

**A Comparison of Physical Properties of a Glass Ionomer, Resin-Modified Glass Ionomer,
Conventional Nanocomposite, and Bioactive Restorative Material**

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Submitted in partial fulfillment of the requirements for the degree of Master of Science in the
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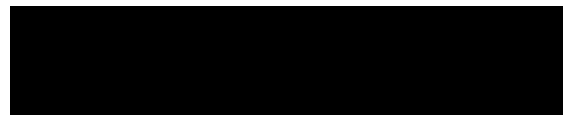
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Abstract

Purpose: The purpose of this *in vitro* study is to test the compressive stress and the diametral tensile strength (DTS) of GC Fuji IX GP® (a glass ionomer), GC Fuji II LC® (a resin-modified glass ionomer), Filtek™ Supreme Ultra (a nanocomposite), and ACTIVA™ BioACTIVE-RESTORATIVE™ (a bioactive restorative material).

Materials and Methods: One hundred eight specimens of commercially available dental restorative materials were assessed for diametral tensile strength (DTS) and compressive stress at 150 lbf. These included 12 samples of five different composites including Filtek™ Supreme Ultra, ACTIVA™ BioACTIVE-RESTORATIVE™ under self-cured (SC) and light-cured (LC) conditions, GC Fuji II LC®, and GC Fuji IX GP®. All testing was completed on an Instron® 5943 with compression platens and a load cell of 1 kN. Exploratory data analyses were conducted on all continuous data (sample height and diameter, DTS, and compressive stress at 150 lbf). Analyses of variance (ANOVAs) were conducted to check for differences in compressive stress and DTS between the different composites. Statistical significance for all statistical tests was declared at $P < 0.05$.

Results: All samples had the same compressive stress at 150 lbf. GC Fuji IX GP® demonstrated the lowest DTS among all tested composites, and ACTIVA™ LC was found to have the highest DTS. Of the four composites with published data, all tested samples significantly underperformed compared to the expected DTS.

Conclusions: This study shows that ACTIVA™, especially under light-cured conditions, is a strong material with additional reported benefits of fluoride and calcium release. In addition, ACTIVA™ is less technique-sensitive than the other materials tested, making it ideal for use in

remote locations. Although further research is needed, ACTIVA™ shows great promise as a restorative material, particularly in austere environments.

Background:

Composite resins are consistently increasing in popularity in the realm of restorative dentistry, both for patients and practitioners.¹ Patients are increasingly focused on esthetics, and as such, amalgams and full coverage metal restorations are falling out of favor with many patients. Many dentists today prefer composite resin to other materials due to factors such as preservation of tooth structure facilitated by conservative preparations, increased retention with the advent of adhesive bonding, limited experience with amalgam in dental schools, mercury toxicity concerns, and marketing benefits of esthetic dentistry.² In fact, in 2006, more than half of posterior direct restorations placed were composites.³ However, composites continue to have some properties which are not so desirable, including volumetric shrinkage, marginal leakage, inadequate bond, and fracture.¹ Bioactive restorative material is one novel material of interest to those who use composite resin.

Composites are defined as something “made up of distinct parts or elements.”⁴ In dentistry, composites are composed of a resin polymer matrix with ceramic fillers. Minimizing the matrix phase or reducing the size of the filler particles can improve properties such as wear resistance.⁵ Increasing the filler content can improve physical properties such as compressive strength and flexural strength.⁶ Traditional composites commonly fail due to bulk fracture in high stress-bearing areas, such as posterior teeth, albeit not to a level that precludes their use in posterior sextants.^{1, 5, 7} The average composite restoration lasts 6-15 years, but new materials may last longer.^{8, 9, 10, 11, 12} Nanocomposites are “composites in which at least one of the phases shows dimensions in the nanometer range.”¹³ The size of these nanoparticles is smaller than the critical crack length of the composite, providing toughness and strength to the material.¹⁴ 3M™

ESPE™ Filtek™ Supreme Ultra (3M™ Oral Care Solutions Division, St. Paul, MN, USA) is one such nanocomposite becoming increasingly popular in restorative dentistry.

