Uniformed Services University of the Health Sciences
Manuscript/Presentation Approval or Clearance

Initiator	
1. USU Principal Author (Last, First, Middle Initial)	Bhend, Carl, P
2. Academic Title	AEGD-2 Resident
3. School/Department/Center	Kessler Dental Clinic
4. Phone 228-376-5118	5. Email carl.bhend.1@us.af.mil
6. Clearance	☑ Paper ☑ Article ☐ Book ☑ Presentation ☐ Other
7. Title	Selected Physical Property Evaluation of Contemporary Composite Resin Core Materials
8. Intended Publication/Meeting	International Association for Dental Research
9. Required by 22 Mar 2017	10. Date of Submission 4 Jan 2017

**Note: It is DoD policy that clearance of information or material shall be granted if classified areas are not jeopardized, and the author accurately portrays official policy, even if the author takes issue with that policy. Material officially representing the view or position of the University, DoD, or the Government is subject to editing or modification by the appropriate approving authority.

☑ Neither I nor any member of my family have a financial arrangement or affiliation with any corporate organization offering financial support or grant monies for this research, nor do I have a financial interest in any commercial product(s) or service(s) I will discuss in the presentation or publication.

☑ **The following statement is included in the presentation or publication:** The opinions or assertions contained herein are the private ones of the author(s) and are not to be construed as official or reflecting the view of the DoD or the USUHS.

☑ **The following items have been included in the presentation and/or publication:** Student and/or faculty USU affiliation. Examples: 1) LCDR Jane Doe, DMD, Resident, Naval Postgraduate Dental School and Uniformed Services University of the Health Sciences Postgraduate Dental College. 2) COL John Doe, DDS, Endodontics Program Director, Fort Bragg, NC and Associate Professor of Endodontics, Uniformed Services University of the Health Sciences Postgraduate Dental College. 3) USUHS logo included on title slide and/or poster

Chair/Department Head Approval**	
Name (Last, First, Middle Initial)	Wajdowicz, Michael, N
Signature	[redacted]

Commander Approval (if applicable)**	
Name (Last, First, Middle Initial)	
School	
Higher approval clearance required (for University- DoD, or US Gov't-level policy, communications systems or weapons review	
Signature	

Uniformed Services University of the Health Sciences
Manuscript/Presentation Approval or Clearance

Service Dean Approval**	
Name (Last, First, Middle Initial) Fallis, Drew, W	
School Air Force Postgraduate Dental School	
Higher approval clearance required (for University-, DoD, or US Gov't-level policy, communications systems or weapons review)	
Signature	
Executive Dean Approval**	
Name (Last, First, Middle Initial)	
Higher approval clearance required (for University-, DoD, or US Gov't-level policy, communications systems or weapons review)	
Signature	
Vice President for External Affairs Action	
Name (Last, First, Middle Initial)	
☐ USU Approved	☐ DoD Approval Clearance Required
☐ Submitted to DoD (Health Affairs) on	
☐ Submitted to DoD (Public Affairs) on	
☐ DoD Approved/Cleared (as written)	☐ DoD Approved/Cleared (with changes)
DoD Clearance Date	DoD Disapproval Date
Signature	

The author hereby certifies that the use of any copyrighted material in the thesis/dissertation manuscript entitled:

"Selected Physical Property Evaluation of Current Resin Composite Core Buildup Materials"

is appropriately acknowledged and, beyond brief excerpts, is with the permission of the copyrighted owner.

[redacted]

Carl P. Bhend
AEGD 24 Month Residency Program
Air Force Postgraduate Dental School
Uniformed Services University
7 July 2017

Distribution Statement

Distribution A: Public Release.

The views presented here are those of the author and are not to be construed as official or reflecting the views of the Uniformed Services University of the Health Sciences, the Department of Defense or the U.S. Government.

