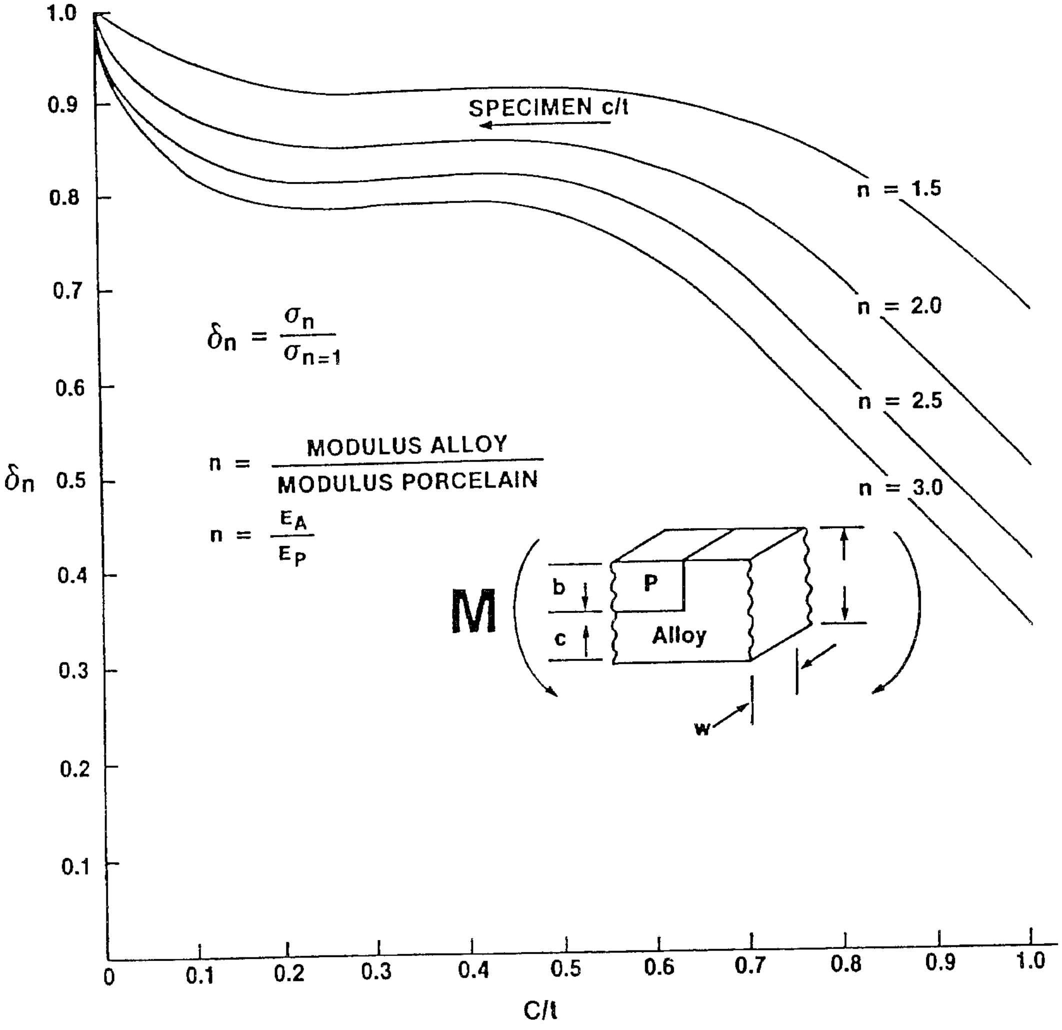
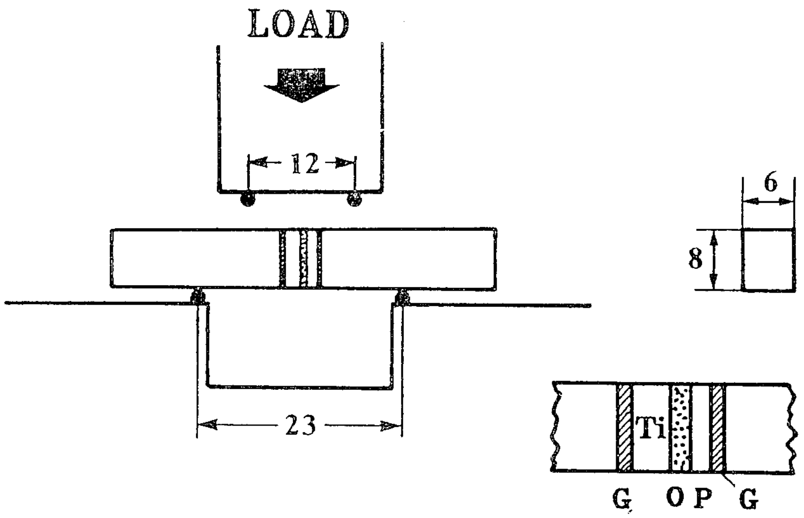


Figure 7. Four-Point Bending of a Specimen Glued to Steel Extension Bars. (Derand and Hero, 1992).

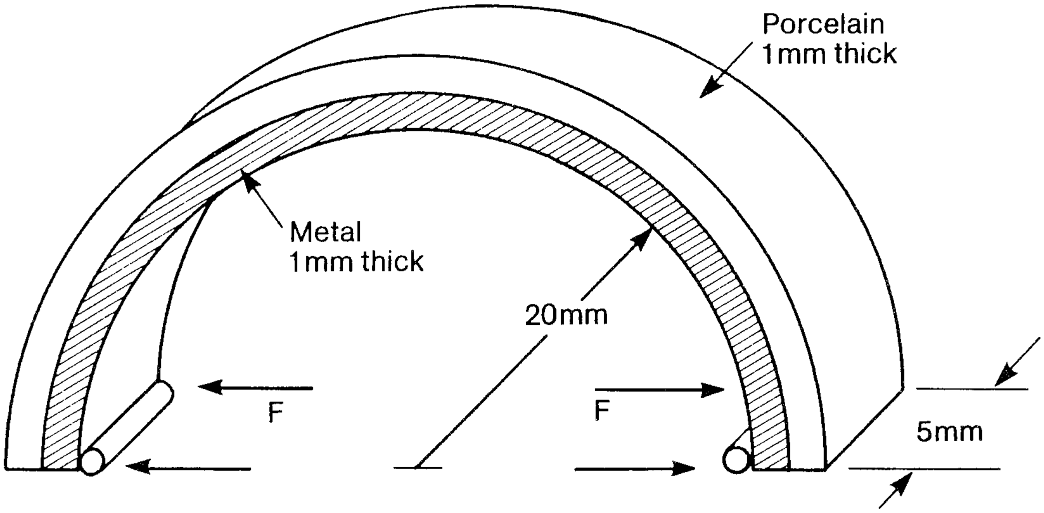


G = GLUE

O = OPAQUE P

P = DENTIN P

Figure 8. Semicircular Arch Flexure Bond Test (Mackert et al., 1976) [from Anusavice, 1983].


Porcelain
1mm thick
Metal
1mm thick
20mm
F
F
5mm

stresses will be concentrated within the porcelain during all phases of deformation which should insure against premature porcelain fracture in areas outside the adherence zone, (4) force application directly on the porcelain is eliminated, and (5) the specimens are suitable for cyclic loading without modification.

3. Planar Shear Tests

The Planar Shear Tests are characterized by a planar metal-porcelain interface. This is in contrast to the push-shear or pull-shear tests, which a curved interfacial area is involved. (Anusavice, 1983).

Civjan et al. (1974) utilized a planar shear test (Figure 9) with a circular cross-sectional area to determine the bond strength of four Au-Pd-Ag alloys and one Ni-Cr-Be alloy with Ceramco porcelain. Another Ni-Cr-Be alloy (Ticon) was tested with Vita porcelain. Instead of gripping an alloy cylinder and shearing off a porcelain disc, this test design involves the application of a force to a metal disc approximately 6.4 mm in diameter and 1.6 mm thick bonded to a porcelain cylinder about 3.2 mm in diameter, which is embedded in an acrylic resin cube.

Susz et al. (1980) utilized a planar shear test (Figure 10) with a rectangular cross-sectional area to determine the effect of surface treatment on bond strength for a Au-Pt-In alloy.

Chong and Beech (1980) reported that the planar shear test can distinguish between poor and excellent bonding systems. This test is not free of the complicating effects of residual stress; however, it is easy to perform, allows for discrimination between surface roughness effects (polished versus abraded surfaces), and produces a relatively

Figure 9. Planar Interface Shear Bond Test with Circular Interfacial Area (Civjan et al., 1974) [from Anusavice, 1983].

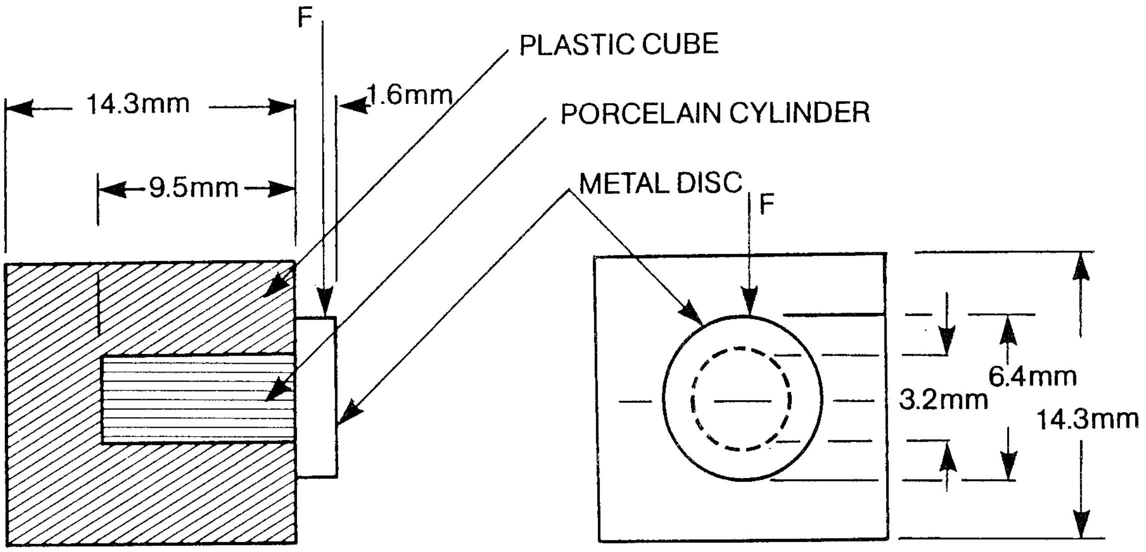
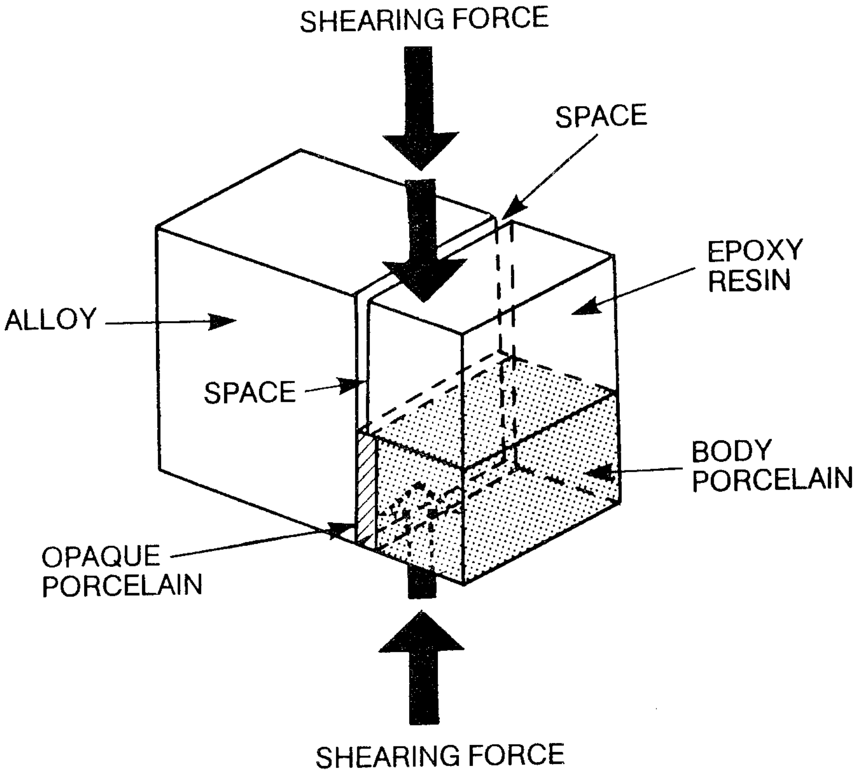

F
PLASTIC CUBE
14.3mm
1.6mm
PORCELAIN CYLINDER
9.5mm
METAL DISC
F
6.4mm
3.2mm
14.3mm

Figure 10. Planar Interface Shear Bond Test with Rectangular Area (Susz et al., 1980) [from Anusavice, 1983].