Filtek™ Supreme (3M™ Oral Care Solutions Division; St. Paul, MN; USA) was launched in 2002 as a composite restorative material aiming to fill the gap between microfill and hybrid composites.¹⁵ Microfills were desired for their esthetics, but lacked strength and wear resistance. Hybrids, with higher filler content, were stronger but had compromised esthetics due to the large particle size. Since 2002, Filtek™ Supreme has evolved into the popular Filtek™ Supreme Ultra, whose manufacturer reports it has excellent “polish retention, improved fluorescence, and improved translucent shades”.¹⁵ Indications for use of Filtek™ Supreme Ultra include: “direct anterior and posterior restorations, core build-ups, splinting, and indirect restorations.”¹⁵ Filtek™ Supreme Ultra offers a shade selector wheel to simplify layering techniques and shade selection for enhanced esthetics.¹⁵ The manufacturer reports a compressive strength of 370-394 MPa and a DTS of 86-90 MPa, depending on shade.¹⁵ Filtek™ Supreme Ultra retails for \$129.35 for a pack of 20 capsules on Amazon.com.¹⁶

Recently, much research has been done to develop composite materials with improved properties for various uses. Bulk fill composites were introduced to minimize some of the negative aspects of incremental placement of conventional composites. These materials are formulated to facilitate placement in 4 mm increments rather than the traditional 2 mm increments with conventional composites.^{7, 8} In addition to higher depth of cure, bulk fill composites have reduced polymerization stress, less cuspal deflection, good marginal integrity, and suitable bond strength.^{7, 8} However, bulk fill restorations experience greater increase in temperature due to the larger volume of material polymerizing; the heat generated could potentially have a negative effect on the pulpal tissue in a vital tooth.¹⁷

Conventional glass ionomers have been used in dentistry since 1971. They are made of fluoroaluminosilicate glass and polyacrylic acid. Glass ionomers have many benefits which make them attractive to providers and patients. They release and recharge fluoride and chemically bond to tooth structure via a calcium chelation bond, providing restorative and biologic benefits. Glass ionomers are typically simple and quick to place, which is beneficial for pediatric dentistry in particular. Excellent marginal adaptation of glass ionomers may be due to the coefficient of thermal expansion, which is similar to that of teeth. Despite the many positive properties of glass ionomers, they are lacking in the realm of esthetics. Glass ionomers tend to appear rather opaque with poor surface finish. In addition, the low flexural strength and high modulus of elasticity make glass ionomers brittle, prone to fracture, and easy to wear. Glass ionomers are therefore not the ideal restorative material for permanent restorations in high stress areas, but are a good option for smooth surface restorations such as Class Vs.¹⁸ GC Fuji IX GP® (GC America, Inc.; Alsip, IL; USA) is one variety of glass ionomer with many indications: pediatric or geriatric restorations, core build-ups, interim restorations, or long-term temporary restorations.¹⁹ Glass ionomers require the use of a cavity conditioner, typically polyacrylic acid, to prepare the tooth surface prior to placing the restoration. In addition, due to high water solubility, glass ionomers require a light-cured coat placed on the surface prior to finishing. Resin-modified glass ionomers were developed to improve the properties of glass ionomers and are considered a hybrid between glass ionomers and composite resin.²⁰ GC Fuji IX GP® retails for \$124.90 for a pack of 50 capsules on Amazon.com.²¹

Bioactive restorative materials have been introduced to the dental materials industry more recently. This study will focus on ACTIVA™ BioACTIVE-RESTORATIVE™ (PulpDent; Watertown, MA; USA). ACTIVA™ BioACTIVE-RESTORATIVE™ is a bioactive composite

restorative material with an ionic resin matrix, rubberized resin, and glass ionomer component.²² PulpDent advertises ACTIVA™ BioACTIVE-RESTORATIVE™ as “a highly esthetic, bioactive composite that delivers all the advantages of glass ionomers in a strong, resilient resin matrix that will not chip or crumble.”²² It is recommended for the following uses: pits; root surface cavities; and Class I, II, III, and V direct restorations without pulpal involvement.²³ ACTIVA™ is composed of diurethane dimethacrylate and bis (2-(methacryloyloxy) ethyl) phosphate with barium glass, ionomer glass, and a polyacrylic acid/maleic acid copolymer.²⁴ The manufacturer reports flexural strength of 102 MPa. ACTIVA™ BioACTIVE-RESTORATIVE™ retails for \$119.49 per 5 mL/ 8 gram syringe and is applied with an automix syringe.²²

