MECHANICAL PROPERTIES OF DENTAL IMPRESSION MATERIALS

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ABSTRACT

Impression materials make a record of tooth structure and provide a critical step in many dental applications. The mechanical properties of the impression material determine its clinical viability. **Purpose:** This thesis examines three novel methods for measuring mechanical properties of elastomeric impression materials including: (1) flow, (2) tear strength, and (3) elastic recovery. **Methods:** Flow of 8 impression materials (6 silicone, 1 polyether, 1 hybrid) was measured using a shark fin testing device. The shark fin height of each material (n = 5) was collected at 30 second intervals beginning 30 seconds after mix and ending when material ceased to flow. Shark fin heights at each interval were compared among material groups. Tear strength specimens of 6 materials (4 silicone, 1 polyether, 1 hybrid material) were prepared using a “thin film” tear strength mold. Specimens were divided into four groups (n=5). Group 1 and 2 were immediately loaded in tension until failure. Groups 3 and 4 were tested 24 hours after fabrication. Groups 1 and 3 were tested at 1mm/min and groups 2 and 4 were tested at 500mm/min tearing rate. Elastic recovery specimens of 6 materials (5 silicone, 1 hybrid) were prepared using an ASTM D412 (dumbbell) mold modified by the addition of notches delineating a 20mm span. The specimens (n=5) were stretched by 50%, 100%, and 150% of their original length at 300mm/min. After one hour, the distance between the two notches was measured and used to calculate the percent elongation. The elastic recovery of the same materials were tested using the ISO Specification 4823.9.7. Statistical
analysis of all testing methods was performed with an ANOVA and Tukey’s Test ($\alpha = 0.05$).  **Results** Polyether materials demonstrated significantly greater flow than hybrid and silicone materials. Silicone materials provided significantly higher tear strength values than polyethers and hybrid materials. Faster tearing rates and longer setting time improved tear strength. The hybrid material provided the second lowest amount of elastic recovery. **Conclusion** The novel testing methods presented in this manuscript can be used to differentiate materials based on their mechanical properties. Properties vary among composition groups and should be considered when selecting a material.
ACKNOWLEDGMENTS

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INTRODUCTION

Rationale

Impression materials are used to record an impression of a tooth preparation in dentistry (fig 1). From the impression, the internal geometry of the prosthetic crown is fabricated. The fidelity of the impression determines the fit of the crown and the success of the prosthesis. Therefore, impression materials are a key factor in clinical procedures and must provide adequate mechanical properties.¹

![Figure 1. Impression material in a tray](image)

The ADA Standards Committee on Dental Products has developed specifications for dental materials. Specification 19 is devoted to dental elastomeric impression materials and Specification 20 is devoted to dental duplicating material. Sections of Specification 19 and 20 include protocols for testing specific properties of impression materials including flow (4.3.12), resistance to tearing (4.3.10), and permanent...
deformation caused by fixed strain (4.3.9). The International Standards Organization (ISO) has also developed a standard method for testing impression materials. ISO Standard 4823 details the tests of dental elastomeric impression materials including a consistency test (9.2) and elastic recovery test (9.7).

For material property testing to be relevant, however, the testing must correlate to clinical situations. Tests that more closely simulate clinical applications have been developed in industry and academics. A novel method for determining flow and elastic recovery was created by 3M ESPE laboratories. A novel tear strength mold was developed by Dr. Alan Boghosian at Northwestern University. Although preliminary data has been established from these new testing methods, thorough objective tests need to be performed to qualify these methods.

Background and Significance

Material Composition

Impression materials can be divided into two major classes: irreversible hydrocolloid materials and elastomeric materials. Hydrocolloid materials (i.e. alginates) have lower tear strength and less dimensional stability than elastomeric impression materials. Elastomeric impression materials, which were introduced in the 1950’s, have improved upon the shortcomings of the hydrocolloid materials. These materials are defined as those “capable of reacting to form a rubber-like material which can be used for taking impressions.” The compositions of elastomeric impression materials include silicone, polyether, and hybrid polyether/silicone.
Silicone impression materials. Silicone materials can further be divided into condensation and addition silicone materials, designated by their mode of polymerization. During polymerization of condensation silicone materials, ethyl alcohol is formed as a by-product.\(^6\) Evaporation of the ethyl alcohol leads to polymerization shrinkage\(^9\) and poor dimensional stability.\(^10\) For this reason, condensation silicone materials have lost clinical popularity and will not be considered in this manuscript.

Addition silicone materials are manufactured in two cartridges, one containing the base and one the catalyst. The base contains siloxane monomers, silica fillers and colorant. The catalyst contains a platinum salt activator, siloxane monomers, silica fillers, retarder and colorant. When the contents from the two cartridges mix, the impression material forms cross links by means of an addition polymerization reaction.\(^11\)

In the polymerized material, there are hydrophobic hydrocarbons surrounding the siloxane bonds. These hydrocarbons make silicone impression materials inherently hydrophobic. To improve wetability, surfactants are grafted onto the monomers to make them more hydrophilic.\(^12\)-\(^14\)

Silicone impression materials can be produced in different consistencies (ie. light-body, heavy-body) depending on the amount of silica filler added. A study by Hosotani\(^15\) revealed that the amount of filler in the material will alter its mechanical properties. He produced addition silicone materials with varying amounts of filler (0 - 50 wt%) and tested viscosity and tensile strength. His results showed that decreased filler content will decrease the viscosity of the material and improve flow; while increased filler content will make the material rigid and improve its strength. He concluded that a “reasonable” amount of filler should be added to make a clinically successful material. Chen et al\(^16\)
produced two experimental silicone materials with different percent filler content (5 and 17.5 wt%) and determined that increased filler content improved accuracy and dimensional stability.

Silicone materials can also be produced with different setting times (ie. fast-set, regular-set) depending on the amount of retarder that is added. Retarder slows the polymerization of the material and extends the working time for the clinician. However a study by Tam showed that the addition of a retarder decreased the tear strength of silicone impression materials. 17

**Polyether impression materials.** Polyether materials are also supplied as a base and catalyst. The base material contains a polyether monomer, silica filler, and a plasticizer. The catalyst contains an aromatic sulfonate ester initiator and colorants. Polymerization occurs as the initiator opens the imine ring of the polyether monomer allowing it to attach to other monomers. 12 The polymerized material contains carbonyl and ethyl groups capable of hydrogen bonding, making this material hydrophilic. 18,19 The polyether polymer is less bulky and cross-linked than the polysiloxane polymer. The reduction in bulk of the polymer is credited for improving the flow of polyether materials. 20

**Hybrid impression materials.** Hybrids are a new class of material introduced to combine the properties of silicone and polyether materials. Currently, the only commercially available Hybrid material is Senn (GC, Aichi, Tokyo Japan). According to their MSDS, this material is composed of dimethylpolysiloxane (silicone monomer), silicone dioxide (silicone monomer) and a proprietary polyether compound that forms a polymer containing both siloxane and polyether groups. 21 These materials are inherently hydrophilic and do not contain a surfactant.
A limited number of studies have reviewed the physical properties of the Hybrid material including detail reproduction\textsuperscript{23,24}, wettability\textsuperscript{24}, dimensional accuracy\textsuperscript{25}, toughness\textsuperscript{26}, and flow\textsuperscript{24,26}. Conflicting evidence has been reported by these studies and no generalizations regarding this material have been broadly accepted.

**Mechanical Properties**

The clinical viability of impression materials depends on several mechanical properties, including flow, tear strength, elastic deformation, hydrophilicity, detail reproduction, and dimensional stability.\textsuperscript{27} The importance of these properties and the standards used to measure these properties (taken from ADA Specification 19 and 20 and ISO Standard 4823) will be individually addressed.