SHEARING FORCE
SPACE
EPOXY RESIN
ALLOY
SPACE
BODY PORCELAIN
OPAQUE PORCELAIN
SHEARING FORCE

uniform stress distribution. It was probably the most ideal test developed at that time for evaluating porcelain-metal adherence.

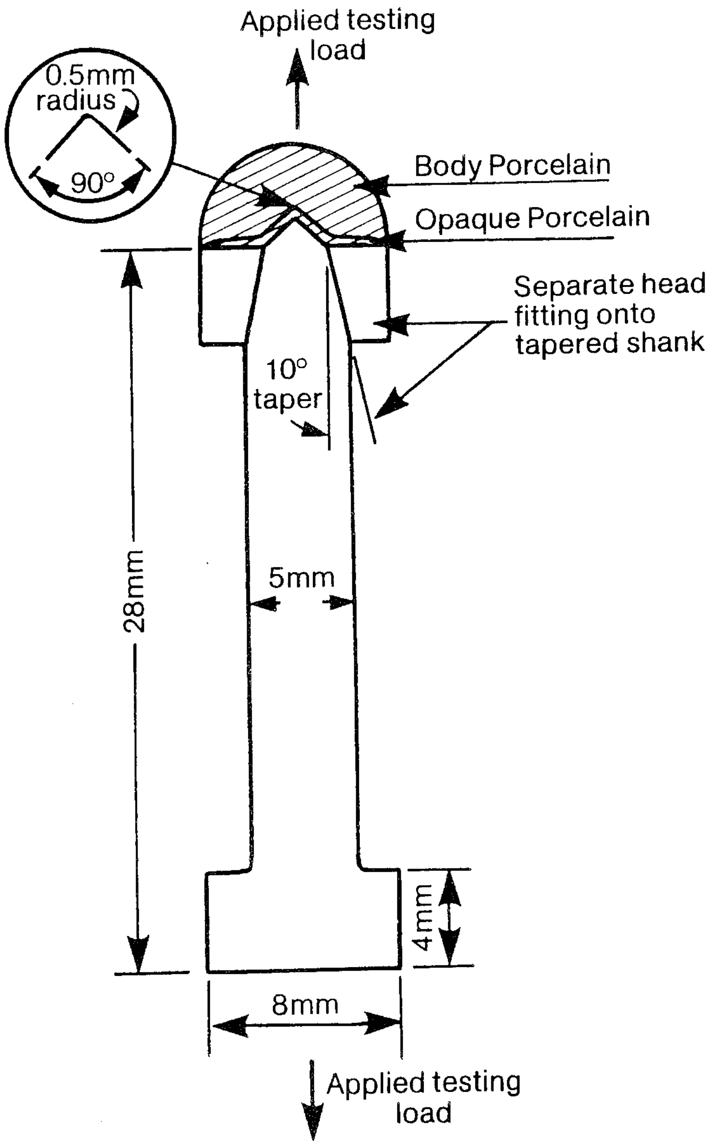
4. Tensile Tests

"The typical bond strength test design consists of a layer of porcelain placed on one flat surface or between the flat surfaces of two metal rods: tensile force is applied along the long axis of the rod (or rods) until tensile failure of the adherence zone or the porcelain occurs. From a finite element stress analysis point of view, this type of test design is associated with a very high probability of porcelain tensile failure. Because of the alignment difficulties of the two-rod specimens and the presence of generalized stress raisers on the external surface of porcelain, failure within porcelain may be due to non uniform stress distribution" (Anusavice, 1983). Thus, one should not assume that the adherence strength is greater than the tensile strength of dental porcelain based on this type of test.

5. Conical Interface Shear Test

This test design represents the adhesion test proposed in the British Standard Institute Specification No. BS 3366 for porcelain-metal systems (Figure 11). Finite element stress analysis of the conical interface design exhibits the same level of stress concentrations but slightly lower probability of porcelain tensile failure compared to the pull-shear or push-shear tests (Anusavice, 1983). Sced and McLean (1972) used the conical interface shear test method to determine mean bond strength values for gold alloy-Vita VMK porcelain specimens, for Co-Cr alloy-Vita VMK porcelain specimens, and Ni-Cr alloy-Vita VMK porcelain specimens.

Figure 11. Conical Interface Shear Test (British Standard Institute Specification No. BS 3366) [from Anusavice, 1983].


Applied testing load
0.5mm radius
90°
Body Porcelain
Opaque Porcelain
Separate head fitting onto tapered shank
10° taper
28mm
5mm
4mm
8mm
Applied testing load

6. Tension-Shear Test

Wight et al. introduced the tension-shear test in 1977 (Figure 12). This test is a modification of ASTM Test D 2295-72. This design tests the bond strength of a porcelain tab (8.00 mm x 5.72 mm) bonded between the ends of two metal strips (20 mm x 5.72 mm); such that, the porcelain tab is bonded between the right end of the lower metal strip and the left end of the upper metal strip. Tension-shear forces are then applied in a direction away from the porcelain tab until shear failure is achieved.

7. Oblique-Shear Test

This test design was developed by Anusavice and Fairhurst in 1977, and represents the most ideal test from a stress analysis point of view (Figure 13). In this test a force is applied vertically by a steel bearing of 2 mm radius upon a block of metal (12.5mm X 6.4mm X 6.4mm) in which a 1 mm tab of porcelain is sandwiched in the metal block at a 45° angle. A very uniform shear stress distribution develops with negligible stress concentration effects at the end points. Plastic deformation of the alloy surface frequently occurs which results from the inability of one or both of the metal blocks to move freely in a lateral direction as fracture is initiated along the porcelain-metal interface. As long as this difficulty exists, this test may lead to erroneous bond strength data (Anusavice, 1983).

8. Torsion Tests

Carter (1975) first introduced the torsion bond strength test (Figure 14). This test involves the torque of a metal strip (26.5 mm x 12.5 mm x 0.50 mm) that is sandwiched

Figure 12. Tension-Shear Bond Test (Wight et al., 1977) [from Anusavice, 1983].

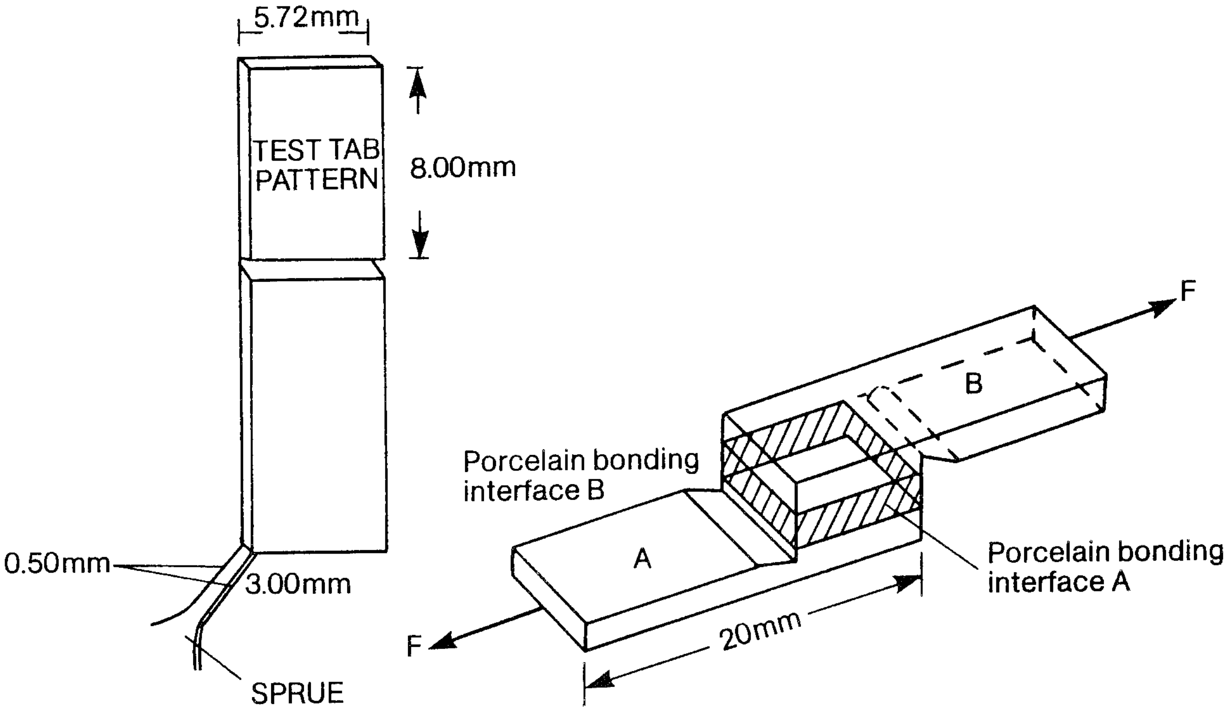
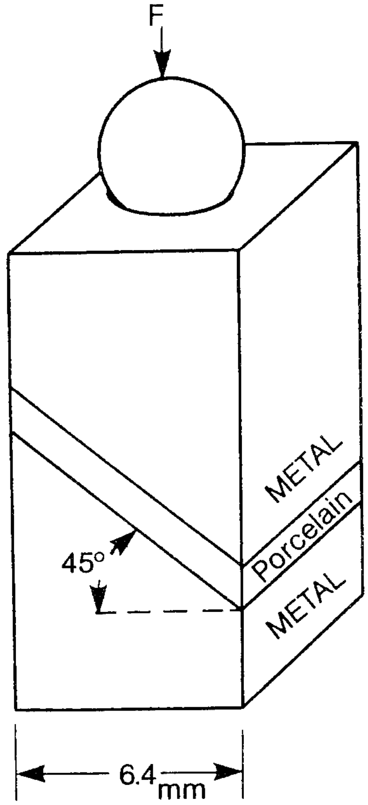

5.72mm
TEST TAB PATTERN
8.00mm
0.50mm
3.00mm
SPRUE
Porcelain bonding interface B
A
B
F
F
20mm
Porcelain bonding interface A

Figure 13. Oblique-Shear Bond Test (Anusavice and Fairhurst, 1977) [from Anusavice, 1983].