Bioactive restorative materials have potential for benefit in Army dentistry. The release of fluoride, calcium, and phosphate can strengthen teeth even after placement of the restoration, and the fluoride can be recharged with fluoride in toothpaste, much like with glass ionomers. Bioactive restorative materials such as ACTIVA™ do not require a separate bonding agent, so retention could be improved versus conventional composites when restoring preparations with sclerotic dentin. The shape and flexibility of the ACTIVA™ metal cannula tip facilitates access to the preparation and increases ease of restoration in hard-to-reach areas, such as the distal aspect of a second molar. A well-placed direct restoration composed of a durable material may delay or eliminate the need for indirect restorations, which are more costly to the patient and to the dental practice, and typically result in greater removal of tooth structure. Fixed dental prostheses fail most commonly due to recurrent caries; if bioactive restorative material proves to be durable enough for large restorations, its bioactive benefits may increase the longevity of overlying full coverage restorations. These potential benefits are of particular importance in the

Army, where fewer placement steps can increase production, resulting in more Soldiers able to be treated each day. In addition, longer-lasting, durable restorations result in increased readiness of the force, with fewer hours spent in the dental office, away from training, repairing or replacing defective restorations. In addition, a material that doesn't require specialized equipment such as a curing light, bonding agent, or cavity conditioner can be beneficial for austere environments such as deployments or dental mission trips.

US Army dentistry cares for over 485,000 active duty Soldiers, retirees, family members, National Guard, and Reserve Soldiers, as well as civilians with humanitarian missions.² Studies such as this are important to the military, because literature and materials studies are of utmost importance to Army dental practice and can guide dentists in the selection of materials for clinical practice. If a new material is developed and shown to be of benefit to the military community, it may be advantageous for the Army to revise policies on dental practice to incorporate these materials in practice. At the same time, if a material is found to be deficient in several properties, it may be safer and more cost-effective for Army dentists to use other proven materials. This study will compare the physical and mechanical properties of three dental materials: GC Fuji IX GP®, a glass ionomer; GC Fuji II LC®, a resin-modified glass ionomer; and ACTIVA™ BioACTIVE RESTORATIVE™, a bioactive composite.

Hypotheses:

A. Compressive stress at 150 lbf

- a. ACTIVA™ BioACTIVE RESTORATIVE™ has a lower compressive stress at 150 lbf than the glass ionomer and resin-modified glass ionomer.
- b. GC Fuji II GP® has a lower compressive stress than the conventional glass ionomer.

B. Diametral tensile strength

- a. Filtek™ Supreme Ultra has a higher diametral tensile strength than the bioactive restorative material, glass ionomer, and resin-modified glass ionomer.
- b. ACTIVA™ BioACTIVE RESTORATIVE™ has a higher diametral tensile strength than the glass ionomer and resin-modified glass ionomer.
- c. GC Fuji II LC® has a diametral tensile strength higher than the conventional glass ionomer.

Materials and Methods:

One hundred eight specimens of commercially available dental restorative materials were assessed for diametral tensile strength (DTS) and maximum compressive stress. These included 12 samples of five different composites including Filtek™ Supreme Ultra, ACTIVA™ Self Cure, ACTIVA™ Light Cure, GC Fuji II LC®, and GC Fuji IX GP®. Filtek™ Supreme Ultra was used in compule form. ACTIVA™ was dispensed with mixing tips with metal cannula from auto-mix syringe. GC Fuji II LC® and GC Fuji IX GP® capsules were triturated in a Kerr OptiMix Programmable Computerized Mixing System for 10 seconds at 4000 cycles per minute.

Compressive Stress at 150 lbf

Compressive stress testing was conducted following the protocol from Keating.²⁵ Specimens were prepared per ISO 9917-1 and 9917-2. A 6 mm x 4 mm cylindrical stainless steel mould was used to form the specimens. The mould was sprayed with model release agent prior to forming specimens. After insertion of the material into the mould, the mould was covered with a polyester film and glass slide on both sides, and firm digital pressure was applied to extrude the excess material.