Selected Physical Property Evaluation of Current Resin Composite Core Buildup Materials

Carl P. Bhend

Selected Physical Property Evaluation of Current Resin Composite Core Buildup Materials
Major Carl P. Bhend

APPROVED:

[redacted]

Colonel Michael N. Wajdowicz

[redacted]

Major James J. Renda

[redacted]

Lieutenant Colonel John A. Brewster (Chairman)

6 JULY 2017

Date

APPROVED:

Col Drew W. Fallis
Dean, Air Force Postgraduate Dental School

Acknowledgements

Special thanks to Col Howard Roberts, Col Michael Wajdowicz, Lt Col John Brewster, and Lt Col Nicholas DuVall.

The author hereby certifies that the use of any copyrighted material in the thesis manuscript entitled:

<u>Selected Physical Property Evaluation of Current Resin Composite Core Buildup Materials</u>

is appropriately acknowledged and beyond brief excerpts, is with the permission of the copyright owner.

____

Signature

____

Printed Name

____

USAF Postgraduate Dental School
Keesler AFB, MS
Uniformed Services University

Table of Contents

LIST OF TABLES

LIST OF FIGURES

Abstract

Objective: The aim of this study was to evaluate the short beam shear strength (SBSS) of resin composite core materials as a function of mode of cure as well as time after cure. The null hypothesis was that there would be no difference in SBSS of the materials evaluated.

Methods: Fourteen resin composite core materials were evaluated at post cure times of 10 minutes, one hour, and 24 hours. Furthermore, dual cure materials were evaluated using both curing strategies while single mode of cure materials were either cured with a LED visible light curing unit or self cure as required. Specimens (n=12) were fabricated using a standardized 5 x 5 x 25mm Teflon mold by placing the mold over a mylar covered glass slab. Materials were prepared as per manufacturer recommendations and was placed into the mold, which was immediately covered by a second mylar strip and a glass laboratory slide with digital pressure applied to form a flat superior surface. Dual cure materials were then photopolymerized in an overlapping fashion on the superior surface while self-cure mode specimens were held with a vise and placed into a chamber with 37 ± 2 °C and 98 ± 2 percent humidity phosphate buffered saline environment. At the appointed time of testing specimens were stressed until failure on a universal testing machine using a three point bend fixture at a cross head speed of 0.5mm. Mean results were analyzed with Kruskal-Wallis/Dunn's post hoc analysis (p=0.05).

Results: SBSS increased with all materials with time, regardless of the cure mode. Light activated dual-cure materials demonstrated higher physical properties as compared to the self-cure mode. Overall, materials demonstrated greater strength development between one and 24 hours.

Conclusions: The highest SBSS values were observed at 24 hour test period with photopolymerized dual-cure materials demonstrating greater physical properties as compared to the self-cure mode. Clinicians should be aware that resin composite core materials prepared at 10 minutes after curing has inferior SBSS as compared to 24 hours.

INTRODUCTION

Clinicians are often challenged with the restoration of the extensively damaged dentition. When insufficient tooth structure exists to possess adequate retention and resistance features, the placement of an appropriate material is placed to compensate for lost tooth structure while also serving as a foundation for a full-coverage restoration. This coronal material, especially when serving as crown foundations, is referred as a core material. Suitable core materials can be directly placed using restorative materials or indirectly luted using laboratory casting techniques. Regardless of the material, for successful function the core should possess certain characteristics, to include stability in a wet environment; ease of manipulation; rapid, hard set for immediate preparation for a full-coverage restorations; and natural tooth color. [1] Optimal core material physical properties would include high compressive and tensile strength; high modulus of elasticity as well as high fracture toughness. Other desired features would be a material that would be both inert and biocompatible, cariostatic, and low cost. [1] While a core material may contain some of these physical properties and characteristics, unfortunately no known material possesses all of these ideal parameters.

Historically, amalgam has been the most common core material. Currently adhesively bonded resin composite is the most common directly placed core material, as it also allows the opportunity for almost immediate preparation for a subsequent full-coverage restoration. [2]

Dental resin composite materials cure either polymerization initiation with visible light or with chemical means (e.g., "self" or "dark" cure). Some materials contain both a visible light cure (VLC) and self-cure mechanisms which is described as a dual-cure material. Dual-cure materials are marketed for situations wherein the core material depth is greater than the usual 2 millimeter depth of cure limitation for visible light activation.