F
METAL
Porcelain
METAL
45°
6.4mm

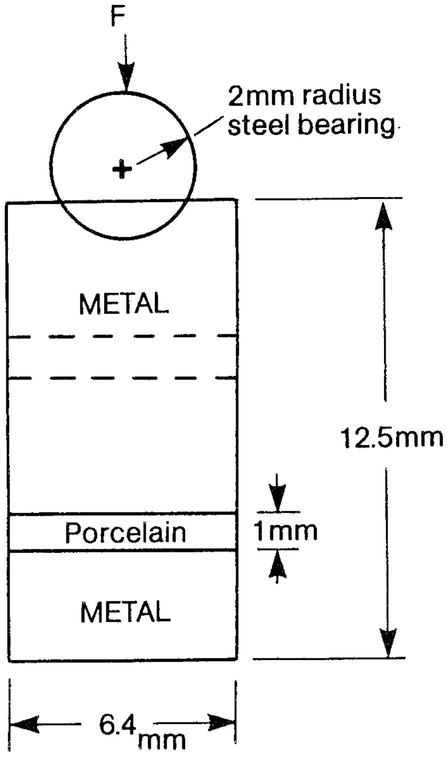
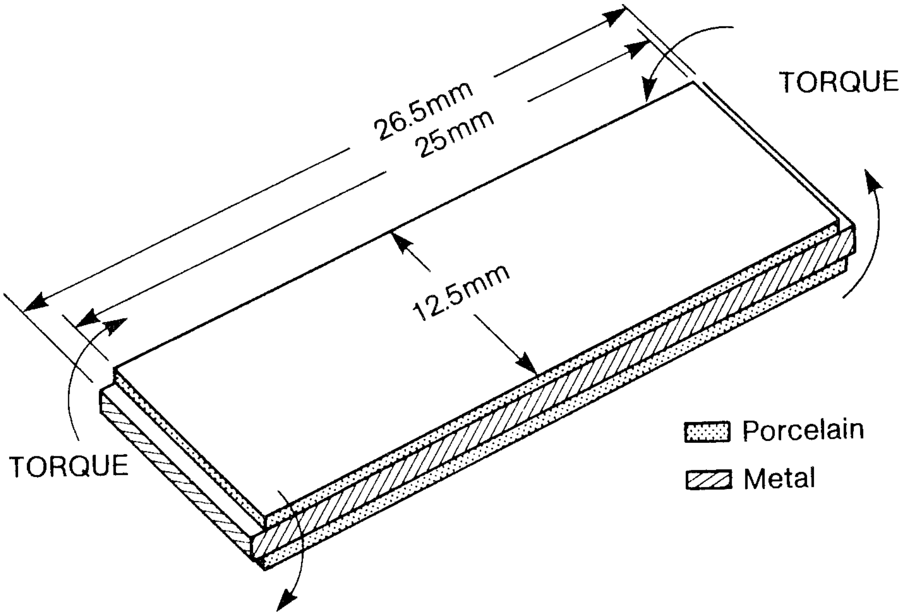

F
2mm radius steel bearing
METAL
Porcelain
METAL
1mm
12.5mm
6.4mm

Figure 14. Torsion Bond Test (Carter, 1975) [from Anusavice, 1983].



Metal Thickness = 0.50mm
Porcelain Thickness = 0.25mm (each layer)

between two porcelain strips (25mm x 12.5 mm x 0.25 mm). Anusavice (1983) has described this test as being too complex for two-dimensional finite element stress analysis. Also, specimen preparation procedures and calculations of stress distributions are also complex. For these reasons, this test is not recommended for general bond strength evaluation.

Menis (1987) reported on two types of bond strength tests. The first involves measuring the bonding strength of porcelain-metal combinations using a variety of configurations of porcelain and metal to build the test systems. These tests included the Shell-Nielsen pull shear test or push shear test; the three-point bending porcelain-on-metal strip composite used by Schwickerath, Bolling, Kraft and others; the semi-circular composite beam (porcelain in compression) of Anusavice; the four point bending porcelain-in-beam test of Tesk, et al., and DeRijk, et al.; the biaxial bending of a layered composite strain of Mackert, et al.; the biaxial shell test of Piddock, et al.; the parallel shear strength of the porcelain-metal interface used by Hausselt and Kondoh; and the quantitative controlled fracture test performed in four point bending of Hausselt. The second type of bond strength test involves thermal compatibility. These tests include the thermal shock resistant tests of Anusavice; the gap change measurement of a porcelain veneered split metal ring of Whitlock, et al. and Tesk et al.; and, other similar tests that investigate the stress and other factors generated by firing porcelain on metal.

Anusavice (1980) analyzed eleven PFM bond tests for interfacial shear stress distribution using finite-element stress analysis. The eleven tests analyzed were (1) rectangular parallel shear (loaded at interface), (2) rectangular parallel shear (distributed loading), (3) conical-interface shear, (4) pull shear (Shell-Nielsen), (5) push-shear, (6) tension/shear, (7) semi-circular arch flexure, (8) three-point bending (porcelain in compression), (9) three-point bending (porcelain under tension), (10) four-point bending (porcelain under compression), and (11) four-point bending (porcelain under tension). He found the test configurations least likely to exhibit failure in porcelain were the rectangular parallel shear test (loaded at the interface), the three-point bending test (porcelain under compression), and the four-point bending test (porcelain under compression).

DeRijk (1982) reported on seven PFT systems [I=Pentillium, II=Howmedica NP II, III=Howmedica A, IV=Dentsply Microbond, V=Dentsply Experimental, VI=Williams Gold I, and VII=Williams Gold 7], in which he tested their bond strengths using the four-point bending of a PFM beam methodology.

Buchness et al. (1988) reported that after experimentation using the methodology described above, reglazing of the fractured samples by refiring them through the glaze cycle, did not appear to have a major negative effect on the PFM bond strength. Stannard et al. (1990) used a different methodology and reported that multiple firings of PFM systems did not specifically reduce the bond strength.

III. MATERIALS AND METHODS

A. Experimental Plan

The objective of this study was to determine bond strengths of two dental porcelains fused to 99.6% pure titanium metal. Porcelain-fused-to-titanium (PFT) composite beams were tested using a four-point bending method as described by DeRijk (1982). For comparison, a commonly-used porcelain-fused-to-metal (PFM) system was tested in the same manner. A total of forty-five composite beam samples were made. The samples were divided as follows: Group 1 consisted of twenty samples of Duceratin porcelain (Ducera, West Germany) fused to Rematitan metal (Dentaurum, Fed. Rep. Germany) (ASTM Grade 1); Group 2 consisted of twenty samples of Vita porcelain (Vita Titankeramik, Vita, West Germany) fused to Rematitan metal (Dentaurum, Fed. Rep. Germany) (ASTM Grade 1), and Group 3 consisted of five samples of Vita porcelain (Vita VMK-68 , Vita, West Germany) fused to Olympia gold alloy metal (Jelenko, Armonk, KY) (control). Individual samples were placed in a four-point bending fixture and loaded under compression in an Instron Universal Testing Machine (Instron model 1125, Instron Corp., Canton, MA) until porcelain fracture was recorded. After experimentation, the fractured samples were reglazed by refiring them through the glaze cycle, and retested for bond strengths in the same manner.

B. Samples

Samples were made to approximate the dimensions of a three-unit fixed partial denture. The size of the porcelain component was similar to that of a maxillary central incisor. The samples were cast rectangular beams of commercially pure titanium, having the porcelain layer or component bonded into the center of one surface. As seen in Figure 15, samples dimensions were as follows: titanium metal beam length (l) = 40mm, width (w) = 9mm, thickness (t) = 3.5mm, thickness underneath porcelain component (c) = 1.5mm; and, porcelain component = 10mm (p) X 9mm (w) X 2mm (b).

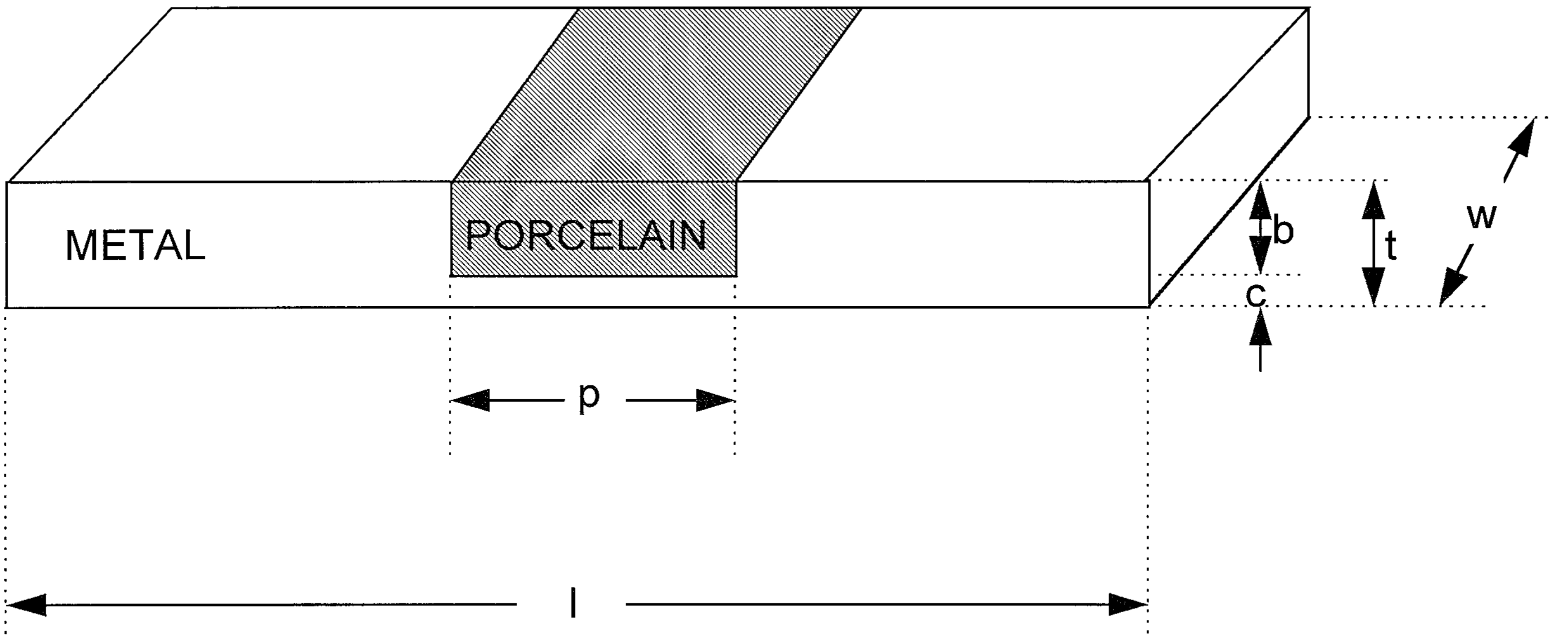
C. Fabrication of Wax Pattern

An aluminum mold was machined for making casting patterns for the titanium component of the composite metal-porcelain samples. Wax patterns were fabricated by lubricating the mold, pouring a molten 50/50 mix of blue inlay wax (Kerr Manufacturing Co., Romulus, MI) and Dr. Peck's hard purple inlay wax (Temrex Corp., Freeport, NY) into the mold, and allowing wax to cool. Excess wax was trimmed away and the wax pattern was removed from the mold (Plate 1).

D. Spruing and Investing

Forty wax patterns for titanium castings were sprued on the flat side of the pattern using two sprues attached to the sprue former. The wax patterns were invested in a phosphate bonded casting investment (Rematitan Plus, Dentaurum, Fed. Rep. Germany) according to manufacturer's instructions (Plate 2).

Figure 15. Sample Dimensions.



Dimensions of Metal:
l = 40 mm
w = 9 mm
t = 3.5 mm
c = 1.5 mm

Dimensions of Porcelain Component:
p = 10 mm
w = 9 mm
b = 2 mm

Plate 1. Aluminum Mold and Wax Pattern.