Diametral Tensile Strength

Diametral tensile strength testing was conducted following ADA Specification No. 27.²⁶ A mould was created by making an EXAFLEX® Vinyl Polysiloxane (GC America, Inc.; Alsip, IL) putty impression of a BIC® (BIC, France) mechanical pencil eraser, which had dimensions approximately the size of the recommended mould (3 mm height and 6 mm diameter). After insertion of the material into the mould, the mould was covered with a polyester film and glass slide, and firm digital pressure was applied to extrude the excess material.

Specimens were light or chemical cured following manufacturer's instructions. Self-cured ACTIVA™ specimens were cured in a distilled water bath at 37°C, as the material does not cure at room temperature. GC Fuji IX GP® specimens were allowed to cure at room temperature for 6 minutes. Light-cured specimens were cured using the 3M ESPE Elipar™ DeepCure-S (3M™ Oral Care Solutions Division, St. Paul, MN, USA) curing light, with the tip directly on the glass slide. Excess material was removed by abrasion with 320 grit abrasive paper. The specimens were stored in distilled water at 37°C in a Whip Mix Digital Water Bath (Whip Mix; Louisville, KY; USA) until time of testing, approximately one hour. Prior to testing, the specimen dimensions were measured using a digital micrometer to 0.01 mm accuracy. Compressive strength testing was conducted using the Instron® 5943 (Instron®; Norwood, MA; USA) with compressive strength platens at a crosshead speed of 1.0 mm/min to 150 lbf.²⁷ Filtek™ samples were not tested for maximum compressive strength due to limitations of the testing apparatus. Diametral tensile strength testing was conducted using the Instron® 5943 with compressive strength platens at a crosshead speed of 1.0 mm/min. For DTS, each specimen was placed on its side between the platens with a small piece of filter paper wet with distilled water on each platen. Specimens which did not fracture were estimated to achieve a maximum load of

1000 N, which was the limit of the Instron machine used. Testing parameters were set using BlueHill® 3 (Instron®; Norwood, MA; USA) software.

Exploratory data analyses were conducted on the all continuous data (sample height and diameter, DTS, and compressive stress at 150 lbf). The Shapiro-Wilk test was used to assess the normality of the data distributions. For continuous data, measures of central tendency are presented as means with associated standard deviations. Analyses of variance (ANOVAs) were conducted to check for differences in compressive stress and DTS between the different composites. Compressive strength was not calculated because the specimens were not compressed to fracture due to limitations with the testing apparatus and software. The DTS was calculated using the formula:

$$DTS = \frac{2F}{\pi dh}$$

where F is the maximum load at fracture in N, d is the diameter of the specimen, and h is the height of the specimen. Eta squared (η^2) statistics are presented as measures of effect size for significant ANOVA results and Cohen's *d* is reported for significant t-test results.²⁸ Statistical significance for all statistical tests was declared at $P < 0.05$. Data were analyzed using SPSS 25.0 (IBM; Armonk, NY; USA).

Results:

Compressive and DTS characteristics are summarized in Table 1. No difference was found among the four composites tested for compressive stress at maximum load, $P=0.96$. The overall mean for compressive strength was 50.95 MPa (SD = 1.45). In contrast, several homogeneous subsets were found among when assessing the composites for DTS, $P<0.001$. The effect size for this difference was $\eta^2 = 0.86$, indicating a large difference in DTS between the groups.

GC Fuji IX GP® demonstrated the lowest DTS among all tested composites with a mean of 6.52 (SD = 2.09), $P < 0.05$. On the other end of the spectrum, ACTIVA™ LC was found to have the highest DTS with a mean of 31.62 (SD = 2.51), $P < 0.05$. No difference in DTS was noted between GC Fuji II LC® (M = 23.16, SD = 4.15) and ACTIVA™ SC (M = 26.74, SD = 5.09), nor was there a difference between ACTIVA™ SC and Filtek™ (M = 28.61, SD = 4.10), both $P > 0.05$ (see Figure 1).

Finally, tested DTS values were compared to expected values published by the manufactures. These comparisons are summarized in Table 2. Of the four composites with published data, all tested samples significantly underperformed compared to the expected DTS, all $P < 0.001$. Moreover Cohen's *d* effect sizes of -3.39 to -7.41 indicated large differences compared to expected values.