**Flow.** Flow is dependent on the ability of the material to resist shear forces. An impression material must be able to penetrate the narrow subgingival sulcus and tight interproximal areas. Therefore, it must be able to resist the shear forces as it is pushed between tooth and gingival walls.\textsuperscript{28} Viscosity, which is resistance to flow, has traditionally been measured on a rheometer.\textsuperscript{29,30}

The ADA Specification for flow (4.3.12) measures the flow of an impression material after it has already undergone polymerization. A cylindrical specimen of impression materials is prepared in a split metal mold (fig 2). 1 hour following polymerization, a load is applied and the change in height of the specimen is recorded. Flow is defined as the change in height divided by the original height of the specimen.\textsuperscript{2} The ISO outlines a test for consistency (9.2), which is defined as the “degree of firmness with which particles of a material cohere so as to allow the material to flow, or resist
flow”. This test requires that a determined amount of material is placed between two glass slides. 25 seconds after dispensing the material, a 14.7N load is applied to the top glass slide. The material will form an approximate outline of a circle (fig 3). The average value between the major and minor diameter of the resulting specimen is reported as an indication of consistency.\(^3\)

![Figure 2. Specimen and mold for ADA flow and ISO elastic deformation test](image)

![Figure 3. Specimen used for ISO consistency test](image)

*Tea strength.* Tear strength is the ability of the material to resist tearing under a tensile stress. Impression materials are subjected to tensile stresses when they are
removed from the oral cavity and from stone models.\textsuperscript{31} The ADA does not specify a method for determining the tear strength of elastomeric impression materials in Specification 19, however, it defines a tear strength testing method (4.3.10) in Specification 20 for dental duplicating materials. This test methods specifies the use of an ASTM standard D624-54 “notched” mold for fabrication of specimens (fig 4). These specimens are loaded in tension at 254 cm/min and the load at failure is recorded as the tear strength.\textsuperscript{2} There is no ISO specification for tear strength.\textsuperscript{3}

Dr. Alan Boghosion presented a new method for testing the tear strength of impression materials at the International Association of Dental Research meeting in 2005. His method uses a bar shaped specimen with a notch in the center (resulting in a 0.2mm thick film). The specimen is tested to failure in tension.\textsuperscript{4}

![Figure 4. ASTM D624-54 mold shape](image)

*Elastic deformation.* Elastic deformation is the change in dimensional shape of the material following an applied stress. The tensile stresses applied to impression materials when they are removed from the oral cavity make them susceptible to deforming from their original dimensions.\textsuperscript{32,33}

The ADA standard for measuring deformation is termed the compression set test (4.3.5). In this test, a cylindrical specimen of impression material is prepared in a split metal mold (fig 2). Three minutes after the specimen is polymerized in a water bath, the
height of the specimen is measured (h₁). Immediately, a twelve percent compressive strain is applied for 30 seconds. Thirty seconds following, the height of the specimen is re-measured (h₂). The percent compression set is given by the formula: \( \frac{h_i - h_2}{h_i} \times 100 \) (where \( h_i \) is the initial height of the specimen). ²

The ISO standard details an elastic recovery test (9.7). A cylindrical specimen is prepared and polymerized in a water bath. 1 minute after polymerization, the height of the specimen is recorded (h₁). Using a specified device (fig4), thirty percent strain is applied for 1 second (and slowly released for 5 seconds). The height of the specimen is measured 2 minutes following the compression (h₂). The percentage of elastic recovery can be determined from the formula: \( 100 - \left( \frac{h_i - h_2}{h_i} \times 100 \right) \) (where \( h_i \) is the initial height of the specimen). ³

Figure 5. ISO elastic recovery device
Hydrophilicity. Hydrophilicity is the material’s attraction to water-based substances. During normal impression making, saliva and blood may be present in the areas being impressed. Impression materials should be able to wet the surface of teeth and gingiva that contains water. However there is no ADA or ISO specification for measuring hydrophilicity. A traditional method for measuring hydrophilicity involved preparing a flat specimen of impression material. Immediately after polymerization of the material, the contact angle of a water droplet was measured on the surface of the specimen (fig 6). This test has been refined by Kugel who examined the hydrophilicity of an unset material. A 20um layer of impression material was spread on a glass plate (controlled by a marking template). The contact angle was measured before the material was completely polymerized (as early as 45 seconds after start of mix of the material). Contact angle of the material was measured using a video-based drop shape analysis system that took measurements 25 times per second.

Figure 6. Contact angle measurement

Detail reproduction. Detail reproduction is the ability of an impression material to capture fine surface features. This property is important for reproducing important
anatomical features of the tooth structure of interest\textsuperscript{12} The ADA specification 3.3.5 for detail reproduction requires materials to have acceptable compatibility with investment, referring to specification 4.3.7. In this test, impression material is pressed against a stainless steel testing block for 30 minutes. The block is inscribed with lines of varying widths (from 0.02 – 0.30 mm). Two minutes after separating the impression material and block, an investment slurry is poured over the impression. After the poured impression has set in an incubator for 30 minutes, the investment cast is removed and inspected. The material is evaluated by the appearance of a continuous line (the width designated by consistency of material) visible without magnification.\textsuperscript{2}

![Figure 7. ANSI/ADA Specification No.19 stainless steel test block](image)

The ISO method for measuring detail reproduction (9.4) is similar, and only the differences between the methods will be described. The test block is heated to 35\textdegree C before use. The impression material is pressed against the test block for the manufacturer’s recommended set time in a 35\textdegree C water bath. Immediately following, the impression material specimen is directly examined under a microscope for the presence of a designated line.\textsuperscript{3}
Tests that attempt to more closely simulate clinical situations have been reported in the literature. For example, Takahashi et al\textsuperscript{37} and Boening et al\textsuperscript{38} have prepared impressions of wet human dentin. The impressions were examined for reproduction of gingival sulcus details. Shah examined detail reproduction of lines inscribed in bovine dentin.\textsuperscript{22} Both studies use wet dentin at oral temperature as a substrate, which more closely simulates actual setting than the ADA standard.

*Dimensional stability.* Dimensional stability is the ability of the material to retain its original shape following oral removal. This property is important when the impression is reused to make stone casts after oral removal.\textsuperscript{38} ADA specification 4.3.9 describes the test for dimensional change. Impression material is poured into an open mold and a plate inscribed with two parallel lines (5mm apart) is pressed on top of the mold. The mold and plate are placed in a water bath for the manufacturers set time. The mold is removed from the bath and the plate is separated from the mold. 24 hours later, the distance between the crosslines reproduced on the impression material ($l_1$) is measured using a microscope with a micrometer stage. Percent dimension change is recorded as:

$$\frac{5\text{mm} - l_1}{5\text{mm}} \times 100$$ \textsuperscript{2} ISO specification 9.5, the linear dimensional change test, details an identical procedure.\textsuperscript{3}

A recent study by Martin examined an updated approach for measuring dimensional change with the ADA specifications. Direct measurements of the recorded lines on the impression material specimens were made with an automatic laser radial micrometer that provided a 0.0001mm resolution. This method improves the accuracy of the microscope measurement technique by reducing operator variability and device limitations.\textsuperscript{39}
Another method for determining dimensional change has been used by Kanehira et al\textsuperscript{40}, Williams et al\textsuperscript{41}, and Piwowarczyk et al\textsuperscript{42}. This method is performed by making an impression of a master model, pouring stone casts of that impression at proceeding time intervals, and determining the difference in dimensions of the stone casts from the master model.

\textit{Advantages and disadvantages of material types.} Many studies have attempted to find the advantages and disadvantages of silicone and polyether impression materials. A study by Chai et al\textsuperscript{43} revealed that polyether materials have higher tear strengths than silicone materials.\textsuperscript{44} A study by Hondrum\textsuperscript{44} and recent studies by Lu et al\textsuperscript{45} and Boghosian et al\textsuperscript{4} reveal the opposite conclusion, that silicones provide greater tear strength. Clinical evaluations suggest that silicone materials have greater tear strength than polyethers.\textsuperscript{46} Kugel revealed that water produces a smaller contact angle on polyethers than silicones, proving polyethers are more hydrophilic. Furthermore, unset silicone materials were considerably more hydrophobic upon contact with water due to the time it takes for surfactants to migrate to the surface of the material.\textsuperscript{35} A recent study by Michalakis compared hydrophilicity pre- and post-set and confirmed that polyethers are more hydrophilic than silicone materials.\textsuperscript{47} Their superior hydrophilicity has demonstrated polyether’s clinical advantages.\textsuperscript{48} A study by Kanehira et al\textsuperscript{7} concluded that the representative silicone material was dimensionally stable regardless of storage time or ambient humidity while the stability of polyether materials was dependent on time and humidity.