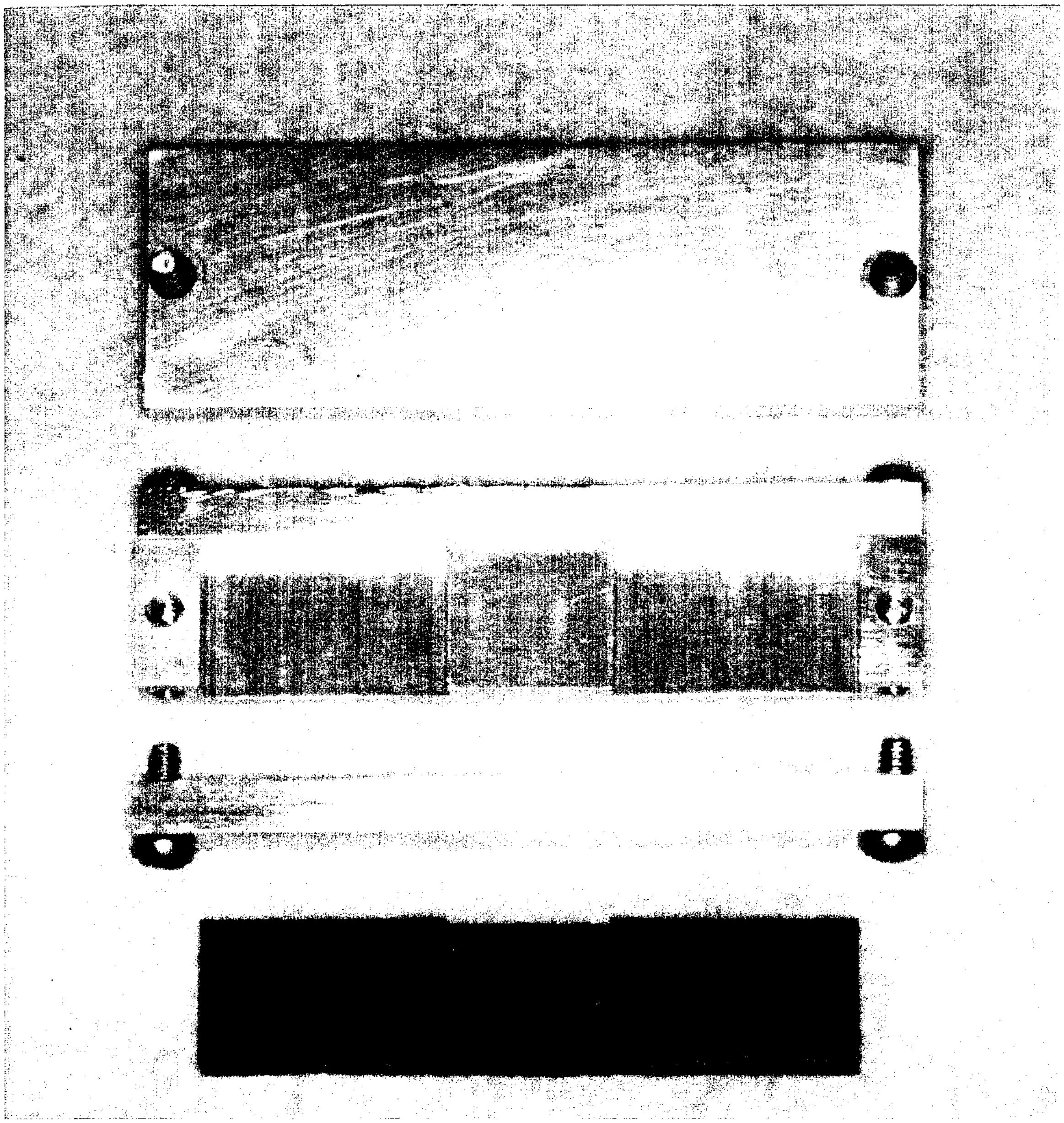


Plate 2. Spruing and Investing Wax Pattern.

Five wax patterns for the gold alloy metal-ceramic were fabricated using a multipurpose phosphate investment (Ceramigold, Whip Mix Corp., Louisville, KY) according to manufacturer's recommendations.

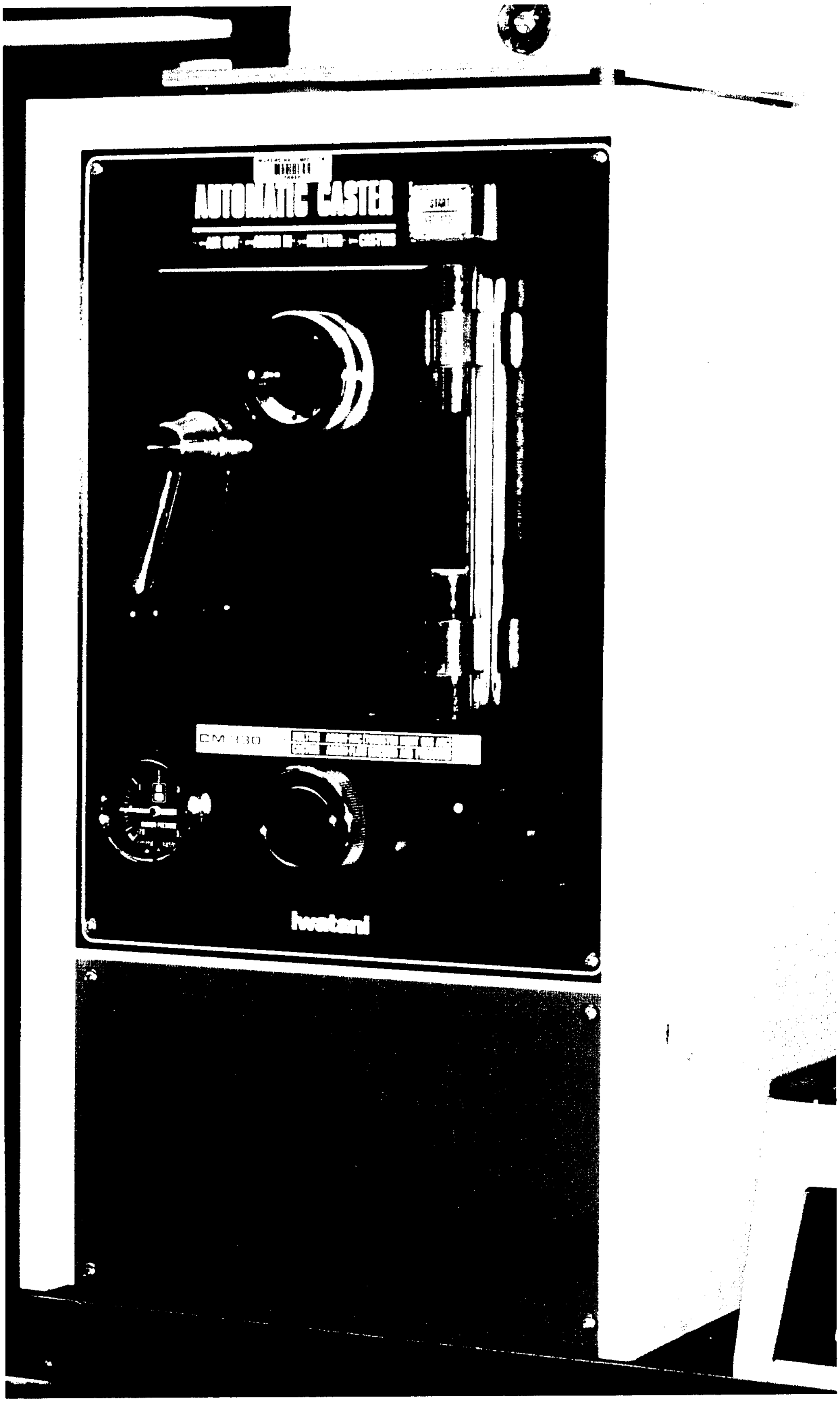
E. Burnout

The wax patterns were then burned out with a four phased processing cycle.(KaVo EWL Furnace, West Germany). All increases and decreases in temperature were at a constant 5°C per minute. Phases were as follows: In the first phase, the temperature was brought up to 150°C in 30 minutes and held for 90 minutes; in the second phase the temperature was raised to 250°C and held for 90 minutes; in the third phase the temperature was raised to 1000°C and held for 60 minutes; and, in the fourth phase, the furnace was cooled slowly to a temperature of 200°C, for casting.

F. Casting

Forty titanium castings were made using the "Castmatic" casting machine (Iwatani Co., Osaka, Japan) (Plate 3). Prior to the machine's first use, argon lines were purged to remove oxygen which would degrade the melt. A 16 gm titanium ingot (Rematitan Grade 1) was then placed on the copper crucible in the upper chamber. The tungsten electrode was positioned 5 mm above the upper surface of the ingot. The casting ring was removed from the furnace (temperature, 200°C) and placed in the middle of the platform plate in the lower chamber with its opening centered below the chamber

Plate 3. “Castmatic” Casting Machine.


AUTOMATIC CASTER
START
CM 330
Iwatani

connector. The platform adjustment handle was then used to raise the ring and press it firmly into an annular seal placed between the ring and the top of the lower chamber around the connector. The door was then closed, the main switch was turned on, and the START button pressed. Argon flooded the upper chamber, while a vacuum was being created in the lower chamber. When the argon reached 6.5 psi the arc ignited and melted the metal. After the titanium was melted and held in a molten state for 10 seconds, the crucible tipped and the melt flowed into the mold. When casting was complete, the ring was quenched in cool water immediately.

Five castings were made using ceramic gold alloy and a conventional lost wax/ centrifugal casting technique.

The castings were then air abraded and cleaned.

G. Metal Finishing

Castings were separated from sprues using separating discs. The outer surfaces of the metal castings were smoothed in a Buehler polisher (Buehler, Lake Bluff, IL) using #400 then #600 carbimet paper discs under water.

H. Measurement of Metal Casting

The metal castings were measured (Mitutoyo Digimatic Indicator, Mitutoyo Corp., Japan) to determine metal width, thickness, and thickness in areas that would not be accessible after adding the porcelain components.

I. Porcelain Application

Duceratin dental porcelain was applied to twenty titanium metal castings according to manufacturer's recommendations and fired in the furnace (Vacumat 300, Vident, Baldwin Park, CA) as follows:

1. The titanium castings were placed in an acid mixture for about 30 seconds. The acid mixture consisted of 35 parts nitric acid, 5 parts hydrofluoric acid, and 60 parts deionized water.
2. The castings were then steam cleaned.
3. The castings were cleaned with tungsten carbide burs.
4. The castings were sandblasted with 110 μm Al_2O_3 (Korox 110, BEGO, Germany) and discontinued after slight spark production.
5. Duceratin Haftbond was applied to the castings using a short-hair brush; samples were pre-heated for 5 minutes at 480°C and then fired according to the firing chart (Table 3).
6. Duceratin Goldbond was applied with a short-hair brush covering the surface well; samples were pre-heated for 3 minutes at 450°C and fired according to the firing chart (Table 3).
7. The opaque and dentin porcelain layers were applied and samples fired according to the firing chart (Table 3).
8. The samples were then glazed according to the firing chart (Table 3).

TABLE 3

DUCERATIN PORCELAIN FIRING CHART

VACUMAT 300	Final Temp	Predrying Time (min)	Heat Rate (min)	Holding Time (min)	Vacuum Time (min)	Drying Lift (min)
Haftbond	480°C	-	1	5	-	-
Goldbond	755°C	3	6	1	6	-
Washbake	755°C	3	6	1	6	-
Opaque Bake	730°C	3	6	1	6	-
Dentine Bake	720°C	9	6	1	6	5
Glaze	685°C	5	6	2	-	-

The basic temperature for all cycles was 450°C.

Vita Titankeramik dental porcelain was applied to twenty titanium metal castings according to manufacturer's recommendations and fired in the furnace (Vacumat 300, Vident, Baldwin Park, CA) as follows:

1. Castings were oxidized according to the firing chart (Table 4).
2. Castings were sandblasted with 280 microns Al_2O_3 (Ivoclar Williams, Amherst, NY).
3. The castings were then steam cleaned.
4. The opaque and dentin porcelain layers were applied and samples fired according to the firing chart (Table 4).
5. The samples were then glazed according to the firing chart (Table 4).