Discussion:

In order to understand the importance of tests such as those conducted in this study and their implications to clinical dentistry, one must first have at least a rudimentary understanding of the mechanical properties of materials. This study assessed the compressive stress and diametral tensile strength of several restorative materials. Stress is the force per unit area within an object when an external force is applied. It causes deformation within the object.²⁹ Strength is the stress necessary to cause fracture or a specified amount of plastic deformation in an object. Strength is “the average level of stress at which a material exhibits a certain amount of initial plastic deformation or at which fracture occurs in test specimens of the same shape and size.”²⁹ Compressive stress, which indicates the ability of a material to withstand forces applied in a vertical direction, is the internal resistance to a load that compresses or shortens an object.^{6, 29} Tensile stress is the tensile force per unit area perpendicular to the force direction. It is caused by

a load that tends to stretch or elongate a body. These forces are important in relation to clinical dentistry because masticatory forces are known to fracture restorations and teeth.⁶ Mastication produces jaw movements in a variety of directions which place forces on teeth in virtually all directions. Restorative materials which exhibit high strength may have increased longevity and prolong tooth survival, saving patients money and improving their dental health over the long term. In addition, strong, durable, long-lasting restorations are important in populations with poor access to care, including impoverished people and Soldiers deployed to remote locations.

This study measured the compressive stress on restorative materials under 150 lbf. This value was chosen because it utilized the testing parameters from a previous study conducted with the same equipment, and because the Instron® machine available was not able to supply enough force to fracture all of the samples. At 150 lbf, all of the materials tested experienced the same compressive stress. This value, which is equivalent to 667 N, exceeds the reported average bite force of 250-280 N.³⁰ Thus, at a force which exceeds the average bite force of an adult human, all of the materials tested experienced approximately 50 MPa of compressive stress. It was not enough to break any of the specimens. Thus, one may conclude that all of the materials tested are strong enough to withstand the compressive forces of an average human's bite. Natural teeth have been reported to have a fracture strength of 305 MPa, which has been suggested as a standard for optimal strength of composite resins in posterior teeth.⁶ More research should be done repeating the compressive strength tests to fracture with a machine that can accommodate heavier forces.

Tensile strength was measured using the diametral compression test. This test is frequently used as a proxy for tensile strength due to difficulty testing brittle materials. Some brittle materials can be strong but they may fracture with little warning because little or no

plastic deformation occurs to indicate high levels of stress.²⁹ This was true for Filtek™ Supreme Ultra. Some Filtek™ Supreme Ultra specimens which barely touched the platens during positioning of the machine fractured prior to initiation of the test. Those specimens were excluded from the analysis. On the other hand, ACTIVA™ visibly deformed during application of pressure by the top platen prior to fracture or end of test. This shows that ACTIVA™ deforms prior to fracture, which may contribute to successful results in the oral cavity, including potential for good wear resistance. Future research should evaluate the resiliency of ACTIVA™ and whether the visible deformation under compression is elastic or plastic until the point of fracture. Most dental materials are brittle and are highly susceptible to crack initiation in the presence of surface flaws when subjected to tensile stress, such as with flexural loading.²⁹ If ACTIVA™ is less brittle than Filtek™ Supreme Ultra, it may be more resistant to crack propagation under tensile stresses, leading to increased clinical success.

The values in this study varied widely from the manufacturers' reported values. The testing protocol from this study may vary from those the manufacturers use when reporting their values. However, within the limitations of this study, light-cured ACTIVA™ maintained the highest DTS values. Eight specimens of light-cured ACTIVA™, one specimen of self-cured ACTIVA™, and five specimens of Filtek™ Supreme Ultra did not fracture during the DTS testing. Due to the limitations of the machine, the maximum load was estimated at 1000 N, which was the capacity of the machine. However, actual values are likely much higher than this. Repeat testing on a higher capacity machine may return results different from that of this study, with regards to the light-cured ACTIVA™ and the Filtek™ Supreme Ultra. There may be more of a difference between the samples if allowed to completely undergo force until fracture.

In this study, the samples used were not completely uniform in dimension although they were formed from the same mould. This may contribute to the deviation in values. Further research should be conducted using a manufactured mould in place of the PVS mould, as manufactured moulds can create samples of more uniform dimension, eliminating sources of error with the sizes of the specimens. In addition, any deviation from a perfectly smooth surface can decrease the strength values. This was evident when, during practice runs, misshapen specimens fractured very easily under compression. However, restorations placed in the mouth are rarely perfectly flat, smooth surfaces. Surface variations such as occlusal anatomy may result in strength values in the mouth less than those generated in a controlled lab environment. More research should be done with a higher capacity machine to evaluate the compressive strength of these materials. That is, research should be conducted to determine the maximum load at fracture of the materials, and to evaluate whether one is superior to the others and compare with manufacturers' reported values, and implications with masticatory forces. ACTIVA™ should be evaluated for resiliency and wear resistance. Testing with different angulations and anatomic forms of the top surface should be tested.

