

Effects of Accelerated Artificial Daylight Aging on Bending Strength and Bonding of Glass Fibers in Fiber-Embedded Maxillofacial Silicone Prostheses

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Abstract

Purpose: The purpose of this study was to test the effect of different periods of accelerated artificial daylight aging on bond strength of glass fiber bundles embedded into maxillofacial silicone elastomer and on bending strength of the glass fiber bundles. **Methods and Materials:** Forty specimens were fabricated by embedding resinimpregnated fiber bundles (1.5-mm diameter, 20-mm long) into maxillofacial silicone elastomer. Specimens were randomly allocated into four groups, and each group was subjected to different periods of accelerated daylight aging as follows (in hours); 0, 200, 400, and 600. The aging cycle included continuous exposure to quartz-filtered visible daylight (irradiance 760 W/m²) under an alternating weathering cycle (wet for 18 minutes, dry for 102 minutes). Pull-out tests were performed to evaluate bond strength between fiber bundles and silicone using a universal testing machine at 1 mm/min crosshead speed. Also a three-point bending test was performed to evaluate bending strength of the fiber bundles. One-way ANOVA and Bonferroni post hoc tests were carried out to detect statistical significance (p < 0.05).

Results: Mean (SD) values of maximum pull-out forces (in N) for groups 1 to 4 were: 13.63 (7.45), 19.67 (1.37), 13.58 (2.61), and 10.37 (2.52). Group 2 exhibited the highest pull-out force that was statistically significant when compared to the other groups. Maximum bending strengths of fiber bundles were in the range of 917.72 MPa to 1124.06 MPa. Bending strength significantly increased after 200 and 400 hours of aging only.

Conclusions: After 200 hours of exposure to artificial daylight and moisture conditions, bond strength between glass fibers and heat-cured silicones is optimal, and the bending strength of the glass fiber bundles is enhanced.

Maxillofacial prostheses are constructed to transform facial disfigurements into natural-appearing reproductions of the missing parts, restoring function and improving appearance. Silicone elastomers have been widely used, as they are biocompatible, elastic, and can be pigmented to simulate skin tone.¹⁻³

In maxillofacial silicone prostheses, the silicone elastomer can be conventionally bonded to a retentive acrylic baseplate that holds the retentive magnets or clips.^{4,5} Also, it can be attached to a polyurethane layer⁶ when the prosthesis is adhesively retained.⁷ In addition, the silicone body can encapsulate a retentive glass fiber-embedded framework as in fiber-embedded maxillofacial prostheses.⁸ The framework is embedded within the prostheses, running through the silicone, and is attached to the retentive parts of magnets or clips. The framework does not strengthen the silicone elastomer properties nor cover the defect site as in traditional facial prostheses where the acrylic or titanium baseplate completely covers the defective site.⁹ Fiberembedded silicone prostheses tend to be more comfortable for patients.⁸ Fiber-reinforced composites (FRC) have proven to be suitable dental and medical biomaterials.^{10,11} They are used as reinforcement in removable prostheses, fixed partial dentures, periodontal splints, orthodontic retainers, and endodontic dowels.¹² Biocompatibility of the glass FRC material has been indicated by cell culture tests and animal experiments.^{13,14}

Serviceability of extraoral maxillofacial prostheses ranges from 6 to 24 months.^{15,16} Different prostheses can remain serviceable for 28, 13, 12, and 4 months for orbital, auricular, nasal, and combination prostheses, respectively;¹⁷ however, siliconebased maxillofacial prostheses require replacement every 6 to 18 months, as they lose elasticity, resistance to tear, and color stability when exposed to environmental factors such as sunlight energy (solar ultraviolet radiation), heat, and moisture.^{18,19} Also dust, air pollutants, and patient mishandling can affect mechanical and physical properties of facial prostheses.





Artificial aging has been used to investigate the interaction of silicone elastomers to simulated conditions that affect silicone prostheses. It can be in the form of accelerated artificial day-light aging, ^{15,18,20} immersion in simulated sebum solution, ^{21,22} acidic and alkaline perspirations, ²² and cleaning solutions; ^{23,24} however, accelerated artificial daylight aging changes the properties of silicones faster and in greater magnitude than other aging procedures. ²⁵

The effect of aging on mechanical properties of maxillofacial silicone elastomers has been reported in the literature, but its effect on the mechanical integrity of glass fibers embedded within silicone elastomer is not known. The aim of this study was to investigate the effect of different periods of accelerated artificial daylight aging (200, 400, and 600 hours) on bond strength of glass fiber bundles to heat-cured maxillofacial silicone elastomers and on the bending strength of the glass-fiber reinforcing framework. Accordingly, there are two null hypotheses: different periods of accelerated daylight aging have no effect on (1) bond strength of glass fiber bundles embedded to maxillofacial silicone elastomer, and (2) bending strength of glass fiber bundles.