Hybrid polyether/silicone materials were introduced as a material that combines the advantages of polyethers and polysiloxanes, however, only a few studies have examined
their mechanical properties. An IADR abstract by Klettke compared the flow, hydrophilicity, and [tear] toughness of polyether and silicone materials and a hybrid material. His results showed that the hybrid ranked in the statistically lowest group for all properties. McCabe et al demonstrated a polyether and hybrid material more accurately recoded details of a moist surface (owing to their hydrophilicity) than a silicone material. However Kanehira et al concluded that the hybrid material had equal detail reproduction and wettability as a polyether and silicone material.

In the projects outlined in this thesis, silicone, polyether, and a hybrid material will be used to evaluate new test methods. The results of these projects will be beneficial to the field of dental materials for two reasons. First, new clinically-focused test methods will be evaluated. Second, the amount of data regarding hybrid polyether/silicone materials will be significantly expanded.

Hypotheses and Aims

This study will examine commercially available dental impression materials using three novel testing methods: (1) shark-fin flow, (2) thin film tear strength, and (3) elastic recovery from tensile strain. Controlled relevant variables will be introduced into the study design including the composition, setting time, and tearing rate of the material. The ability of the novel test methods to distinguish materials in different experimental groups will give an indication of the value of these tests. Also, when applicable, the results of the novel testing methods will be compared to the results of ADA specified tests.
Aim 1

To evaluate the “shark fin” flow testing device.

Hypothesis 1: (null)

There will be no difference in flow between silicone, polyether, and hybrid silicone/polyether impression materials.

Specific Aim 1. Four silicone, one polyether, and one hybrid polyether/silicone materials will be evaluated. Flow, as determined by the “shark fin” device, will be measured at 30 second intervals following mix of these materials. Flow will be qualitatively evaluated by a graphical representation of flow values vs. time. Flow will be quantitatively evaluated by a one-way ANOVA of flow values at individual time points.

Aim 2

To evaluate the thin film tear strength mold.

Hypothesis 2: (null)

The test will find no difference in tear strength between silicone, polyether, and hybrid silicone/polyether impression materials.

Specific Aim 2. Four silicone, one polyether, and one hybrid polyether/silicone materials will be evaluated using a novel specimen design. A three-way ANOVA will be used to find significant differences among materials.

Hypothesis 3: (null)

Tear rate and setting time will have no effect on the tear strength of the materials.

Specific Aim 3. Tear strength specimens will be divided into four experimental groups. Tear strength of the impression materials will be evaluated at 1 mm/min and at
500 mm/min. These specimens will be tested immediately after preparation and 24 hours following preparation.

Aim 3

To evaluate the elastic recovery from tensile stress test.

Hypothesis 4: (null)

The test will find no difference in elastic recovery between silicone, polyether, and hybrid silicone/polyether impression materials.

Specific Aim 4. Five silicone materials and a hybrid polyether/silicone material will be evaluated. A one-way ANOVA and Tukey’s Test will be used to find significant differences among materials.

Hypothesis 5: (null)

The elastic recovery from tensile stress test will not correlate with the elastic recovery from compressive strain test.

Specific Aim 5. Elastic recovery of six elastomeric impression materials will be determined using the elastic recovery from tensile strain test and the ISO 4823 (section 9.7) elastic recovery test. The correlation of the two tests will be examined with a linear regression analysis.
FLOW OF REGULAR AND FAST SETTING ELASTOMERIC IMPRESSION MATERIALS

by

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Format adapted for thesis

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ABSTRACT

Statement of problem. Precise impressions are crucial for preparing accurate dental restorations. For dental impressions, it is important to have materials with increased flow at the end of the setting time to increase the ability of the impression material to capture the prepared subgingival margins and decrease folds in the impression material. Purpose. This research compares flow, as measured by the shark fin test, over thirty second intervals of eight different impression materials with two viscosities (light and extra light) and two setting times (fast and regular). Material and methods. Flow was measured using a shark fin testing apparatus (3M ESPE, Seefeld, Germany). Each impression material was injected into a metal cup. A weighted metal caste was suspended above the metal cup of impression material. The caste was dropped into the impression material following different time intervals for different specimens. Material flowed up into a triangular notch in the caste creating a “shark fin”. The mold was then placed into an incubator for the manufacturers setting time. When the mold was removed, the height of the “shark fin” was measured. The data was analyzed using a three factor ANOVA, separate ANOVA’s and Tukey HSD post-hoc analysis was used to determine significant inter group differences (p=.05). Results. Significant differences in shark fin height were recorded for the various impression materials. In general low viscosity impression materials produced higher flow. The Polyether material produced greater flow than the addition silicone and hybrid materials. Impression material, fast or regular setting time and measurement time were highly significant factors (p<.01). Conclusions. Based on the limitations of this study and the materials used, polyether impression materials have better flow than silicone and hybrid materials. Impression
material selection should be based in part upon the flow of the impression material, the depth of the preparation and the speed of the operator.
INTRODUCTION

It is important for impression materials to have a low viscosity when trying to make impressions of interproximal spaces and gingival crevices. The lower the viscosity, the better the material will flow into these spaces and the more accurate the impression. Addition silicone impression materials are the most widely used impression materials for final restorations. Broadly, these materials are available in four consistencies (viscosities): [extra] light-bodied, medium-bodied, heavy-bodied, and putty. Light-bodied materials contain a lower volume percent of silica fillers than heavy-bodied materials. Decreased filler content in light-bodied materials increases their flow. The multiple mix technique for making impressions uses a combination of impression materials, generally a heavy bodied material in a tray and a light or extra light consistency impression material in the syringe. The heavy body material provides support for the light or extra-light bodied material, which is responsible for flowing into the spaces that heavy bodied material could not reach.

3M ESPE developed the shark fin test, a device for testing the flow of impression materials. Benchimol et al, Broome et al, Stipho et al and Klettke et al published results with this shark fin testing apparatus. The initial design for this study simulated the flow of impression material from 1.5 N of applied force. The seating forces for elastomeric impression materials, as determined by Sotiriou, reach as high as 10N. To more closely simulate clinical forces, the device was modified by the addition of a 268.20g (~2.6N) weight on top of the metal caste. The weight increases the pressure applied to the material to more than 4 N. The weight more closely approximates the
average pressure used by a clinician while loading an impression tray into a patient’s mouth.

An ideal impression material will have adequate working time but a fast intraoral setting time. The clinician needs time to inject material into the sulcus, place the impression material into the tray and position it in the mouth, but the material should set rapidly to reduce time in the patient’s mouth. The “perfect material” will exhibit high flow initially and quickly transition to no flow. The high flow initially will allow material to flow into crevices while a rapid reduction in flow will reduce distortion while the material polymerizes in the mouth. Generally, impression materials are available with a regular and fast setting time. Materials that have a fast setting time have less retarder. Their polymerization is less delayed resulting in a shorter working time than regular set materials. Fast set materials would be expected to flow for a shorter period of time than regular set materials.11

This study examined the flow of 4 addition silicone, 1 polyether, and 1 hybrid silicone/polyether materials with both regular and fast setting times. Materials tested were light-bodied consistency, and when available, extra-light bodied consistency. The null hypothesis was that there will be no difference in flow between silicone, polyether, and hybrid silicone/polyether impression materials.
MATERIAL AND METHODS

Flow was measured using a metal device designed and manufactured by 3M ESPE (Seefeld, Germany) termed the shark fin testing apparatus. The apparatus consisted of a cup (A), a cylindrical caste with a triangular notch transversing the interior axially (B), a housing to suspend the caste over the cup (C), a pin to attach the caste to the housing (D), and a weight to apply a force for the caste to drop into the cup (E) (fig 1).

Five impression materials were tested and listed in Table I. Each impression material was injected into the cup which was kept at oral temperature. A timer was started immediately after injecting the material. All excess material was wiped away with a spatula making the material flush with the rim of the cup. At 30 second intervals, the pin was released, dropping the caste into the impression material. Specimens of each material were prepared by dropping the caste after 30 sec, 60 sec, 90 sec, 120 sec, 150 sec, 180 sec, and 210 sec after injecting the impression material. Material flowed into a triangular notch in the caste creating a “shark fin”. The mold was then placed into a 37C incubator (Kendro Laboratory Products, Asheville, NC) for the manufacturers setting time. After the setting time, the mold was removed and disassembled. Excess material was cut away to reveal the shark fin. The shark fin height was defined as the vertical distance from the tallest part of the fin to the juncture where the fin intersected the flat surface of specimen. The height of the shark fin was measured to the nearest hundredth of a millimeter using a digital caliper (DC150; Duratool, Tali City, Taiwan).
RESULTS

The mean shark fin height of each material is graphed by time interval in Figure 3 (light body) and Figure 4 (extra-light body).