Vita VMK-68 dental porcelain was applied to the five gold alloy metal-ceramic castings according to manufacturer's recommendations and fired in the furnace (Vacumat 300, Vident, Baldwin Park, CA) as follows:

1. Castings were oxidized according to the firing chart (Table 5).
2. Castings were sandblasted with 25 microns Al_2O_3 (Faskut, Dentsply International Inc., York, PA).
3. The castings were then steam cleaned.
4. The opaque and dentin porcelain layers were applied and samples fired according to the firing chart (Table 5).
5. The samples were then glazed according to the firing chart (Table 5).

TABLE 4

VITA TITANKERAMIK PORCELAIN FIRING CHART

VACUMAT 300	Program	Final Temp	Predrying Time (min)	Heat Rate (min)	Holding Time (min)	Vacuum Time (min)
Oxidation	1	800°C	-	-	3	-
Washbake	4	810°C	-	3	1	4
Opaque Bake	4	810°C	-	3	1	4
Dentine Bake	8	780°C	6	6	1	7
Glaze	7	780°C	4	3	1	-

The basic temperature for all cycles was 400°C.

TABLE 5

VITA VMK-68 PORCELAIN FIRING CHART

VACUMAT 300	Program	Final Temp	Predrying Time (min)	Heat Rate (min)	Holding Time (min)	Vacuum Time (min)
Oxidation	1	1038°C	-	3	-	-
Washbake	10	980°C	3	6	1	6
Opaque Bake	11	970°C	3	6	1	6
Dentine Bake	14	920°C	6	6	1	6
First Correction Dentine Bake	15	915°C	6	6	1	6
Glaze	20	920°C	1	6	1.5	-

The basic temperature for all cycles was 600°C.

J. Finishing of Samples

Overbuilt porcelain samples were finished on the Buehler polisher using #240, #400, and #600 carbimet paper discs under water respectively. The samples were then glazed to simulate the preparation of a metal-ceramic restoration for clinical use. "This procedure places the porcelain-metal junction into a state similar to that following the final glazing of a porcelain-fused-to-metal prosthesis" (DeRijk, 1982) (Plate 4).

K. Measurement of Samples

The samples were then measured (Mitutoyo Digimatic Indicator, Mitutoyo Corp., Japan) a second time to obtain composite beam measurements needed for calculating fracture loads: width, total thickness, and porcelain thickness (Tables 6-8).

L. Experimental Testing of Samples

The samples were individually placed on a four point bending fixture (Plate 5) (Figure 16) and loaded to the point of fracture (Instron Corp., Canton, MA). The bending arm for the four point bending fixture was 6mm; thus, with a span of 12mm. The head speed on the Instron was set at 0.5 mm/min. During testing, fracture of the samples were characterized by a sudden drop in the applied load, as well as a distinct sound. The fracture load data was recorded and bond strengths calculated (Tables 6-8).

Plate 4. Finished Sample.

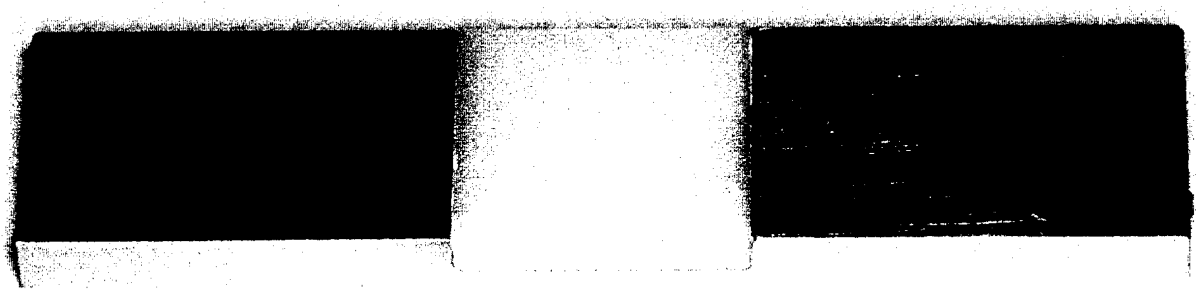


TABLE 6

DUCERATIN / REMATITAN FRACTURE LOAD DATA

Sample No.	Width (mm)	Total Thickness (mm)	Metal Thickness (mm)	Porcelain Thickness (mm)	**Fracture Load (N)**
1	8.7	3.4	1.5	1.9	225
2	8.7	3.2	1.4	1.8	293
3	8.6	3.0	1.3	1.7	239
4	8.8	3.1	1.4	1.7	243
5	8.7	3.1	1.4	1.7	261
6	8.8	3.3	1.4	1.9	179
7	8.8	3.2	1.4	1.8	326
8	8.6	3.1	1.4	1.7	354
9	8.6	3.0	1.3	1.7	358
10	8.7	3.1	1.4	1.7	196
11	8.6	3.2	1.4	1.8	312
12	8.7	3.2	1.4	1.8	219
13	8.7	3.1	1.4	1.7	311
14	8.8	3.2	1.4	1.8	187
15	8.7	3.6	1.6	2.0	354
16	8.8	3.4	1.5	1.9	330
17	8.8	3.3	1.4	1.9	263
18	8.9	3.3	1.4	1.9	163
19	8.8	3.2	1.4	1.8	262
20	8.9	3.3	1.4	1.9	155

TABLE 7

VITA TITANKERAMIK / REMATITAN FRACTURE LOAD DATA

Sample No.	Width (mm)	Total Thickness (mm)	Metal Thickness (mm)	Porcelain Thickness (mm)	**Fracture Load (N)**
1	8.8	3.5	1.5	2.0	223
2	8.7	3.2	1.4	1.8	151
3	8.8	3.3	1.4	1.9	215
4	8.7	3.2	1.4	1.8	172
5	8.7	3.2	1.4	1.8	189
6	8.7	3.1	1.4	1.7	167
7	8.8	3.4	1.5	1.9	245
8	8.9	3.4	1.4	1.9	219
9	8.8	3.3	1.4	1.9	239
10	8.8	3.3	1.4	1.9	238
11	8.7	3.3	1.4	1.9	320
12	8.8	3.2	1.4	1.8	192
13	8.9	3.4	1.5	1.9	229
14	8.8	3.3	1.4	1.9	240
15	8.8	3.4	1.5	1.9	216
16	8.8	3.2	1.4	1.8	228
17	8.7	3.1	1.4	1.7	189
18	8.8	3.4	1.5	1.9	290
19	8.7	3.4	1.5	1.9	224
20	8.7	3.5	1.5	2.0	236

TABLE 8

VITA VMK-68 / OLYMPIA FRACTURE LOAD DATA

Sample No.	Width (mm)	Total Thickness (mm)	Metal Thickness (mm)	Porcelain Thickness (mm)	**Fracture Load (N)**
1	8.7	3.3	1.3	2.0	180
2	8.9	3.4	1.5	1.9	163
3	8.9	3.3	1.4	1.9	182
4	8.9	3.3	1.4	1.9	137
5	8.9	3.3	1.4	1.9	217

Plate 5. Experimental Testing of Sample Using Four-Point Bending.

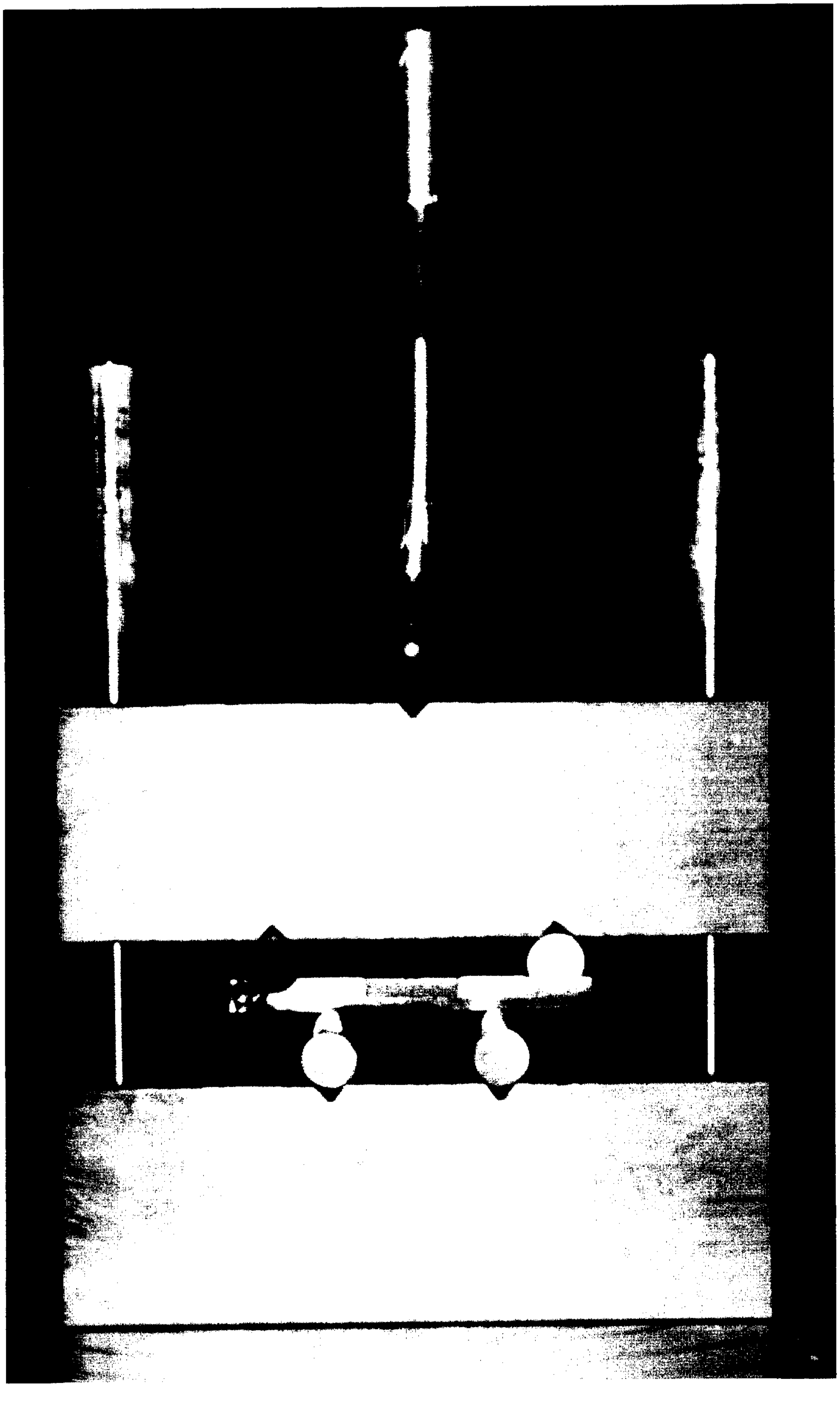
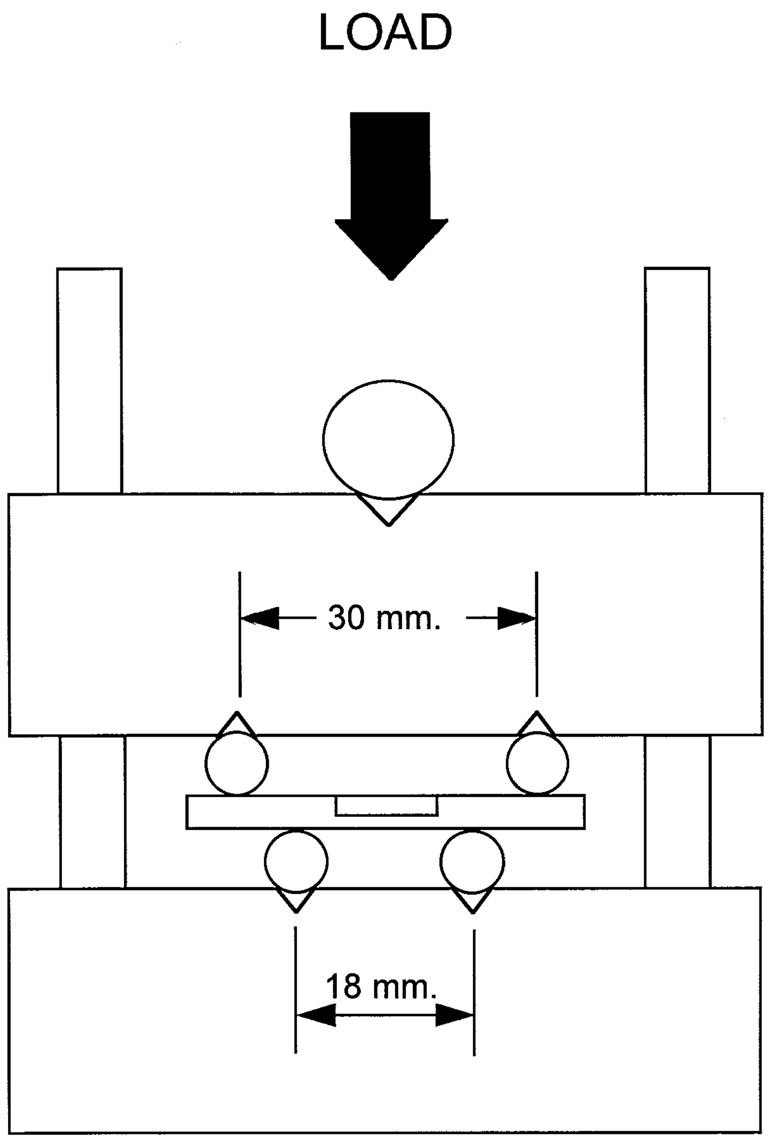