Multiple resin composite core materials have been marketed with little independent evaluation of their physical properties. The physical properties usually reported for core materials are that seen at the standard 24 hours after fabrication. Notwithstanding, core materials are marketed as possessing the ability to withstand preparation almost immediately after curing. Little is known about core material physical properties immediately after curing. The purpose of this study was to evaluate the strength of resin composite core materials at various times utilizing a short-beam shear strength (SBSS) methodology. The null hypothesis was that there will be no difference in SBSS of the materials evaluated.

Methods and Materials

The materials used in this study are presented in Table 1.

Table 1. Materials Used

Material	Cure Mode	Depth of Cure (mm)	Curing Time VLC/Self	Preparation Time After Cure	Filler Content	Manufacturer Information
Build-It FR A2	Dual	NA	40s per surface/4m	Immediately	Chopped glass fiber	Pentron America, Orange, CA, USA
Build It Blue	Self	Self cure	4m	Immediately	Chopped glass fiber	Pentron America
Clearfil DC Core Plus	Dual	2mm	20s/6m	Immediately	Silica/Barium Glass 74%/volume	Kuraray America New York, NY USA
Clearfil Photocore	VLC	7mm	40s	Immediately	Silica/Barium Glass 65%/volume	Kuraray America
Fluorocore Blue	Dual	3mm	40s/7-8m	VLC: immediately; self cure 10m	Barium/Silica Glass Up to 70% volume	Dentsply Sirona, York, PA, USA
Fluorocore TC	Dual	3mm	40s/7-8m	VLC: immediately; self cure 10m	Barium/Silica Glass Up to 70% volume	Dentsply Sirona
Gradia Core	Dual	2.9mm	10s/5m	Immediately	NA	GC America, Alslp, IL USA
Multicore Flow Blue	Dual	NA	VLC 20s per surface/ Self-cure 4-5 min	VLC: immediately Self-cure 5m	Barium/Silica 54% volume	Ivoclar Vivadent America, Amherst, NY, USA
Multicore Flow TC	Dual	NA	VLC 20s per surface/ Self-cure 4-5 min	VLC: immediately Self-cure 5m	Barium/Silica 54% volume	Ivoclar-Vivadent
Rebilda Blue	Dual	2mm	40s/self cure 5m	VLC: immediately Self-cure 5m	NA	Voco America, Indian Land, SC, USA
Rebilda TC	Dual	2mm	40s/self cure 5m	VLC: immediately Self-cure 5m	NA	Voco America
TiCore Auto E	Dual	4mm	40s/self cure 6m	VLC immediately Self cure NA	Lathanide series elements	Essential Dental Systems America, South Hackensack, NJ, USA
TiCore Flow +	Dual	4mm	40s/self cure 6m	VLC immediately Self cure NA	Lathanide series elements	Essential Dental Systems
TiCore Gray	Self	Self cure	NA	NA	Lathanide series elements	Essential Dental Systems

All information obtained from manufacturer literature. NA = not available. Filler content is noted when information supplied

The 14 materials were classified into three groups based on curing mode, namely dual, self-cure, and VLC. One material would be investigated as VLC only, two materials as self-cure only, and the other 11 materials would be dual-cure. The dual-cure materials were further subdivided into two curing modes, VLC and self-cure. Each material was tested at 10 minutes, 1 hour, and at 24 hours after curing. Twelve specimens per material was fabricated for each testing time.