Materials and methods

The specimen fabrication procedure has been previously described.²⁶ Forty specimens were constructed by embedding unidirectional glass fiber bundles (C&B Fibers, StickTech, Turku, Finland) (diameter = 1.5 mm, embedded length = 20 mm) into a heat-polymerized silicone elastomer (Cosmesil M511, Principality Medical, Newport, UK). The specimens were constructed using a sectional flask $(100 \times 80 \times 30 \text{ mm}^3)$ of two parts: lower section (100-mm long \times 80-mm wide \times 10-mm high) and upper section (100-mm long \times 80-mm wide \times 20-mm high) (Fig 1). The basal (lower) section had 16 holes (diameter 1.50 mm, 5 mm deep) into which the fiber bundles were fixed. The upper section had 16 cylindrical-shaped molds (14.40mm diameter, 20-mm long), where the silicone was packed. The two parts were isolated with a thin layer of sodium alginate (Hillier Dental, Kent, UK). Forty unidirectional glass fiber bundles were light polymerized for 4 minutes in a curing unit

(ESPE Visio Beta Vario, 3M ESPE, Seefeld, Germany). Each fiber bundle was 25 mm long, corresponding to 5 mm inserted into the holes of the basal part and 20 mm projecting above the surface, and acted as embedded length within the silicone elastomer.

The second part of the flask was assembled in place over the basal part, with the fiber bundles projecting through the center of cylindrical molds. Maxillofacial silicone elastomer was weighted according to manufacturer's instructions; 10 g: 1 g (rubber:hardener ratio) using a microbalance. Forty grams of rubber were mixed with 4 g hardener, and this quantity was enough to fabricate 10 specimens (≈ 4.4 g/specimen).

The silicone was mixed manually for 5 minutes followed by mechanical mixing under vacuum for 5 minutes (Multi Vac 4, Degussa, Germany). After completing the mixing, the silicone was poured into the molds of the flask, with the aid of vibration.

The flask contents were heat-polymerized in an oven (Gallenkamp, Leicestershire, UK) according to the manufacturer's instructions (100°C for 1 hour), and left to bench cool for 2 hours. Then the specimens were cautiously removed and dry stored for 24 hours. Four groups of specimens (n = 10) were fabricated. The specimens were aged using an environmental chamber (Heraeus Suntest Chamber CPS, Atlas Material Testing Solutions Gmbh, Linsengericht, Germany) under

Table 1 Groups of the study

Groups					
(n = 10)	Composition	Conditioning			
1	<i>EverStick</i> glass fiber bundles embedded into maxillofacial silicone elastomer	Dry storage for 24 hours			
2		Accelerated daylight aging for 200 hours			
3		Accelerated daylight aging for 400 hours			
4		Accelerated daylight aging for 600 hours			





Figure 2 Pull-out test performed to test the bond strength of glass fiber bundles to silicone elastomer (A), and its graphical representation of forces with displacement (B).

accelerated exposure to artificial daylight for different periods of time (Table 1). Quartz-filtered radiation was generated using a Xenon lamp. The radiation spectrum was similar to that of natural sunlight (irradiance was 760 W/m²). The UV component of the radiation that was directed upward was reflected on the specimens by mirrors presented over the Xenon lamp. A complete weathering cycle lasted for 120 minutes, including 18 minutes of wet weathering ($29^{\circ}C \pm 2^{\circ}C$) by distilled water, followed by 102 minutes of dry weathering ($36^{\circ}C \pm 2^{\circ}C$). Relative humidity was approximately 70%, and air pressure was 700 hPa to 1060 hPa. Specimens were stored for 24 hours at room temperature ($23^{\circ}C$), and then pull-out tests were conducted using a low-load cell (0.5 kN) installed on a universal testing machine (Zwick/Roell Z020, Leominster, UK) (Fig 2A). Maximum pull-out force and work were obtained.²⁷ The maximum pull-out (debonding) force was recorded. The pull-out work (R_p) required to extract the fiber-bundles was calculated according to Equation 1:²⁸

$$R_{p} = \frac{A_{2}}{\pi \ dL_{e}} \tag{1}$$

where A_2 stands for area 2 under pull-out curve (Fig 2B); d for diameter of fiber bundle; and L_e for length of embedment. The area under the curve (A_2) was measured using SigmaPlot software (release 8, SPSS Inc., Chicago, IL).