Figure 16. Four-Point Bending Fixture Dimensions.


LOAD
30 mm.
18 mm.

M. Refiring and Retesting of Samples

After experimentation, the fractured samples were refired through the glaze cycle, and these samples were then retested in the same manner as above. The fracture load data was recorded (Tables 9-11).

N. Statistical Analysis

Statistical analysis of the fracture data was accomplished using the Weibull distribution. The equation for the two parameter Weibull distribution is:

$$F(S) = 1 - e^{-(S/S_o)^m}$$

In which:

$F(S)$ is the cumulative probability of failure

S is the applied stress

S_o is the characteristic stress or scale parameter

m is the Weibull parameter or shape parameter

e is the exponential

The confidence intervals for the parameters were obtained from the tables by Thoman et al. (1969), assuming a 90% confidence level. The calculation of the parameters is based on the maximum likelihood estimates obtained with Newton-Raphson iteration. (Thoman et al., 1969; Mann, 1974; Abernethy et al., 1983; Huysmans, 1992; Scherrer and DeRijk, 1992).

TABLE 9

RETESTED DUCERATIN / REMATITAN FRACTURE LOAD DATA

Sample No.	Width (mm)	Total Thickness (mm)	Metal Thickness (mm)	Porcelain Thickness (mm)	**Fracture Load (N)**
1	8.7	3.4	1.5	1.9	91
2	8.7	3.2	1.4	1.8	51
3	8.6	3.0	1.3	1.7	-
4	8.8	3.1	1.4	1.7	-
5	8.7	3.1	1.4	1.7	-
6	8.8	3.3	1.4	1.9	-
7	8.8	3.2	1.4	1.8	-
8	8.6	3.1	1.4	1.7	61
9	8.6	3.0	1.3	1.7	-
10	8.7	3.1	1.4	1.7	-
11	8.6	3.2	1.4	1.8	-
12	8.7	3.2	1.4	1.8	56
13	8.7	3.1	1.4	1.7	-
14	8.8	3.2	1.4	1.8	81
15	8.7	3.6	1.6	2.0	-
16	8.8	3.4	1.5	1.9	116
17	8.8	3.3	1.4	1.9	-
18	8.9	3.3	1.4	1.9	-
19	8.8	3.2	1.4	1.8	-
20	8.9	3.3	1.4	1.9	-

\- Sample bond failure

TABLE 10

RETESTED VITA TITANKERAMIK / REMATITAN FRACTURE LOAD DATA

Sample No.	Width (mm)	Total Thickness (mm)	Metal Thickness (mm)	Porcelain Thickness (mm)	**Fracture Load (N)**
1	8.8	3.5	1.5	2.0	178
2	8.7	3.2	1.4	1.8	-
3	8.8	3.3	1.4	1.9	-
4	8.7	3.2	1.4	1.8	116
5	8.7	3.2	1.4	1.8	177
6	8.7	3.1	1.4	1.7	72
7	8.8	3.4	1.5	1.9	-
8	8.9	3.4	1.4	1.9	146
9	8.8	3.3	1.4	1.9	-
10	8.8	3.3	1.4	1.9	-
11	8.7	3.3	1.4	1.9	-
12	8.8	3.2	1.4	1.8	-
13	8.9	3.4	1.5	1.9	-
14	8.8	3.3	1.4	1.9	131
15	8.8	3.4	1.5	1.9	-
16	8.8	3.2	1.4	1.8	126
17	8.7	3.1	1.4	1.7	-
18	8.8	3.4	1.5	1.9	96
19	8.7	3.4	1.5	1.9	-
20	8.7	3.5	1.5	2.0	-

\- Sample bond failure

TABLE 11

RETESTED VITA VMK-68 / OLYMPIA FRACTURE LOAD DATA

Sample No.	Width (mm)	Total Thickness (mm)	Metal Thickness (mm)	Porcelain Thickness (mm)	**Fracture Load (N)**
1	8.7	3.3	1.3	2.0	158
2	8.9	3.4	1.5	1.9	192
3	8.9	3.3	1.4	1.9	134
4	8.9	3.3	1.4	1.9	292
5	8.9	3.3	1.4	1.9	150

IV. RESULTS

The bond strengths of all tested dental porcelain-fused-to-metal systems investigated are shown in Table 12 through Table 14 and graphically depicted in Figure 17. The bond strengths of all retested dental porcelain-fused-to-metal systems investigated are shown in Table 15 through Table 17 and graphically depicted in Figure 18. The mean bond strength values for all tested dental porcelain-fused-to-metal systems investigated are shown in Table 18 and graphically depicted in Figure 19.

The Duceratin-fused-to-Rematitan system had the highest mean bond strength value (46.4 MPa), the Vita Titankeramik-fused-to-Rematitan system (36.5 MPa) was next, and the Vita VMK-68-Fused-To-Olympia system (28.6 MPa) was lowest.

The mean bond strength values for all retested dental porcelain-fused-to-metal systems investigated are also shown in Table 18 and graphically depicted in Figure 19. Virtually all of the Duceratin-fused-to-Rematitan samples and the Vita Titankeramik-fused-to-Rematitan samples exhibited bond failure after refiring through the glaze cycle. The Vita VMK-68-fused-to-Olympia system (control) was not adversely affected by the reglazing process and had a mean bond strength value of 30.1 MPa.

The Weibull modulus (m) and characteristic strength (S_O) for the tested and retested groups are shown in Table 19. The cumulative probability of fracture is shown graphically in Figure 19.

TABLE 12

DUCERATIN / REMATITAN
BOND STRENGTH VALUES

Sample No.	Bond Strength (MPa)
1	35.5
2	52.2
3	49.0
4	45.6
5	49.5
6	29.6
7	57.4
8	68.0
9	73.4
10	37.2
11	56.2
12	39.0
13	59.0
14	33.0
15	49.8
16	51.5
17	43.5
18	26.7
19	46.1
20	25.4

TABLE 13

VITA TITANKERAMIK / REMATITAN BOND STRENGTH VALUES

Sample No.	Bond Strength (MPa)
1	32.8
2	26.9
3	35.6
4	30.6
5	33.7
6	31.7
7	38.2
8	33.8
9	39.6
10	39.4
11	53.6
12	33.8
13	35.3
14	39.7
15	33.7
16	40.1
17	35.9
18	45.2
19	35.3
20	35.1

TABLE 14

VITA VMK-68 / OLYMPIA BOND STRENGTH VALUES

Sample No.	Bond Strength (MPa)
1	30.1
2	25.1
3	29.8
4	22.4
5	35.5

TABLE 15

RETESTED DUCERATIN / REMATITAN BOND STRENGTH VALUES

Sample No.	Bond Strength (MPa)
1	14.4
2	9.1
3	-
4	-
5	-
6	-
7	-
8	11.7
9	-
10	-
11	-
12	10.0
13	-
14	14.3
15	-
16	18.1
17	-
18	-
19	-
20	-

\- Sample bond failure

TABLE 16

RETESTED VITA TITANKERAMIK / REMATITAN BOND STRENGTH VALUES

Sample No.	Bond Strength (MPa)
1	26.2
2	-
3	-
4	20.7
5	31.5
6	13.7
7	-
8	22.5
9	-
10	-
11	-
12	-
13	-
14	21.7
15	-
16	22.2
17	-
18	15.0
19	-
20	-

\- Sample bond failure

TABLE 17

RETESTED VITA VMK-68 / OLYMPIA BOND STRENGTH VALUES

Sample No.	Bond Strength (MPa)
1	26.4
2	29.6
3	21.9
4	47.8
5	24.5

Figure 17. Bond Strength Values: All Porcelain / Metal Systems.