For specimen fabrication, a standardized 5 x 5 x 25 millimeter Teflon mold was placed on a mylar-strip-covered glass slab. The prepared material was placed into the mold and immediately covered with a second mylar strip followed by placement of a glass microscope slide using digital pressure to form a flat surface. The entire assembly of the self-cure materials were clamped together and allowed to sit in a 37 °C incubator for the indicated time for curing. The light cure material was similarly prepared with the superior surface irradiated in an overlapping fashion for 40 seconds using a multi-mode LED-based, VLC unit (Bluephase G2; Ivoclar-Vivadent, Amherst, NY, USA). The VLC-activated dual-cure materials were fabricated in the same fashion as the light cured material while the dual-cure specimens designated for self cure were fabricated identically as the self-cure materials. The philosophy of curing molds and times were based on a possible worst-case scenario of core materials thicker than usually prescribed by the manufacturer.

After polymerization and/or self-cure, specimens were removed from the mold and refined as needed using surgical scalpels to remove flash materials. Specimens were stored in physiologic phosphate buffered saline solution at 37°C until the appointed testing period. At the appointed testing period, specimens were placed on a three point bend apparatus mounted on a universal testing machine (Alliance RT/5, MTS Corporation, Eden Prairie,MN, USA). Specimens were stressed using a cross head speed of 0.5mm/min until failure with the maximum force obtained recorded. SBSS was determined using the formula:

SBSS = 3 Fl where F = maximum load recorded (in Newtons)

2 bh2 l = distance between supports (in mm)

b = width of specimen (in mm)

h = height of specimen (in mm)

Mean data was first analyzed with the Kolmogorov-Smirnov Test and Bartlett's test which identified both a non-normal distribution and inhomogeneity within the variances, respectively. The mean data Kruskal-Wallis Test with Dunn's post hoc analysis was performed at a 95 percent level of confidence ($p = 0.05$).

Results

SBSS mean results are listed in Table 2.

Table 2: Short Beam Shear Strength (MPa)						
Materials Evaluated	VLC/Dual-Cure VLC Activated			Self-Cure/Self-Cured Dual-Cure		
	10 minutes	1 hour	24 hours	10 minutes	1 hour	24 hours
Build-It FR A2	92.2 (12.5) BC b	96.0 (13.4) AB b	122.4 (17.1) BC c	73.1 (10.1) A a	81.3 (15.9) AB ab	120.2 (15.0) AB c
Build It Blue*	N/A	N/A	N/A	72.8 (10.1) A a	89.7 (8.1) ABC b	106.2 (16.3) BC c
Clearfil DC Core Plus	80.8 (8.1) CD bc	99.5 (7.9) AB de	120.4 (20.1) B e	45.1 (9.3) C a	67.2 (11.3) C b	95.0 (19.1) C cde
Clearfil Photocore**	113.6 (13.3) A a	122.0 (8.1) A a	146.4 (14.2) A b	N/A	N/A	N/A
Fluorocore Blue	90.3 (10.9) BC b	94.2 (11.3) B bc	113.7 (16.1) BC c	68.0 (11.2) AB a	91.3 (15.3) AB bc	105.6 (17.5) BC bc
Fluorocore TC	74.2 (11.2) D a	100.8 (16.3) B bc	118.3 (13.0) BC c	73.1 (11.0) A a	90.7 (13.2) AB ab	119.1 (10.1) AB c
Gradia Core	88.76 (8.3) BC b	118.9 (13.4) AB cd	130.1 (5.2) AB d	44.0 (8.6) C a	79.1 (9.3) BC b	108.4 (22.3) ABC c
Multicore Blue	108.5 (8.7) A b	105.4 (14) B b	137.5 (13.5) AB c	76.1 (17.0) A a	102.9 (14.6) A b	131.1 (14.8) A c
Multicore TC	101.3 (12.9) AB bc	110.3 (10.3) AB c	128.3 (13.3) AB d	65.7 (8.3) AB a	90.1 (7.3) AB b	127.3 (11.4) AB d
Rebilda Blue	105.3 (18.0) AB bc	103.2 (17.9) AB c	134.3 (12.4) AB d	72.3 (15.4) A a	83.8 (16.2) ABC ab	128.8 (20.2) AB d
Rebilda TC	100.8 (12.5) AB b	110.5 (17.2) AB b	137.6 (18.2) AB c	69.2 (12.7) AB a	99.7 (18.6) AB b	107.8 (28.7) ABC c
TiCore Auto E	97.0 (10.1) ABC c	99.2 (7.9) AB cd	111.4 (9.1) BC d	56.6 (9.2) BC a	79.7 (9.7) BC bc	107.5 (13.5) ABC cd
TiCore Flow +	100.3 (7.9) AB b	101.1 (15.5) AB b	122.9 (12.4) BC c	71.8 (8.5) AB a	69.3 (9.8) C a	105.9 (12.7) BC b
TiCore Gray*	N/A	N/A	N/A	49.0 (9.6) C a	69.2 (9.8) C b	70.8 (15.1) b