A qualitative examination of Figure 3 reveals that the polyether material produced high shark fin values initially, maintained high shark fin values for 2 to 3 minutes and abruptly produced short shark fin specimens. Figure 4 reveals that extra-light bodied materials (excluding fast set Aquasil Ultra) behaved with similar flow characteristics to the polyether. This qualitative analysis was quantitatively confirmed by the results of the ANOVA and Tukey’s Test among the materials at each time interval.

The ANOVA revealed that there were significant differences among materials at all time intervals. The Tukey’s Test differentiated materials into significantly different groups at each time point. At the first time interval (30 seconds), the hybrid, polyether, extra-light bodied silicones (excluding fast set Aquasil), and several silicones were all grouped in the statistical group with the greatest flow. Between the 1 minute and 2 minute time interval, the polyether and the extra-light silicones (excluding fast set Aquasil) were in group(s) significantly greater than all silicone materials. At the 2:30 interval, the regular set polyether material and the regular set extra-light bodied materials were in the group with the greatest flow. By 3:30 minutes, all materials stopped flowing excluding the regular set polyether and extra-light bodied silicones, and after 4 minutes, all materials stopped flowing.
DISCUSSION

The results of this study suggest that the null hypothesis should be rejected. The shark fin test measured significantly different values of flow for different materials. The ability of the device to discriminate between materials validates its utility.

The results of qualitative and quantitative analysis show that polyether materials and the extra-light bodied materials (excluding fast set Aquasil) demonstrate an ideal flow profile. These materials experienced high initial flow (at the 30 second interval), maintained high flow (from the 1 minute to the 3 minute interval) and ceased to flow by 3:30-4 minutes.

Previous studies that have examined the flow of impression materials using the shark fin testing device have achieved similar results. Benchimol et al\textsuperscript{5}, Broome et al\textsuperscript{6} and Klettke et al\textsuperscript{8} concluded that Impregum, a polyether impression material, provided significantly greater flow than the addition silicone materials tested. As expected, a comparison of the shark fin height data to previous studies reveals that shark fin heights in this study of identical materials are considerably higher, due to the addition of the 350g weight. The addition of the weight made it more difficult to find statistical differences among materials at early time intervals, however, it assisted in differentiating materials at later time intervals. For example, Benchimol\textsuperscript{5} could statistically differentiate between Impregum and Aquasil XLV after a 25 second delay, however, in the current study, these two materials could not be differentiated after a 30 second delay.

Balkenhol performed a correlation analysis between the shark fin test and relevant rheological properties of a silicone, polyether and hybrid impression material. A rotational rheometer was used to measure storage modulus and phase angle, two
parameters that measure chain-linking in polymers. Surprisingly, not all materials showed a correlation between shark fin height and storage modulus or phase angle. Balkenhol suggests that measuring shear viscosity, a parameter influenced by the interaction of monomer molecules, might better explain the results of the shark fin test.\textsuperscript{12}

The current study measured flow of impression materials on a dry surface. Clinically, impressions are taken in an environment exposed to saliva. Future research could examine the ability of impression material to flow on a wet surface. This study could be performed by modifying the shark fin testing method; the notched caste could be coated in water or artificial saliva before it is dropped into the impression material.

**CONCLUSION**

Polyether impression materials provide significantly greater flow than silicone and hybrid polyether/silicone materials. Impression material selection should be based in part upon the flow of the impression material, the depth of the preparation, the number of preparations and the speed of the operator.
REFERENCES


11. Klettke T, Hampe R. Prolongation of working time with polyether retarder. 0122
Table I. Impression materials used in this study

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<tr>
<th>Product</th>
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<th>Type</th>
<th>Viscosity</th>
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<td>Silicone</td>
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<td>060404</td>
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<td></td>
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<td></td>
<td></td>
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### Table II Shark fin heights of light bodied impression materials $\textit{mean}(SD)$

<table>
<thead>
<tr>
<th>Material</th>
<th>Shark fin height after 0:30 (mm)</th>
<th>Shark fin height after 1:00 (mm)</th>
<th>Shark fin height after 1:30 (mm)</th>
<th>Shark fin height after 2:00 (mm)</th>
<th>Shark fin height after 2:30 (mm)</th>
<th>Shark fin height after 3:00 (mm)</th>
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<tr>
<td>reg</td>
<td>20.98 (0.53)</td>
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<td>25.60 (0.49)</td>
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<td>fast</td>
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<td>reg</td>
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</table>

*Denotes extra-light bodied material

Values with similar superscripts are not statistically different
LEGENDS

Figure 1. Shark fin testing device

Figure 2. Shark fin specimen
Figure 3. Shark fin height of light bodied materials versus time (regular set materials are represented with a dashed line and fast set materials are represented with a solid line)
Figure 4. Shark fin height of extra-light bodied materials versus time (regular set materials are represented with a dashed line and fast set materials are represented with a solid line)
TEAR STRENGTH OF FIVE ELASTOMERIC IMPRESSION MATERIALS AT TWO SETTING TIMES AND TWO TEARING RATES

by

Nathaniel C. Lawson, John O. Burgess, DDS, MS, Mark Litaker, PhD

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Format adapted for thesis
ABSTRACT

**Problem/Aims.** Thin sections of impression materials are susceptible to tearing in gingival crevices and interproximal spaces. This study measures the tear strength of six fast and regular set impression materials after different setting times and at different tearing rates.

**Materials/Methods.** Tear strength specimens were prepared of four addition silicone materials: Aquasil (Dentsply, Konstanz, Germany), Imprint 3 (3M ESPE, Seefeld, Germany), Stand Out (Kerr, Orange, CA), Virtual (Ivoclar Vivadent, Schaan, Liechtenstein); one polyether material: Impregum (3M ESPE, Seefeld, Germany) and a new hybrid material: Senn (GC, Aichi, Japan) using a split mold. Specimens were divided into four groups (n=5). Group 1 and 2 were immediately removed from the mold and loaded in tension until failure using an Instron testing device. Groups 3 and 4 were tested 24 hours after fabrication. Groups 1 and 3 were tested at 1mm/min and groups 2 and 4 were tested at 500mm/min.

**Results.** A 1 factor ANOVA and Tukeys HSD test revealed differences among material brands (α=0.05) in all experimental groups. The polyether and hybrid material were in the lowest statistically significant ranking group for all experimental groups. A 3 factor ANOVA determined that a 500mm/min tearing rate and a 24hr set time produced higher tear strengths and that fast set materials produced greater tear strength than regular set materials.

**Conclusions:** Most addition silicone materials provide higher tear strengths than polyether and hybrid materials. Materials display higher tear strengths after longer set
times and at faster tearing rates. Impressions should be removed from the mouth with the fastest possible speed.

**Key words:** tear strength, impression materials, tearing rate, setting time, addition silicone

**CLINICAL RELEVANCE**

Addition silicone materials should be used in impressions requiring replication of gingival crevices or interproximal spaces to prevent tearing of thin sheets of material. Impressions should be removed from the mouth and separated from the model rapidly.
INTRODUCTION

Impressions should resist tearing when tensile stresses are applied during impression removal and cast separation from the set impression. Impression materials are most susceptible to tearing in gingival crevices and interproximal areas. Tearing in the impression causes defects which affect the accuracy of the final restoration\(^1\). Additionally, some impression material remnants remaining in the sulcus may produce inflammation reactions\(^2,3\). Therefore, it is necessary for impression materials to have maximum tear strength at the time of removal\(^4\).

The tear strength of impression materials has been measured using several different tests including the Trouser tear test\(^5,6,7\) which measures tear propagation and the Die C tear test\(^8,9\) which measures tear initiation and propagation. ANSI/ADA Specification 20 (4.3.10) describes the tear test for non-aqueous dental duplicating material and specifies the use of an ASTM standard Die C tear specimen. According to their specifications, specimens are to be fabricated and stored at 23\(^°\)C. One hour following fabrication, the specimen should be tested in tension at 254 mm/min\(^10\). A more clinically relevant tear strength specimen, developed by Boghosian\(^11\), was used in this study which mimics thin sheets of impression material in gingival crevices and interproximal areas.