Suggestions for further research in addition to those stated above include:

- assessment of dry versus wet testing: measure compressive strength and DTS with varying times stored in a wet environment
- dimensional change of wet samples: measure sizes of samples immediately after fabrication and again after varying times stored in a wet environment
- continuation of self-curing: measure compressive strength and DTS at varying times following preparation of self-cure ACTIVA™.

Conclusion:

Bioactive restorative material holds a plethora of clinically relevant concepts worth exploring. This study shows that ACTIVA™ especially under light-cured conditions, is a strong material with additional benefits of fluoride and calcium release. In addition, with a retentive preparation design, it does not require the use of a separate bonding agent or etchant, and it does not require a protective coating. These properties make the material less technique-sensitive and ideal for use in an austere environment. In addition, the capability of using a self-cure component is beneficial in situations where electricity may not be available, because under self-cured conditions it is still much stronger than the conventional glass ionomer. Although further research is needed, ACTIVA™ shows promise as a restorative material for use in austere environments, such as on military deployments or dental mission trips.

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Appendix

Table 1. Material Characteristics, M(SD)

Material	Fuji II LC	Fuji IX GP	Activa SC	Activa LC	Filtek	P ¹
Compressive Stress at 150 lbf (MPa)	51.02 (1.42)	50.9 (1.87)	50.78 (1.12)	51.09 (1.47)	N/A	0.96
Diametral Tensile Strength (MPa)	23.16 (4.15)	6.52 (2.09)	26.74 (5.09)	31.62 (2.51)	28.61 (4.10)	<0.001

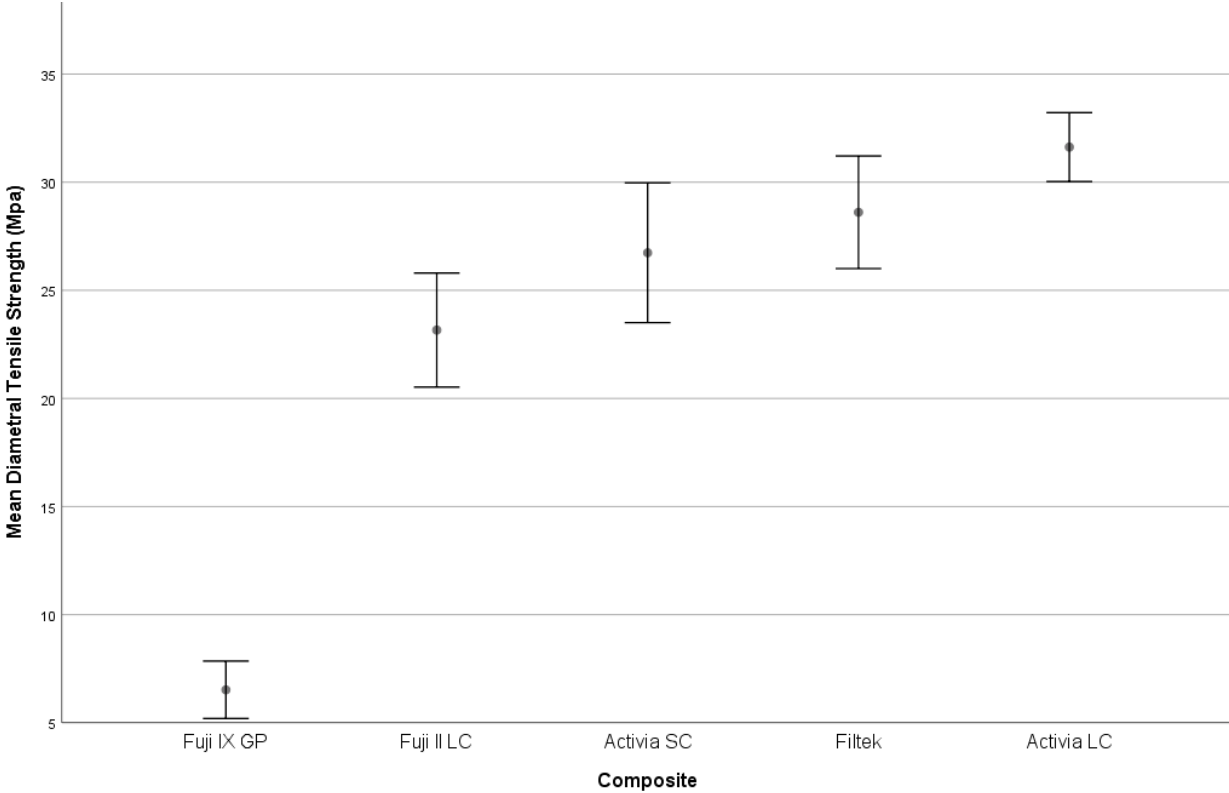
1. Significance based on ANOVA.