n = 12. Groups with same capital letter statistically similar within each column. Lower case letters annotate similarity within each treatment group row (Kruskal Wallis/Dunn's p=0.05)
*Denotes Self-Cure Only, **Denotes Light-Cure Only.

All materials exhibited increased SBSS with time, regardless of activation method. Overall there was considerable overlap of similar groups at all time/activation periods with no one material demonstrating significant higher results over the majority of materials. Of the light-activated and light-activated dual-cure materials, only seven materials (Build-It FR; Clearfil Photocore; Multicore Blue and TC; Rebilda Blue and Rebilda TC; TiCore Flow+) demonstrated a significantly greater SBSS strength at 24 hours as compared to one hour. Of the self-cure or the dual-cure with self-cure mode, all materials except three (Fluorocore Blue, Ticore Auto E, and Ticore Gray) were noted to exhibit greater SBSS at 24 hours as compared to one hour.

When reviewing global results in Table 3, it is interesting to note when the 24-hour SBSS results are compared with the dual-cure materials, there was not statistical SBSS difference between light-activated dual-cure and the self-cured dual cure materials.

Table 3. SBSS by Curing Modes Global Comparison

Test Time and Cure Mode	SBSS (MPa)
10 Minutes VLC/Dual-Cure VLC	91.1 (20.1) BC
10 Minutes Self-Cure/Dual Self Cure	64.4 (15.4) D
1 Hour VLC/Dual-Cure VLC	97.5 (20.0) ABC
Hour Self-Cure/Dual Self Cure	84.7 (16.6) CD
24 Hour VLC/Dual-Cure VLC	121.5 (22.5) A
24 Hour Self-Cure/Dual Self-Cure	110.3 (26.8) A

n = 144; Capital letters identify similar groups (Anova/Tukeys p=0.05)

Discussion

This evaluation investigated resin composite direct core materials physical strength at thickness described as the depth of cure by the manufacturer's promotional literature. The traditional flexure testing methodology is described in ISO 4049 which requires a length to thickness ration of 10 to 1 for the proper development of internal forces for flexure testing. However, core material flexure testing at thicknesses usually described as the visible light depth of cure would equate a length of 50 millimeters for a five millimeter thick sample. This would require an inordinate amount of material whose cost would be prohibitive for an evaluation containing adequate sample size.

This study utilized an alternative testing method, the Short Beam Stress Shear (SBSS) Test. The American Society for Testing and Materials (ASTM) in Designation 2344 describes SBSS as utilizing a three-point bend for the testing of high-modulus laminar composites up to six millimeters in thickness. [5] Although extensively utilized for laminar-veneered composite materials, the SBSS has been reported in the testing of nano-composite resins. [6]

The methodology used in this evaluated utilized the fabrication of five millimeter thick and wide beams at a length of 25 millimeters. Although direct resin core materials have various described depths of cure, the five-millimeter specimen depth was chosen as the standard testing thickness. This was designed to serve as a worst case scenario clinical thickness materials with four millimeter or less described VLC curing depth, but would also allow for evaluation of material's self-cure ability for clinical situations. The visible light curing duration of forty seconds was also chosen as to provide the most optimal curing conditions at the five-millimeter depth.