This study examined the effect of setting time on the tear strength of the materials. Two setting times were examined, immediately after setting and 24 hours following setting. Testing immediately following specimen preparation mimics oral removal and 24 hour testing mimics cast removal. Shorter setting times for impression materials are more convenient for clinicians, particularly when a single tooth has been
prepared. If the manufacturer’s suggested set time is not accurate and the impression material has not completely polymerized before removal, the impression material will tear. Therefore, testing materials 24 hours after setting will also determine if setting time beyond the manufacturer’s directions will affect tear strength.

Another variable examined in tear strength testing is the tearing rate, the speed at which the materials are removed from the mouth or the cast from the impression. Elastomeric impression materials are viscoelastic and the tearing rate will affect the tear strength of the material\textsuperscript{12}. Clinically, the speed at which impressions are removed from the oral cavity and the cast will affect the tear strength of the impression material. Therefore, the impression should be removed rapidly\textsuperscript{13}. Klooster performed a study loading ASTM specimens at 100, 200, and 500 mm/min and determined that higher strain rates produced higher tear strength\textsuperscript{14}.

This experiment measured the tear strength of five regular and fast set elastomeric impression materials with two variables: setting time (immediately after setting and 24 hours after setting) and tearing rate (1mm/min and 500mm/min). The hypothesis is that setting time, tearing rate, and if the material is regular or fast set will have no effect on the tear strength of the material.

**METHODS AND MATERIALS**

A plexiglass mold was fabricated at the UAB School of Dentistry to perform tear strength testing. The mold contained a 70mm (length) x 10mm (width) indentation that was 1.9mm deep. A 90° triangular notch was inserted along the 10mm width of the indentation at the center of its length. The mold produced a 0.1 mm thick space between
the top of the triangular notch and the lid of the mold. The section of the specimen that was between the top of the triangular notch and the lid of the mold is most susceptible to tearing and the thickness of the specimen in that section is referred to as the film thickness.

Six commercially available impression materials were used for this study (Table 1). The specimens were prepared by dispensing impression material into the plexiglass mold. A small amount of material was extruded and discarded to ensure proper mixing in the dispensing tip. A timer was started immediately after the impression material was first dispensed from the cartridge into the mold. The cover of the mold was applied with finger pressure and secured to the base. Excess material flowed out of the mold from two holes in the lid (figure 1). The specimens were fabricated at 24° C and 51% humidity before being placed in the incubator (Kendro Laboratory Products, Asheville, NC) at 37° C for the manufacturer’s set time (listed in Table 1). After setting, the mold was removed from the incubator and the specimen was removed from the mold. The excess material from the edges of the specimen was trimmed using a razor blade and benchmarks were drawn on the specimen 10mm on either side of the center line.

The specimens were divided into four groups with n = 5 for each group. Immediately following specimen preparation, the specimens from group 1 and 2 were secured into the Instron universal testing machine (Instron Corp., Canton Mass). The specimen was gripped on both sides by a pneumatic clamp at the location of the previously applied benchmarks (figure 2). Before the test began, the jig was adjusted so that the specimen was neither in compression or tension. Starting 2.5 minutes after the
specimens were removed from the incubator, the specimens were loaded in tension until failure with a crosshead speed of 500 mm/min (group 1) and 1 mm/min (group 2).

Groups 3 and 4 were stored at 24°C for an additional 24 hours after preparation. Tear strength testing was performed identically to group 1 and 2 at 500 mm/min (group 3) and 1 mm/min (group 4). The area of the tear was nominally 1mm². The tear strength was calculated as: tear strength = ultimate tensile strength / (10mm x 0.1mm).

The data from every group was subjected to a two-way analysis of variance (ANOVA) and Tukey’s HSD test ($\alpha = 0.05$). A three-way ANOVA was used to compare regular and fast set materials ($\alpha = 0.05$). The groups (1-4) were compared using a three-way ANOVA and a Tukey’s HSD test ($\alpha = 0.05$).

RESULTS

The tear strength of each regular setting material for each group is graphed in Figure 3 and the tear strength of each fast setting material for each group is graphed in Figure 4. Means and standard deviations of tear strength values of each group are provided in Table 2. The one-way ANOVA revealed a significant difference between brands of materials in each experimental group (1-4). A Tukey’s HSD test ranked materials into statistically different categories. In all groups, the polyether and hybrid material were ranked in the statistically significant category with the lowest tear strength. A three-way ANOVA revealed that fast set materials provided a greater tear strength than regular set materials ($p=0.004$).

A three-way ANOVA test showed a significant difference between overall materials tested after different setting times and at different tearing rates. A Tukey’s
comparison HSD revealed that longer setting time and a faster tearing rate produced statistically higher tear strengths. Individual materials were tested to see if there was a statistically different tear strength for longer setting times and increased tearing rates for each material. Longer setting times produced significantly greater tear strength for all materials except: Imprint 3 LB Fast, Aquasil LB, Virtual XL Fast. Fast tearing rates produced significantly greater tear strength for all materials except: Imprint 3 LB, Virtual XLB, Senn LB Reg and Fast, and Impregum Reg and Fast.

**DISCUSSION**

The specimens fabricated for this study are different than tear strength specimens used in previous studies. Our specimens measure the maximum tensile yield stress of a thin film of impression material. This test was developed by Boghosian to produce clinically relevant results\(^\text{11}\). The thin film of impression material models the thin sheets of material in interproximal and subgingival areas subject to clinical tearing. Impression material has been shown to penetrate a crevice as thin as 0.05mm in vitro\(^\text{15}\). Our study used specimens with a film thickness of 0.1mm (instead of 0.2mm\(^\text{11}\) or 0.4mm\(^\text{16}\)) to mimic the very thin regions of impression material produced clinically.

Boghosian’s tear strength study evaluated 10 impression materials. Comparing the numerical results of our study to Boghosian’s data reveals that our tear strength values are almost twice as great as his reported data. Ripps also reports much greater tear strength values of identical materials. The most important difference between our study and Boghosian’s and Ripps’ studies is that our specimens had a 0.1mm film thickness and Boghosian used a 0.2mm film thickness\(^\text{11}\) and Ripps used 0.4mm film thickness.
This observation suggests that thicker film thickness produces a lower tear strength (which is measured as force/area).

A study by Whiteman examined Aquasil, Imprint 3 and Impregum using the ADA specification 19 tear test. The numerical results from Whiteman’s study can not be directly compared to this study because the two tests measure different properties (tear initiation and propagation vs. thin sheet tensile strength). In Whiteman’s study, Impregum and Aquasil LB showed a statistically greater tear strength than Imprint 3 LB Quick. This conclusion contradicts our results which show that Imprint 3 LB Quick and Aquasil LB have no statistical difference from each other but both have statistically greater tear strength than Impregum.

The ANSI/ADA standard specifies that tear strength specimens should be tested 1 hour following polymerization. Clinically, impressions are subjected to tearing forces immediately after the manufacturer’s setting time. This study reveals that there are significant differences between testing immediately after the setting time and 24 hours following the setting time. Testing immediately following the setting time is a clinically relevant method.

As noted in the results, several materials did not show significant differences between 1mm/min tearing rate and a 500mm/min tearing rate. Impregum and Senn both showed greater tear strength values at a 1mm/min tearing rate than 500mm/min immediately after setting. An explanation for this behavior is that these impression materials are undergoing further polymerization in the additional amount of time it takes to deform these materials at 1mm/min.
A limitation of this study is that the exact cross-sectional area of the specimen during tearing could not be accurately determined. As the specimens were deformed, they experienced necking and the cross-sectional area decreased. The cross-sectional area at the time of tearing is needed to calculate tear strength. The most feasible method to measure the change in cross-sectional area would be to record the specimen thickness with a video extensometer. We could not find an extensometer capable of accurately measuring such small changes in length.