Table 2. Comparison of tested and advertised diametral tensile strength

Material	Diametral Tensile Strength (MPa)	Manufacturer Value	P^1	d
Fuji II LC	23.16 (4.15)	22	<0.001	-7.41
Fuji IX GP	6.52 (2.09)	N/A	-	-
Activa SC	26.74 (5.09)	44	<0.001	-3.39
Activa LC	31.62 (2.51)	44	<0.001	-4.94
Filtek	28.61 (4.10)	86	<0.001	-3.75

1. Significance based on one-sample t-test comparison of published manufacturer data

Figure 1. Mean Diametral Tensile Strength by Composite Type



*Error bars represent 95% Confidence Intervals



Image 1. Laboratory Set-Up



Image 2. Compressive Stress Specimen



Image 3. Compressive Stress Test

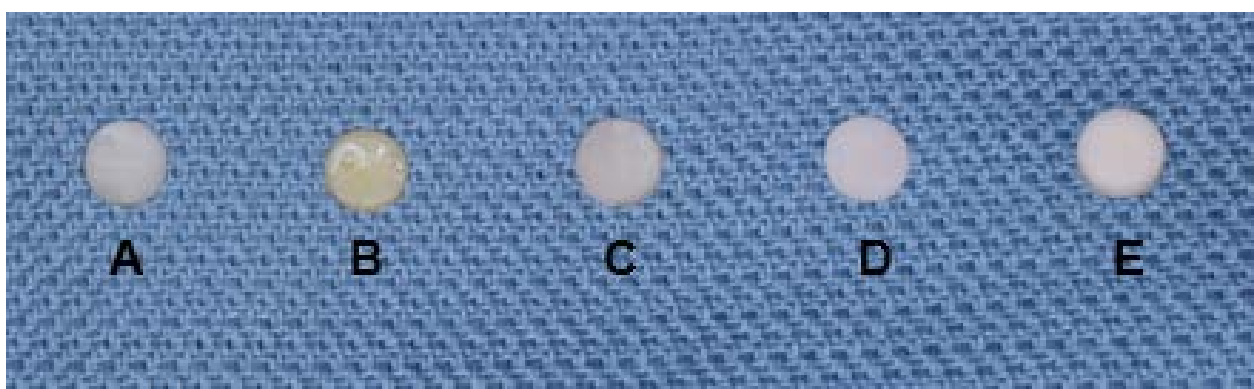


Image 4. Tensile Strength Specimens
A: Activa Light-Cured
B: Activa Self-Cured
C: Filtek Supreme Ultra
D: Fuji IX GP
E: Fuji II LC

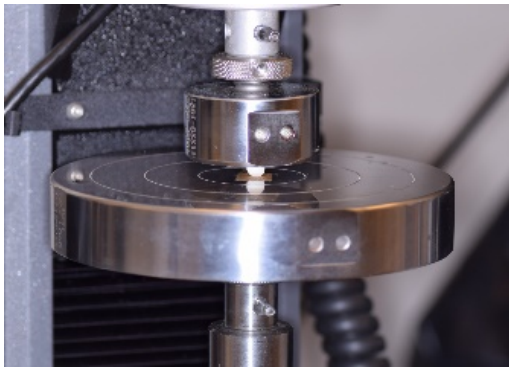


Image 5. Tensile Strength Test



Image 6. Specimens after testing