BOND STRENGTH VALUES
ALL PORCELAIN / METAL SYSTEMS

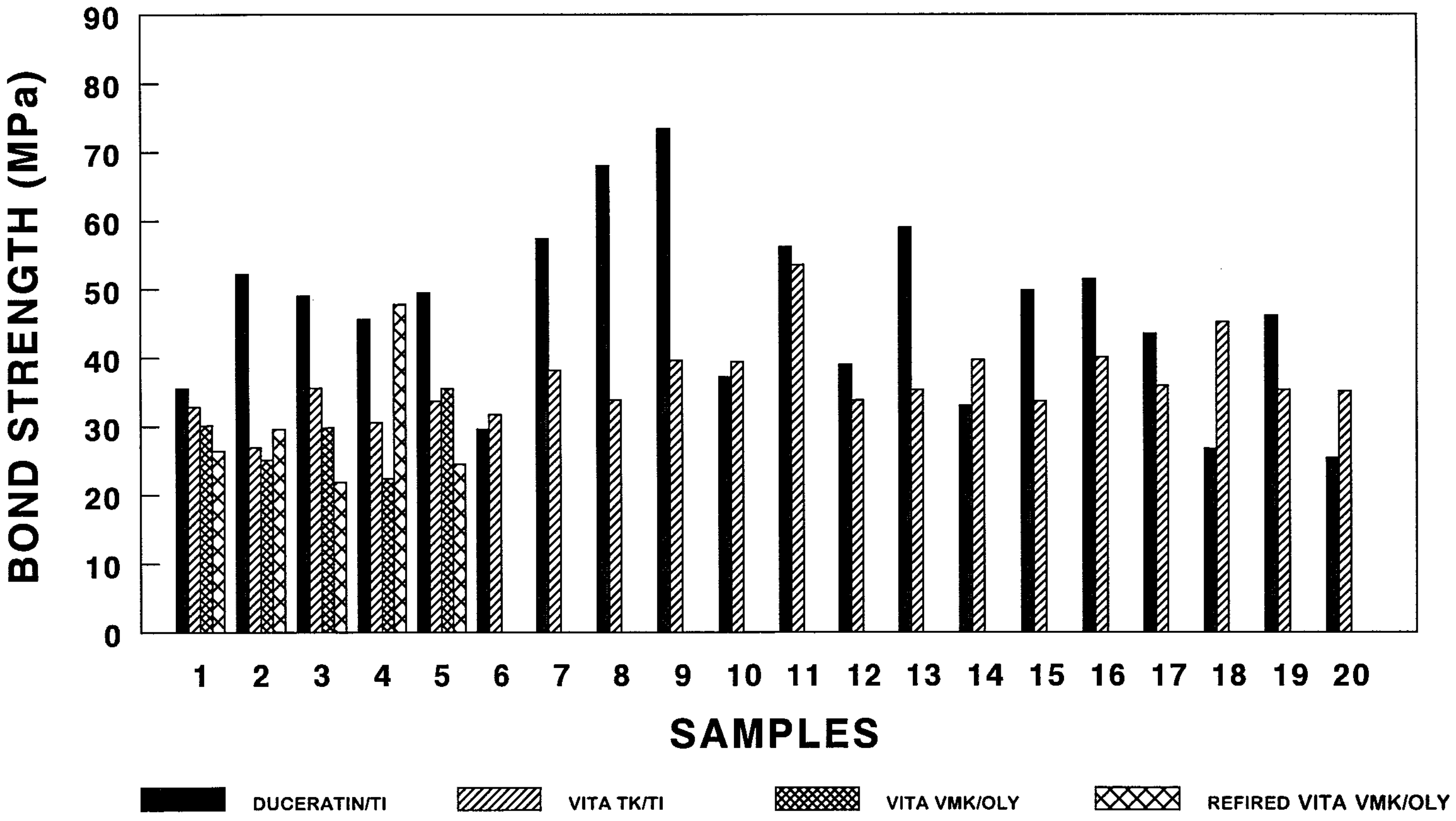


TABLE 18

MEAN BOND STRENGTH VALUES ALL DENTAL PORCELAIN / METAL SYSTEMS

Tested Group (Porcelain / Metal)	Number (n)	Mean Bond Strength (MPa)	Standard Deviation	Standard Error
Duceratin / Rematitan	20	46.4	13.0	2.9
Vita Titankeramik / Rematitan	20	36.5	5.7	1.3
Vita VMK-68 / Olympia	5	28.6	5.0	2.3

Retested Group (Porcelain / Metal)	Number (n)	Mean Bond Strength (MPa)	Standard Deviation	Standard Error
Duceratin / Rematitan	20	N\A	N/A	N/A
Vita Titankeramik / Rematitan	20	N/A	N/A	N/A
Vita VMK-68 / Olympia	5	30.1	10.3	4.6

Figure 18. Mean Bond Strength Values: All Dental Porcelain / Metal Systems.

MEAN BOND STRENGTH VALUES
ALL DENTAL PORCELAIN / METAL SYSTEMS

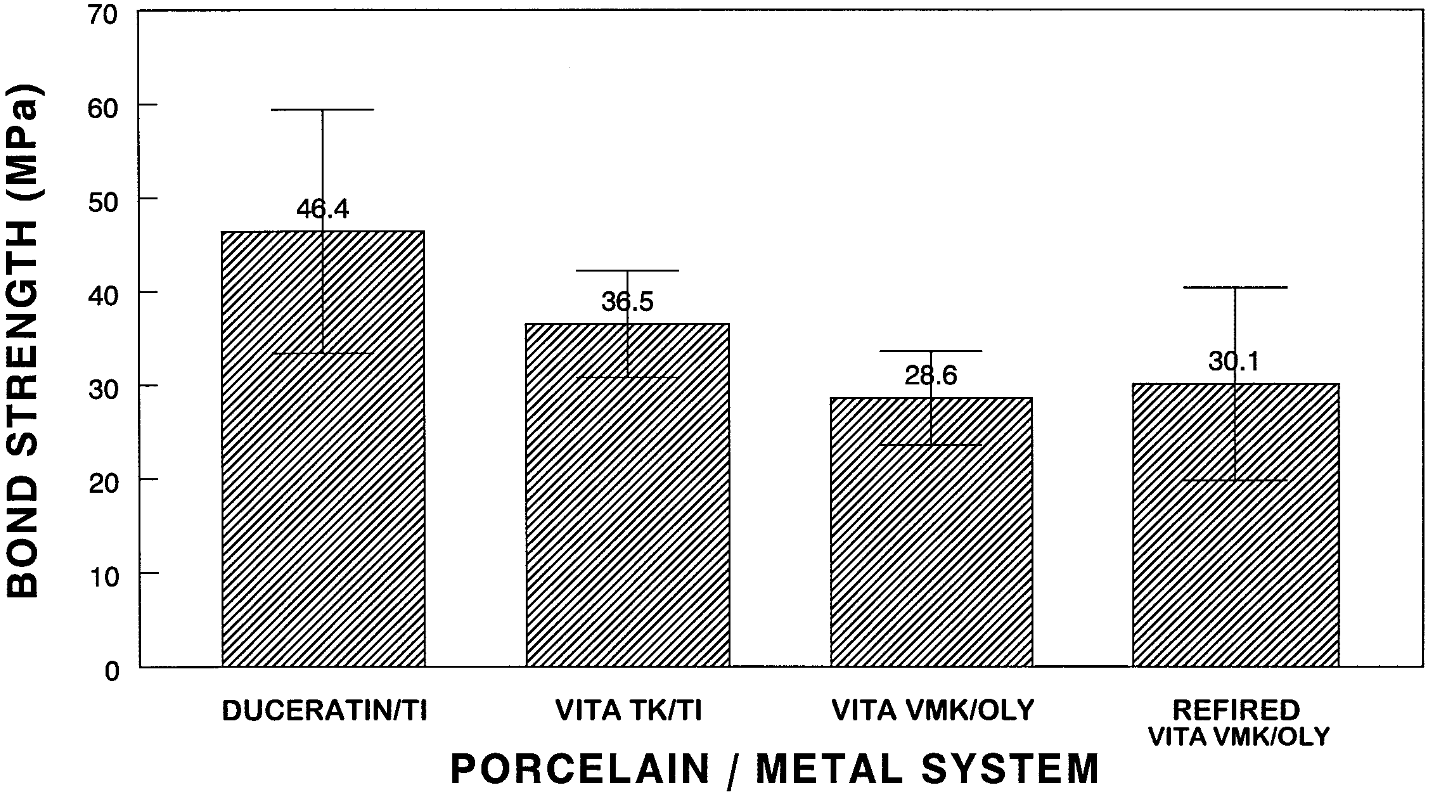


TABLE 19

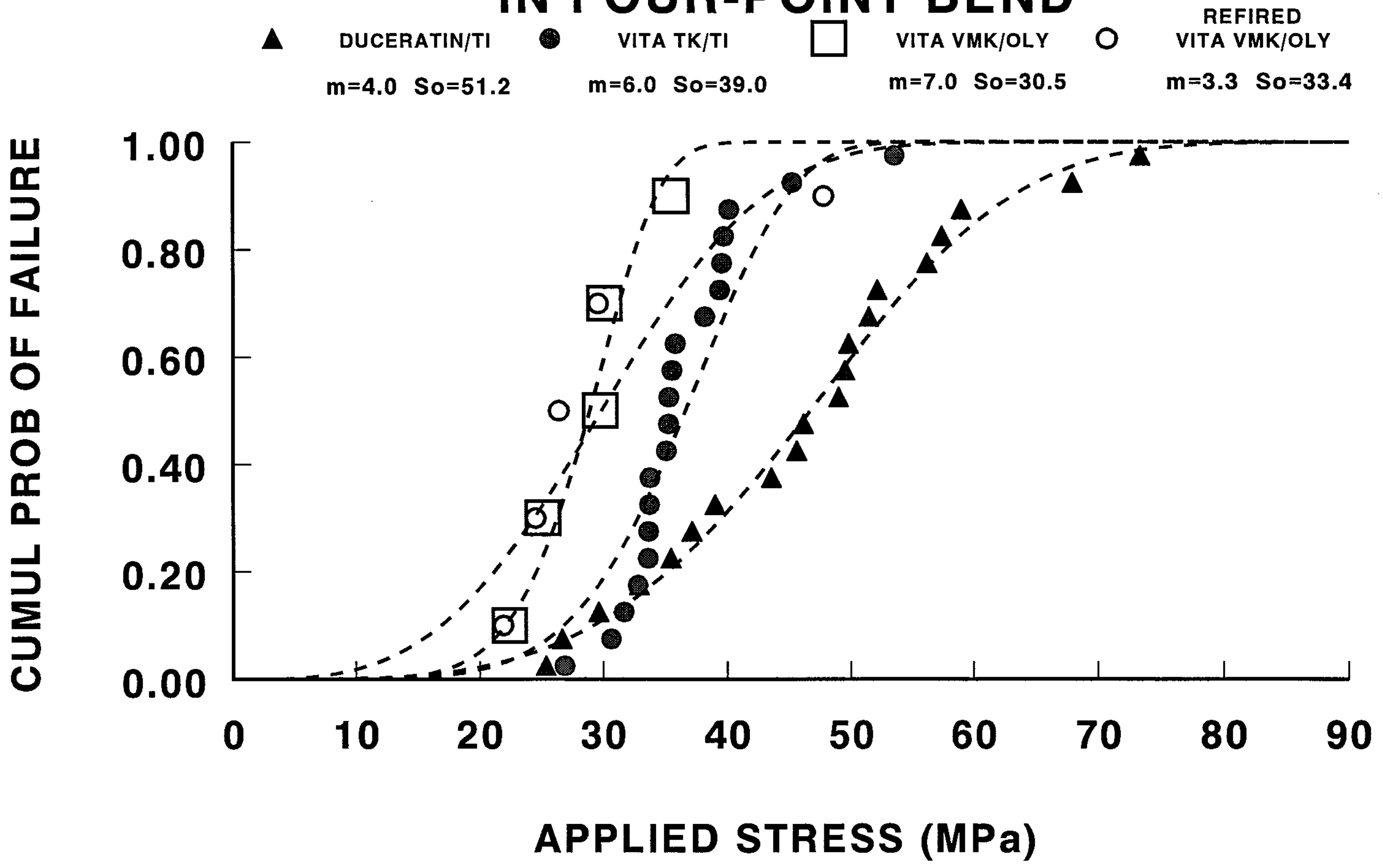
CONFIDENCE LIMITS FOR THE WEIBULL PARAMETERS AND THE MODULUS OF RUPTURE AT 90% CONFIDENCE

Tested Group (Porcelain / Metal)	Number (n)	Weibull Modulus (m)	Characteristic Strength (S_0) [in MPa]
Duceratin / Rematitan	20	2.8 < 4.0 < 5.1	46.0 < 51.2 < 56.9
Vita Titankeramik / Rematitan	20	4.1 < 6.0 < 7.6	36.3 < 39.0 < 41.8
Vita VMK-68 / Olympia (control)	5	2.5 < 7.0 < 10.3	25.5 < 30.5 < 35.7

Retested Group (Porcelain / Metal)	Number (n)	Weibull Modulus (m)	Characteristic Strength (S_0) [in MPa]
Duceratin / Rematitan	20	N/A	N/A
Vita Titankeramik / Rematitan	20	N/A	N/A
Vita VMK-68 / Olympia (control)	5	1.2 < 3.3 < 4.8	22.9 < 33.4 < 46.8

Figure 19. Cumulative Probability of Failure in Four-Point Bend.