CONCLUSION

In conclusion, addition silicone materials have greater tear strength than polyether and polyether/addition silicone hybrid materials. Materials undergo continued cross-linking past the manufacturers suggested set time which is indicated by the increased tear strength these material experience after 24hrs of additional setting. Materials also have higher tear strength at increased tearing rates. Clinically, impressions should be removed from the mouth and separated from the model at the fastest possible speed.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the manufacturers for donating all of the impression materials. These materials were received from the US divisions. The authors would like to acknowledge Sandre McNeal for her assistance with the statistical analysis.
REFERENCES


## TABLES

Table 1 Light body impression materials used in this study

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<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Composition</th>
<th>Set Time</th>
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<td>Aquasil Ultra</td>
<td>Dentsply</td>
<td>Addition Silicone</td>
<td>reg 5:00 min</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>fast 3:00 min</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>reg 5:00 min*</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>fast 3:00 min*</td>
</tr>
<tr>
<td>Imprint 3</td>
<td>3M ESPE</td>
<td>Addition Silicone</td>
<td>reg 6:30 min</td>
</tr>
<tr>
<td></td>
<td></td>
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</tr>
<tr>
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<td>Ivoclar Vivadent</td>
<td>Addition Silicone</td>
<td>reg 7:05 min</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>fast 4:05 min</td>
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<td></td>
<td></td>
<td>fast 4:15 min*</td>
</tr>
<tr>
<td>Impregum Soft</td>
<td>3M ESPE</td>
<td>Polyether</td>
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<tr>
<td></td>
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<td></td>
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</tr>
<tr>
<td>Senn</td>
<td>GC</td>
<td>Hybrid (Addition Silicone/Polyether)</td>
<td>reg 7:00 min</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>fast 4:10 min</td>
</tr>
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</table>

*denotes extra-light body material
Table 2 Tear strength of regular set and fast set impression materials (mean ± SD)

<table>
<thead>
<tr>
<th>Material</th>
<th>Group 1 (MPa)</th>
<th>Group 2 (MPa)</th>
<th>Group 3 (MPa)</th>
<th>Group 4 (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aquasil LB</td>
<td>reg 8.12 ± 0.86</td>
<td>fast 8.15 ± 0.88</td>
<td>9.02 ± 0.86</td>
<td>7.64 ± 0.78</td>
</tr>
<tr>
<td></td>
<td>fast 8.15 ± 0.88</td>
<td>fast 7.13 ± 0.61</td>
<td>10.29 ± 0.60</td>
<td>8.08 ± 1.19</td>
</tr>
<tr>
<td>Aquasil XLB</td>
<td>reg 8.24 ± 0.89</td>
<td>fast 6.92 ± 0.68</td>
<td>8.92 ± 0.66</td>
<td>7.79 ± 1.19</td>
</tr>
<tr>
<td></td>
<td>fast 8.24 ± 0.89</td>
<td>fast 6.01 ± 0.43</td>
<td>10.20 ± 0.39</td>
<td>7.20 ± 0.71</td>
</tr>
<tr>
<td>Imprint 3 LB</td>
<td>reg 7.00 ± 1.60</td>
<td>fast 8.08 ± 1.46</td>
<td>10.22 ± 1.41</td>
<td>8.27 ± 1.35</td>
</tr>
<tr>
<td></td>
<td>fast 7.00 ± 1.60</td>
<td>fast 6.68 ± 0.25</td>
<td>8.81 ± 0.89</td>
<td>7.32 ± 1.05</td>
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<tr>
<td>Virtual LB</td>
<td>reg 6.07 ± 0.85</td>
<td>fast 5.47 ± 0.28</td>
<td>7.40 ± 0.70</td>
<td>5.54 ± 0.49</td>
</tr>
<tr>
<td></td>
<td>fast 6.07 ± 0.85</td>
<td>fast 4.71 ± 0.35</td>
<td>5.98 ± 1.19</td>
<td>5.47 ± 0.27</td>
</tr>
<tr>
<td>Virtual XLB</td>
<td>reg 4.71 ± 0.41</td>
<td>fast 4.89 ± 0.39</td>
<td>5.42 ± 0.34</td>
<td>4.74 ± 0.44</td>
</tr>
<tr>
<td></td>
<td>fast 4.71 ± 0.41</td>
<td>fast 4.91 ± 0.35</td>
<td>5.34 ± 0.93</td>
<td>4.30 ± 0.46</td>
</tr>
<tr>
<td>Impregum LB</td>
<td>reg 2.05 ± 0.28</td>
<td>fast 2.03 ± 0.21</td>
<td>3.95 ± 0.33</td>
<td>3.66 ± 0.71</td>
</tr>
<tr>
<td></td>
<td>fast 2.05 ± 0.28</td>
<td>fast 2.21 ± 0.17</td>
<td>3.43 ± 0.40</td>
<td>3.42 ± 0.26</td>
</tr>
<tr>
<td>Senn LB</td>
<td>reg 1.89 ± 0.37</td>
<td>fast 2.16 ± 0.15</td>
<td>3.48 ± 0.22</td>
<td>2.75 ± 0.29</td>
</tr>
<tr>
<td></td>
<td>fast 1.89 ± 0.37</td>
<td>fast 2.49 ± 0.04</td>
<td>3.52 ± 0.20</td>
<td>2.75 ± 0.38</td>
</tr>
</tbody>
</table>
FIGURES

Figure 1 Tear strength mold

Figure 2 Tear strength specimen in Instron testing device
Figure 3  Tear strength (mean ± s.d.) for regular set materials

Figure 4  Tear strength (mean ± s.d.) for fast set materials
NOVEL METHOD FOR MEASURING ELASTIC RECOVERY OF ELASTOMERIC IMPRESSION MATERIALS

by

Nathaniel C Lawson, John O Burgess, DDS, MS, Mark Litaker, PhD

In preparation for Dental Materials

Format adapted for thesis
ABSTRACT

Objectives: This study compares two test methods, a novel elastic recovery from tensile strain test and the ISO elastic recovery test for five polyvinyl siloxane materials (Aquasil Ultra, Examix, Genie, Imprint 3, and Stand Out) and one hybrid material (Senn) with normal set times and light consistencies.

Methods: In the novel tensile recovery test, material was dispensed into a custom brass mold, pressed between glass slides and placed in a 34°C water bath for the manufacturer’s oral setting time. After polymerization, specimens (n=5 per group) were loaded in tension with a crosshead speed of 300 mm/min until their length was increased by 50% or 100% of the initial length. 2 hours ± 15 minutes following specimen deformation, the change in length of the specimens was measured using a light microscope. Specimens were produced (n=5) with the same mold and tested in tension until failure at 200 mm/min. The maximum elongation at failure was recorded. Elastic recovery from compressive strain was tested using the ISO elastic recovery test. Specimens of each materials (n=3) were prepared and tested following ISO standard 4823 (9.7). The data was analyzed with an ANOVA and Tukey-Kramer HSD Test (p=.05). A correlation between tests was analyzed by a linear regression

Results: Materials were found to be significantly different in both tests. The Tukey’s test ranked Genie and Senn into the group(s) with the least elastic recovery in all tests. All materials exceeded a 125% elongation before failure. A linear correlation was found between the ISO method and a 100% tensile strain, but not a 50% tensile strain.

Significance: Elastic recovery from compressive strain can only partially predict elastic recovery from tensile strain, suggesting that elastic recovery from tensile strain is a
relevant test for measuring elastic deformation when removing an impression form the mouth.

KEYWORDS
Elastic recovery, memory test, impression material, permanent deformation, tensile strain
INTRODUCTION

The accuracy of an impression is crucial for the success of a final restoration\(^1\). In order for an impression material to accurately replicate a given tooth structure, it must not permanently deform while being subjected to the forces of oral removal\(^2\). ISO 4823 specifies the requirements for elastic recovery of elastomeric impression materials. The ISO test measures recovery from a 30% compressive strain, but does not specify a test for elastic recovery from tensile strain\(^3\). Other tests have been explored for measuring elastic recovery from tensile stain\(^4,5\), shear strain\(^4\), and bending and torsion\(^6\).

Clinically, impression materials are subjected to both compressive and tensile forces. For example, materials are stretched in tension when they are pulled over undercuts, sharp line angles, and interproximal spaces\(^7\). A study by Sotiriou et al. shows that maximum [tensile] removal forces of impression materials are greater than maximum [compressive] seating forces. They also show that maximum removal forces of elastomeric impression materials are greater than irreversible colloid materials\(^8\). To better predict the clinical performance of an elastomeric impression material, it is therefore necessary to measure both recovery from compressive and tensile elongation.

Many studies have compared the tensile and tear strength of elastomeric impression materials\(^9-12\). In tensile and tear strength testing, a specimen is stretched to its maximum tensile strength. The maximum tensile strength of a material is not relevant if the material undergoes permanent deformation during elongation. The tensile strength achieved by elastomeric impression materials is most clinically relevant when the material can be subjected to high forces but still recover to its original dimensions.
The purpose of this study is to examine the elastic recovery of six elastomeric impression materials after 50% and 100% tensile strain and compare the results to their elastic recovery from compressive strain. The maximum percent elongation of each material will also be determined to validate the test method. The null hypothesis is that (1) there will be no difference between elastic recovery of the different materials and (2) there will not be a linear correlation between elastic recovery values when materials are subjected to tensile and compressive strain.