Specimens were tested at ten minutes, 1 hour, and 24 hours. While some manufactures state that their materials may be prepared immediately after curing, specimens were tested at ten minutes to allow the optimal development of early physical properties. Testing at one hour was accomplished to evaluate the magnitude of the physical property development at that time. Testing at 24 hours was performed to determine the most optimal physical properties.

For the VLC cured or activated dual-cure materials, all materials displayed a numerical trend with increasing SBSS, however only Clearfil DC Core Plus, Fluorocore TC, and Gradia Core significantly increased in strength between ten minutes and one hour. Between one and 24 hours, all materials demonstrated a significant increase in SBSS, except for Clearfil DC Core Plus, Fluorocore Blue, Fluorocore TC, Gradia Core, and TiCore Auto E.

When the self-cured materials or the dual-cure materials under self-cure mode are considered, all materials demonstrated a significant increase in SBSS between ten minutes and one hour except for Fluorocore TC, Rebilda Blue, and TiCore Flow +. Furthermore, between one hour and 24 hours all materials displayed a significant increase in strength except for Fluorocore Blue, Ticore Auto E, and Ticore Gray. Collectively, one should be cautioned when inferring the efficiency of material's self-cure mechanism as the SBSS method, like flexure, can be altered by porosity within the materials. The ultimate method for evaluating efficiency of self-cure modes would be a time based Fourier Transmitted Infrared Spectroscopy study.

When the results for each curing mode/time are considered globally, some remarkable findings can be noted. The most striking observation is the comparison of the 24 hour results. Regardless of curing mode, there was no statistical difference between the light activated and self-cured dual-cure core materials. Furthermore, the one-hour light-activated materials were similar to the 24 hour groups. However, the SBSS of the ten-minute self-cured materials was noted to be the lowest.

Based on the results of this study, the null hypothesis was rejected due to the differences noted between some of the times and curing modes. However, some generalized inferences can be offered. First, some resin composite core materials contain a matrix at ten minutes whose strength is significantly less than that observed at one hour. Second, a number of the light-activated dual-cure materials SBSS at one hour is similar to that observed at 24 hours. Third, the majority of the materials in the self-cure mode demonstrate that SBSS is significantly increased over 24 hours; and fourth, there is no statistical difference in SBSS at 24 hours, regardless of curing mode.

Although direct resin composite core materials were observed to develop SBSS strength by ten minutes, clinicians should be aware that the core material does not contain fully-developed physical properties at that time. A delay in the preparation of the resin core will allow the improvement of physical properties, however this may not be clinically practical. Clinicians should consider this lag in physical property development and to avoid overly stressing the resin composite core material during preparation. Means of reducing preparation stresses, such as diamond bur roughness, pressure applied during preparation should be investigated.

Conclusions

Under the conditions of this study, the direct resin composite dual-cure core materials overall exhibited less shear strength immediately after curing, but was observed to increase with time. The SBSS developed by VLC activation at one hour was similar to that seen at 24 hours. Clinicians should be aware of the immature nature of resin core materials immediately after curing and not overly stress the core during preparation procedures.

Bibliography

1. Summitt JB, Robins JW, Schwartz RS. Fundamentals of operative dentistry, 2nd Ed. 2001.

2. Taubock TT, Bortolotto T, Buchall W, Attin T, Krejci I. Influence of light-curing protocols on polymerization shrinkage and shrinkage force of a dual-cured core build-up resin composite. Eur J Oral Sci 2010;118:423-429.

3. Vandewalle KS, Swift EJ. Estimated Light Exposure Time for Composite Resin Restorative Materials. J Esthet Rest Dent 2011; 23: 201-204.

4. Combe EC, Shaglouf AMS, Watts DC, and Wilson NHF. Mechanical properties of direct core build-up materials. Dent Mater. 1999;15:158-165.

5. ASTM Committee. Standard test method for short-beam strength of polymer matrix composite materials and their laminates. ASTM D2344. 2000.

6. Rodriguez-Gonzales JA, Aviles F. A modified short beam shear specimen for characterization of interfacial strength of nanocomposites. Poly Test 2012;31:792-799.