CUMULATIVE PROBABILITY OF FAILURE IN FOUR-POINT BEND



V. DISCUSSION

This study investigated the bond strengths of two porcelain-fused-to-titanium (PFT) systems and one porcelain-fused-to-gold alloy (PFM) system. As presented in the literature review there are several tests that have been used for determining bond strengths of metal-ceramic systems. The four-point bending of composite beams as described by DeRijk et al. (1982) was the preferred testing methodology for this investigation because it determines the level of stress at which failure of the dental porcelain-to-metal system occurs, i.e. the failure strength; and, not the rupture strength which is a reflection of the porcelain fracture strength (PFS) or porcelain-metal bond strength (PMBS) of a porcelain-fused-to-metal system. The PFS is usually determined by modulus of rupture tests or bars of porcelain subjected to three or four point bending; and, the PMBS tests are mostly based on a determination of interfacial shear strength (Tesk et al., 1982). This property is extremely important clinically because the strength of the metal-ceramic system determines the service life of the restoration (DeRijk et al., 1982). The advantages of this testing methodology are that it: (1) evaluates the system in terms of brittle failure, i.e., in tension, (2) does not require absolutely precise alignment in the test fixture, (3) provides a direct evaluation of strength of the system, (4) avoids significant stress concentration effects due to wettability and spreadability, (5) minimizes stress concentrations due to loading and provides a uniform stress along the sample, through the PFM bond interface, (6) has relatively easy sample preparation, with porcelain opaque and body layers applied in the same manner as used in the dental laboratory, (7) has a thickness and mass of metal which approximates those found at some locations on a clinical crown or fixed partial denture, and (8) for efficiency provides more than one test result per sample, and (9) the normal tensile stress is essentially the same as the surface stress, when the ratio of the metal thickness underneath the porcelain component (c) to total thickness of the metal and porcelain component (t) is between 0.2 and 0.5 inclusive (Tesk et al., 1982) (Figure 6). In this investigation, the calculated n (modulus alloy/modulus porcelain) for all dental porcelain/metal systems ≈ 1.45, and the ratio of the metal thickness underneath the porcelain component (c=1.5mm) to total thickness of the metal and porcelain component (t=3.5mm) = c/t = 1.5mm/3.5mm = 0.43. The c/t value of 0.43 meets the criteria of being between 0.2 and 0.5 inclusive. Also, by looking at the graph in Figure 6 we can locate the c/t value of 0.42 on the x-axis, construct a line perpendicular to the x-axis until it intersects the top curve of n = 1.5, and then construct a line parallel to the x-axis until it intersects the y-axis designating a δ_n (variation of normalized maximum tensile stress) ≈ 0.92. This means that the stress in porcelain is ≈ 92% of that in the adjacent metal section during testing.

A Weibull statistical analysis was performed to analyze the fracture load data. This analysis provides a graphical solution by the process of plotting a curve and analyzing it (Figure 19). The horizontal scale (x-axis) is the independent variable (applied stress in this case), and the vertical scale (y-axis) is the probability of failure. The slope of the line (m) is extremely significant and may provide a suggestion towards the physics of the failure in question. Also, this statistical analysis works well with small sample groups (Abernethy et al., 1983). The mean bond strengths of the dental porcelain/metal systems were calculated to show that these values are the same as the characteristic strengths seen in the Weibull distribution at 50% probability of failure (Figure 19). This is accomplished by locating the 0.50 probability of failure on the y-axis and constructing a line perpendicular to the y-axis until it intersects the plotted curve of the respective dental porcelain/metal system, and then constructing a line parallel to the y-axis until it intersects the x-axis designating the applied stress (MPa) for that respective system. "The confidence level of 90% is customarily selected for data that conforms to the Weibull distribution and is lower than would be expected based on experience with the normal or Gaussian distribution. The normal distribution is symmetric about the mean, so the distribution becomes well defined even with relatively few data points, which translates into a (often artificially) narrow confidence interval. In contrast, the Weibull probability distribution does not have this requirement of symmetry (i.e., not bell shaped), hence a wider confidence interval is found. The width of the confidence interval is entirely determined by the number of specimens. For small sample sizes (N < 25) the 90% level is empirically meaningful. When the confidence intervals for two data sets do not overlap, then the difference is called statistically significant at the 81% level of confidence. If the confidence levels do overlap, no definitive statement can be made at this confidence level" (Scherrer and DeRijk, 1992). In Figure 19 we can see that the plot for Duceratin/Ti has the highest characteristic strength; however, the values for applied stress (MPa) fall within a wide range from 25.4-73.4. Although the higher characteristic strength is desirable, the wide range is not. A smaller range of applied stress values as seen in the other dental porcelain/metal systems is actually more desirable (Vita Titankeramik/Ti = 26.9-53.6 MPa; Vita VMK-68/Oly = 22.4-35.5 MPa; Refired Vita VMK-68/Oly = 21.9-47.8 MPa).

The Duceratin-fused-to-Rematitan had the highest mean bond strength value (46.4 MPa) of all the systems tested. The Vita Titankeramik-fused-to-Rematitan had a mean bond strength value of 36.5 MPa. These mean bond strength values are similar to other such values being reported in the literature. Derand and Hero (1992) reported Duceratin mean bond strength values of 13 MPa (using Goldbond), 28 MPa (sandblasted with 50 μm Al_2O_3), and 52 MPa (sandblasted with 250 μm Al_2O_3) using their four-point testing methodology. They also found the mean bond strength value of Ohara porcelain to be 47 MPa (sandblasted with 250 μm Al_2O_3). The manufacturer's of Duceratin developed Haftbond powder for their porcelain system to enhance the bonding between Duceratin and titanium. The Haftbond enters the titanium surface structure sealing the surface and consequently the oxides within. Ducera reported shear test (DIN 13927) results of 26 N/mm2 (MPa) without Haftbond and 33 N/mm2 (MPa) with Haftbond (Ducera, 1991).

Baez (1994) reported on the bond strengths of four titanium porcelains fused to pure titanium using a push-shear test mode. His results were as follows (in MPa): Tibond 25.4 (2.6), Titanbond 21.9 (1.7), Duceratin 22.5 (3.3), and Vita Titankeramik 23.1 (4.3). Sommer et al. (1991) reported similar results for Biodent, Duceram, and Vita using a three- point bend test.

During early pilot studies, titanium castings were frequently obtained with sizable porosities in the area of sprue attachment. This was probably due to improper burnout and mold cracking with first generation Rematitan phosphate bonded investment. A more programmable furnace (KaVo EWL Furnace, West Germany), and the use of a second generation Rematitan Plus phosphate bonded investment eliminated the problem of large porosities, however, small porosities in castings continued to exist. Prasad et al. (1994) reported differential pressure casting machines produce castings with porosity because of the following reasons: (1) the inert gases, used to maintain inert atmosphere, invariably got drawn in with the flow of the molten metal resulting in the internal entrapment of gases, (2) the high velocity of molten metal created turbulence-voids which led to porosity and miscasts, and (3) the molten metal had a tendency to aspirate upon entry into the mold.

A group of pilot study samples were made according to manufacturer's recommendations with the exception that 25μm Al_2O_3 was used for the sandblasting step. A bond was not obtained in any of the samples that were sandblasted with 25μm Al_2O_3. Visual inspection of these samples revealed the entire surface of the delaminated porcelain component to be covered with a gray oxide layer. Kimura et al. (1990) reported an excess layer of TiO_2 may weaken the porcelain-titanium bond strength. Adachi et al. (1990) reported lower adherence of low-fusing porcelain to titanium when the oxide film reaches approximately 1μm thickness.

Seventy percent (14/20) of the Duceratin/Rematitan samples and sixty percent (12/20) of the Vita Titankeramik/Rematitan samples were observed to have bond failures due to delamination during testing. One hundred percent (5/5) of the Vita VMK-68/Olympia samples were observed to have Class III bond failures due to fracture within the porcelain component during testing.

The Vita VMK-68-fused-to-Olympia system had the lowest mean bond strength value (28.6 MPa) of the systems tested; however, this value is comparable with the reported values for similar metal-ceramic systems. O'Brien (1989) reported the bond strength in tension of porcelain-fused-to-metal systems to be about 35 MPa.

Buchness et al. (1988) reported that after experimentation using the methodology described above, refiring of the fractured samples through the glaze cycle did not appear to have a major negative effect on the PFM bond strength. In this study, both dental porcelain-fused-to-titanium metal systems exhibited bond failure after refiring them through the glaze cycle. This bond failure may have been due to metal surface oxidation during firing at high temperatures. Since most bond failures were accompanied by the delamination of porcelain from the metal, this could also explain why the refiring process did not reconstitute the adhesion between the porcelain and metal. Kononen and Kivilahti (1994) reported: (1) when SiO_2-based dental porcelains and commercially pure titanium are in contact at firing temperatures of 720-750°C for a given time, the reaction zone consists of a two-layer structure containing an oxygen-rich Ti-O solid solution and an oxygen-containing silicide, most probably $Ti_5Si_3(O)$, (2) preoxidation of titanium to increase the bond strength of porcelain fused to titanium is not recommended, and (3) it is likely that the more firing cycles will result in thicker reaction layers and thus worsen the titanium-ceramic bond.

Clinically, if one were adjusting the occlusion of a PFT crown or fixed partial denture that broke the glaze, it may be preferred to polish the porcelain surface rather than refire the prosthesis through the glaze cycle.

Refiring the Vita VMK-68-fused-to-Olympia through the glaze cycle actually showed an increase in mean bond strength value (30.1 MPa). Unlike the delamination of most PFT samples, all of the bond failures of the porcelain-fused-to-gold alloy samples were cohesive within porcelain (Class III). Therefore, refiring these samples apparently repaired the fracture within the porcelain component which allowed for successful retesting of the samples.

The future of cast titanium restorations is bright, but there are still many questions to be answered before titanium can be considered to be the material of choice in the restoration of the coronal portions of the dentition. Existing titanium casting technology is expensive, yet, narrow-angle crown coping margins cannot be accurately cast (Blackman et al., 1992). Intraoral welding and laboratory soldering technology is currently available (Hruska and Borelli, 1991), but this is also expensive. Further research in areas such as sprue design (Verrett and Duke, 1989), investments (Burkett et al., 1995), and titanium's wear characteristics is recommended before titanium becomes the clinical metal of choice for prosthodontic restorations of the future.

VI. SUMMARY

The following conclusions can be made from the results of this investigation:

1. The bond strength of Duceratin-fused-to-Rematitan was the highest of the systems investigated.
2. The difference between the bond strength of Duceratin-fused-to-Rematitan and Vita Titankeramik-fused-to-Rematitan was statistically significant (81% confidence).
3. The bond strength of Vita VMK-68-fused-to-Olympia (control) was the lowest, but comparable with the other reported values for metal-ceramic systems.
4. Glaze cycle refiring did not adversely affect the control group; and, the increase in bond strength was not statistically significant (81% confidence).
5. Both porcelain-fused-to-titanium systems exhibited bond failure after the glaze cycle refiring process. This may be due to oxygen or oxide contamination of the metal at high temperatures.
6. More research is needed in the areas of casting variables, investments, and wear characteristics.

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VITA

Jose M. Gutierrez, III, was born on September 25, 1953, in San Antonio, Texas. He is the first born of Jose M. Jr. and Faye C. Gutierrez. His parents were born and raised in Hebbronville, Texas, and moved to San Antonio after their marriage in 1952.

He attended Thomas Jefferson High School, San Antonio, Texas, and graduated in 1971. After high school, he attended San Antonio Junior College, San Antonio, Texas, from July 1971-July 1972. He then transferred to the University of Texas at Austin, Austin, Texas, graduated and was awarded a B.A. with honors on May 15, 1975. Afterwards, he attended UTHSCSA Dental School, San Antonio, Texas, graduated and was awarded his D.D.S. on May 19, 1979.

After two years of private practice in San Antonio and Kenedy, Texas, he entered the United States Air Force (USAF) in July 1981, and was assigned to Wright-Patterson AFB, Ohio. In September 1984 he was re-assigned to Torrejon Air Base, Spain. In September 1987 he was re-assigned to Wilford Hall Medical Center, Lackland AFB, Texas. He served there until entering the USAF graduate training program in Prosthodontics on July 1, 1992.

On July 16, 1994, he and Lisa R. Burkett were married. At this time, he is serving in the grade of Lieutenant Colonel. Presently, he is in his third year of graduate training in Prosthodontics at Wilford Hall Medical Center, Lackland AFB, TX.