**MATERIALS AND METHODS**

**Materials**

Five addition silicone polyvinyl siloxane materials and one hybrid polyether/polyvinyl siloxane material were used in this study. The materials are listed in Table 1.

**Elastic recovery from tensile strain**

A custom brass mold was fabricated by 3M ESPE (Seefeld, Germany). The mold was paddle shaped with a 2mm wide and 1.5mm thick inner bar. A 20mm long section of the inner bar was delineated by four semicircular notches in the mold (figure 1). Five specimens were made for each material. A timer was started as the impression material was dispensed into the brass mold. The mold was surrounded on both sides by a mylar film and pressed between two glass slides. After a 25 second working time, the specimen was placed in a water bath (Haake W13, Karlsruhe, Germany) at 34º C for the
manufacturer’s oral setting time. After setting, the specimen was removed from the water bath (Karlsruhe, Germany).

The specimen was dried and flash was trimmed using a spatula. The specimen was then removed from the mold and secured into the Zwick Z020 universal testing machine (Zwick, Ulm, Germany). The specimen was gripped on both sides by a clamp. After 1.5 minutes, the loading program was started. The specimen was initially loaded in tension to remove slack, and the distance between the two clamps was then recorded as an initial length. The specimen was loaded in tension with a crosshead speed of 200 mm/min until the distance between the two clamps was increased by 50% or 100% of the initial length. The specimens were returned to the initial length and removed from the mold. All specimens were stored at room temperature 24C on a piece of paper.

A control group of each material (n=5) was prepared using the brass mold. These specimens were not elongated following polymerization.

2 hours ± 15 minutes following specimen deformation, the specimens were placed on a stage and viewed under a light microscope. Using a computer attached to the stage, the distance between the centers of the semicircular notches on each side of the sample was measured. The average of the two distances was used as the final length. The percentage of elastic recovery was calculated using the formula: 100% - [(final length – control length) / control length] * 100%.

**Maximum elongation in tensile train**

Initially, specimens (n=5) were prepared and treated identically to elastic recovery from tensile strain specimens. Instead of the loading program used in the elastic recovery
protocol, however, these specimens were loaded in tension to failure at 200 mm/min. The maximum elongation at failure was recorded.

**Elastic recovery from compressive strain**

Elastic recovery from compressive strain was measured according to the specifications of ISO 4823 section 9.7. A timer was started as the impression material was dispensed into a cylindrical split mold. The filled mold was pressed between two polyethylene sheets covered by glass plates. After a 1 minute working time, the specimen was placed in a water bath (Haake W13, Karlsruhe, Germany) at 34º C for the manufacturer’s oral setting time.

After setting, the mold was removed from the water bath, dried, and placed in the ISO deformation device. 1 minute following setting, the specimen was compressed to 30% of its original height. The material was compressed from 1-2 seconds. After allowing the material to recover for 2 minutes, the height of the specimen was re-measured. Percent elastic recovery was calculated using the formula: 100% - [(final length – initial length) / initial length] * 100%. All specimens were cut in half and checked for air voids. Specimens with voids were excluded and repeated.

**Statistical Analysis**

Differences between percent elastic recovery (tensile and compressive) among materials was analyzed using a one-way ANOVA (α = 0.05) and a Tukey’s Test (α = 0.05). A correlation between elastic recovery from tension and elastic recovery from compression was analyzed by linear regression (α = .05).
RESULTS

Values are presented of percentage of elastic recovery from compressive strain, 50% tensile strain, 100% tensile strain and percentage elongation prior to failure (Table II). A one-way ANOVA revealed significant differences in elastic recovery from tensile strain (50% and 100%) and compressive strain among materials. The Tukey’s test divided the materials into significantly different groups. All materials provided percent elongations above 125% prior to failure.

To analyze the relationship between elastic recovery from tensile strain and compressive strain, the mean tensile elastic recovery of each material from a 50% strain (figure 3) and 100% strain (figure 4) was plotted against its corresponding mean compressive elastic recovery value. Elastic recovery from 50% tensile elongation was not significantly linearly correlated with elastic recovery from compressive strain (p=.08). A significant linear correlation between tensile elastic recovery from 100% strain and compressive elastic recovery was discovered (p=.04). Increased tensile elastic recovery is associated with increased compressive recovery. The linear relationship accounts for 83% of the variation (r = 0.83).

DISCUSSION

The results of the study revealed that all elastomeric impression materials experience permanent deformation following tensile strain. Additionally, different materials produce greater elastic recovery from tensile strain than others disproving the first null hypothesis.
Senn, the only material that is not a traditional polyvinyl siloxane, consistently ranked among the two materials with the least elastic recovery. Senn is a class of material termed hybrid which is a polymer containing polyether and siloxane groups. Hybrid materials were introduced to combine the physical properties of polyether and polyvinyl siloxane materials. Polyether materials yield less elastic recovery than polyvinyl siloxane materials. Therefore, the reduced elastic recovery achieved from hybrids can be attributed to the addition of polyether chemistry.

A linear correlation was observed between the elastic recovery of materials from compressive strain and a 100% tensile strain. A significant linear correlation was not found between elastic recovery from compressive strain and a 50% tensile strain. Therefore, the second null hypothesis can only be partially accepted. A study by Blomberg\textsuperscript{4} found a strong correlation between elastic recovery from tensile and compressive strain and concluded that only one method is needed. Blomberg’s study only considered one brand of material from each available compositional group while this study explored five silicone materials. Mansfield et al\textsuperscript{5} performed a very similar study comparing recovery from tensile and compressive strain. He prepared tensile specimens that were elongated by a 50% tensile strain. He concluded that both recovery from compressive and tensile strain were necessary tests. The results of the present study suggest that elastic recovery from compressive strain and elastic recovery from tensile strain are both distinctive and clinically relevant properties. This test method can identify materials that will not perform well clinically in dentition with large undercuts, and resulting high tensile strain.
In this study, specimens are elongated to up to 100%. Mansfield that reported that impression materials are subjected to 50% tensile strains clinically.\textsuperscript{5} Although it has not been proven that impression materials are stretched beyond 50% of their length in clinical situations, it was observed in this study that specimens of all materials exceeded 125% tensile elongation before failure or tearing. Therefore, in situations of clinical tearing, some portion of the material must also be elongated beyond 125% of its length for the tearing to occur. In clinical situations where tearing \textit{almost} occurs, it is reasonable to assume that a material elongates \textit{almost} 125% of its length.

After an addition silicone impression material sets, it will undergo continued cross-linking causing shrinkage\textsuperscript{13}. Shrinkage was determined by measuring the change in length of a specimen in the control group that was treated identically to all other specimens excluding the elongation. Our results compensate for the effects of shrinkage and present only the change in length from elongation recovery. The effect of shrinkage is accounted for while measuring the dimensional stability of an impression material. ISO\textsuperscript{3} and the ADA\textsuperscript{14} have developed separate methods for measuring the dimensional change of impression materials and this study reports purely elastic recovery.

Future studies might consider the permanent deformation of materials from strains below 50%.

\textbf{CONCLUSION}

All materials exceeded elongations of 100% in tension prior to failure, justifying the elongation protocol of this study. Recovery from compressive strain was only correlated to recovery from tensile strain after a 100% strain, not 50% strain. Assuming
some materials are subjected to 50% tensile strain *in vivo*, measuring elastic recovery from tensile strain *in vitro* is a relevant test. The Senn (hybrid) and Genie (silicone) had significantly less recovery than all other materials after tensile and compressive strain. Clinically, these materials should be avoided in impressions that are subjected to large tensile and compressive forces.
REFERENCES


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Table I. Impression materials used in this study

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Type</th>
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<tr>
<td>Aquasil Ultra</td>
<td>Dentsply Caulk</td>
<td>Silicone</td>
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<td>Examix</td>
<td>GC</td>
<td>Silicone</td>
<td>4:00 min</td>
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<tr>
<td>Genie</td>
<td>Sultan Chemists</td>
<td>Silicone</td>
<td>4:30 min</td>
<td>070725269</td>
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<td>Imprint 3</td>
<td>3M ESPE</td>
<td>Silicone</td>
<td>3:30 min</td>
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<td></td>
<td></td>
<td></td>
<td>K246870</td>
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<tr>
<td>Standout</td>
<td>Kerr</td>
<td>Silicone</td>
<td>2:30 min</td>
<td>6-1030</td>
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<td>Senn</td>
<td>GC</td>
<td>Hybrid</td>
<td>4:00 min</td>
<td>0508222</td>
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Table II. Elastic properties of impression materials in this study mean(SD)

<table>
<thead>
<tr>
<th>Material</th>
<th>Max Elongation (%)</th>
<th>Compression (%)</th>
<th>50% Tension (%)</th>
<th>100% Tension (%)</th>
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</thead>
<tbody>
<tr>
<td>Aquasil Ultra</td>
<td>212.34 (14.58)c,d</td>
<td>99.83 (0.01)b</td>
<td>99.91 (0.06)a,b</td>
<td>99.21 (0.15)b</td>
</tr>
<tr>
<td>Examix</td>
<td>278.35 (36.21)a,b</td>
<td>99.58 (0.01)a</td>
<td>99.89 (0.05)a,b</td>
<td>99.85 (0.03)a</td>
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<tr>
<td>Genie</td>
<td>164.94 (27.80)d,e</td>
<td>99.54 (0.08)b</td>
<td>99.13 (0.20)d</td>
<td>97.72 (0.25)d</td>
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<tr>
<td>Imprint 3</td>
<td>238.29 (17.34)b,c</td>
<td>99.39 (0.03)a</td>
<td>100.04 (0.10)a</td>
<td>99.91 (0.17)a</td>
</tr>
<tr>
<td>Standout</td>
<td>301.72 (35.69)a</td>
<td>99.74 (0.02)a</td>
<td>99.73 (0.05)b</td>
<td>99.28 (0.05)b</td>
</tr>
<tr>
<td>Senn</td>
<td>140.96 (20.01)c</td>
<td>99.82 (0.01)c</td>
<td>99.35 (0.06)c</td>
<td>98.35 (0.15)c</td>
</tr>
</tbody>
</table>

Values with similar superscripts are not statistically different
FIGURES

Figure 1. Elastic recovery from tensile strain specimen (upper) and mold (lower)

Figure 2. Specimen elongation
Figure 3. Elastic recovery from 50% tensile strain vs. elastic recovery from compressive strain
Figure 4. Elastic recovery from 100% tensile strain vs. elastic recovery from compressive strain
SUMMARY

Conclusions

1. The shark fin test is a valid device for discriminating the flow of elastomeric impression materials. Polyether materials provide significantly more flow than silicone materials. The hybrid polyether/silicone material provided flow comparable to a silicone material. Extra-light bodied materials provided significantly more flow than light bodied materials. *Hypothesis 1 (null) is rejected.*

2. The thin film tear strength test is a valid and reproducible method for determining the resistance of elastomeric impression materials to tearing. Materials rank in the following order in regard to tear strength: silicone > polyether and hybrid. A longer setting time and faster tearing rate increases the tear strength of impression materials. *Hypotheses 2 (null) and 3 (null) are rejected.*

3. The elastic recovery from tension test is a viable method for measuring the elastic recovery of elastomeric impression materials. This test correlates with the results of the ISO specification for elastic recovery when materials are stretched to a 100% strain. There is not a significant linear correlation between recovery from compressive strain and a 50% tensile strain. The hybrid polyether/silicone
material ranked among the materials with least elastic recovery from tensile and compressive strain. Hypothesis 4 (null) is partially rejected and hypothesis 5 (null) is rejected.

Significant Recent Publications

Following the laboratory conclusion of the experiments presented in this study, a Summer 2007 ADA laboratory testing method review was released for elastomeric impression materials. The document’s scope was described as a review of protocol for obtaining “clinically relevant laboratory information” for elastomeric impression materials. The following properties, selected to provide comparative scientific consumer information, were described: (1) detail reproduction, (2) linear dimensional change, (3) compatibility with gypsum, (4) elastic recovery, (5) strain in compression, (6) tear strength, and (7) working time. The document provides a brief description of the justification for the current ADA testing methods and the clinical limitations of these tests. For example, the detail reproduction test is criticized for operating in a dry environment and the dimensional stability test is questioned for operating in an environment with uncontrolled temperature and humidity. Although the problems are addressed, no changes were added to the protocols.

The most significant contribution contained in the document is the addition of a tear strength testing method for elastomeric impression materials. The protocol for this test is based on the thin film test developed by Boghosian. This protocol is nearly identical to the protocol used in this manuscript with several minor alterations. First, the ADA suggests placing the impression material into a temperature/humidity chamber
while injecting it into the mold and then transferring the mold into a water bath. In the
current study, the material was injected into the mold in room temperature/humidity and
placed in an incubator for polymerization. Additionally, the ADA protocol specifies that
the cross sectional area of the specimen at failure should be measured with a Nikon
profile projector. In this study, the area at failure was assumed to be unchanged from the
specimen’s original dimensions. The justification for that decision is based on the
assumption that the measured permanent deformation of the specimens would be
nominal.

Future Work

Two step and putty impression techniques are used in dentistry to utilize the
increased stability of heavy-bodied and putty materials and the superior flow of light
bodied materials. Performing the two-step impression technique, a clinician fills a tray
with heavy bodied material and simultaneously injects light-bodied material around the
desired tooth structure. The tray is then fitted into the patient’s mouth. In the putty
technique, the clinician fills the tray with putty material and fits the tray into the patient’s
mouth. The tray is removed, revealing a customized tray. The putty material on the tray
is then covered with a light-bodied material and inserted back into the patient’s mouth.
In both of these techniques it is critical that the light-bodied material bonds to the heavy-
bodied or putty material.6

Several methods have been developed for testing the bond between the material
used in the tray (tray material) and the light-bodied material used in conjunction (wash
material).50-54 Tjan et al50,51 and DeWald52 developed a device with two perforated
hollow metal cylinders measuring 20mm high and 12.5 mm inner diameter. The bottom cylinder (tray cylinder) was filled with a tray material and set against a clean glass plate. The material was allowed to polymerize and the glass slide was removed. The other cylinder (wash cylinder) was placed on top of the tray cylinder and wash material was injected. A plunger was inserted into the top of the wash cylinder that pushed the wash material against the tray material. Excess material exited through the perforation, providing mechanical retention. After the wash material polymerized, the two cylinders were removed in tension at 10cm/min. The load at debonding was used to calculate the bond strength.

Sandik et al measured bond strength using a standard dumbbell shaped tensile strength mold. A resin spacer was fitted at the midpoint of the length of the mold. Tray material was inserted into mold up to the spacer, such that half the length of the mold was empty. After polymerization of the tray material, the spacer was removed and the other half of the mold was filled with wash material. Immediately after setting, the specimen was separated in tension at 10in/min. The maximum load was used to calculate bond strength.

Although tray to wash material bond strength is a clinically valuable parameter, neither ISO or ADA has developed a standard for measuring this property. To better determine this bond strength, we have developed a novel testing device at UAB. The device consists of a bottom tray cylinder, an alignment ring, an upper wash cylinder, and a threaded Instron attachment (figure 8). The interior of the upper wash cylinder is the shape of a truncated cone. The apex of the cone is a circle (2.5mm radius) that defines the area of contact between the tray and wash material. The protocol developed for this
test will be described. The tray cylinder will be filled with tray material and covered with a glass plate. Following polymerization, the glass plate will be removed and the alignment ring and wash cylinder will be placed on top of the tray cylinder. Wash material will be injected into the wash cylinder and pushed against the tray material. The threaded Instron attachment will be screwed onto the wash cylinder and the device will be mounted into a universal testing machine. After polymerization of the wash material, the two cylinders will be separated at 100 mm/min. The bond strength will be calculated using the following formula: 
\[
\frac{\text{max force}}{\pi \times 2.5 \text{mm}^2}.
\]

![Figure 8. Tray-wash bond strength device](image)

Preliminary testing has been conducted to evaluate this device. Although no presentable data has been generated, it was observed that materials failed by three mechanisms: (1) adhesive failure between the tray and wash materials (2) cohesive failure in the wash material and (3) cohesive failure in the tray material. Further testing should be done with this device to better understand the bond between wash and tray materials.
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