The fiber bundles were tested using a three-point bending test. A load was applied to the fiber bundles by a steel bar attached to a load cell (20 kN) of the Zwick testing machine, at 2 mm/min

Group $(n = 10)$	Maximum pull- out force (F1) (N)	Force where pull- out arrested (F ₂) (N)	Drop in force $(F_1 - F_2)$ (N)	Pull-out work (R₅) N.mm
1	13.63ª (7.45)	8.52 (2.28)	5.11ª (6.56)	0.94ª (0.32)
2	19.67 ^b (1.37)	4.72 (0.89)	14.95 ^b (1.66)	0.48 ^b (0.30)
3	13.58ª (2.61)	7.26 (1.03)	6.32 ^a (2.33)	0.69 (0.51)
4	10.37ª (2.52)	6.96 (1.16)	3.41 ^a (1.74)	0.77 ^a (0.16)

Table 2 Mean (SD) values of pull-out force and work

Within each column, different superscripts indicate statistically significant differences between paired groups (p < 0.05).

speed. Maximum deflection and bending forces (F) were recorded by the machine, and maximum bending strength (\mathbf{G}) was calculated according to Equation 2:²⁹

$$\mathbf{\overline{0}} = \frac{8\mathrm{FL}}{\pi\mathrm{d}^3} \tag{2}$$

where F is the maximum force applied (N); L is the length of fiber bundle (15 mm); and d is the average diameter of the fiber bundles (\sim 1.5 mm).

Diameter of fiber bundles was obtained by three measurements performed by a digital caliper (Mitutoyo, Tokyo, Japan) at three locations on each bundle (one at the middle, and two at both ends). Pull-out parameters (forces and work) and bending values (maximum bending strength and deflection) were analyzed (p < 0.05) using one-way ANOVA and Bonferroni post hoc tests (release 14, SPSS Inc.).

Results

Pull-out results of tested groups are presented in Table 2. Group 2 had highest pull-out force, which was statistically significant when compared to the remaining groups (p < 0.05). Group 1 had the highest pull-out work, which was statistically significantly higher than Group 2. Group 4 also had a statistically significantly higher pull-out work than Group 2.

The first drop in force after the maximum force was noticeably different among all groups (Fig 3). Specimens tested after aging for 200 hours had the largest drop, statistically significantly different from the remaining groups (p < 0.05).

Maximum bending strength and deflection are presented in Table 3. Group 3 showed the highest bending strength, which was statistically significantly higher than Group 1 (p < 0.05). Also, Group 2 exhibited statistically significantly higher bending strength than Group 1 (p < 0.05) (Fig 4). There were no statistically significant differences in maximum deflection between all groups (p > 0.05).



Figure 3 Graphs representing drop in debonding force (FD) as a result of different aging intervals. Drop in force differed significantly among the aging intervals. The smooth line presents force drop, while the thick line immediately after debonding presents frictional forces during extracting the fiber out of the silicone.

 Table 3
 Mean (SD) values of maximum bending force, bending strength, and deflection

Group (n = 10)	Maximum bending force (F _{max}) N	Maximum bending strength (G _{Fmax}) MPa	Maximum deflection (S _M) mm
1	60.84 (4.63)	917.72ª <i>(69.79)</i>	2.23 (0.45)
2	70.04 <i>(8.18)</i>	1056.44 ^b <i>(123.38)</i>	2.54 (0.34)
3	74.52 <i>(9.49)</i>	1124.06 ^b (143.15)	2.29 <i>(0.2)</i>
4	69.47 <i>(7.07)</i>	1047.84 ^b <i>(106.67)</i>	2.37 (0.31)

For maximum bending strength property, different superscripts indicate statistically significant differences between paired groups (p < 0.05).

Discussion

Extraoral maxillofacial silicone prostheses play a vital role in the rehabilitation of patients with facial disfigurements. Delamination of silicone from the retentive baseplate is one of the common problems that necessitate refabrication of a prosthesis. Prolonged exposure to daylight and to moisture greatly weakens the bond strength between silicone elastomers and acrylic substrates. In fiber-embedded maxillofacial prostheses, the accelerated exposure to artificial daylight aging statistically significantly affected the bending strength of glass fiber bundles and their mechanical integrity to silicone elastomers. Accordingly, we rejected the null hypotheses.

Bond strengths between glass fibers and silicone elastomers

An earlier study indicated that the pull-out bond strength of glass fiber bundles embedded within a heat-cured silicone elastomer were in the range of 13 N to 16 N.²⁶ In this study, specimens were subjected to different aging intervals (200, 400, and 600 hours) to simulate prostheses being in service for a period of 7 to 20 months, as patients wear their prostheses for an average of 8 to 12 hours per day. This timeframe was selected because it is consistent with the literature on average service periods for



Figure 4 Error bars of maximum bending strength, along with statistical significances presented between paired groups.

silicone prostheses, which depend on patients' habits, climate, and environmental conditions.^{20,21,24,30}

A pull-out test was performed to record maximum pull-out forces required to break the interfacial bond between fiber and surrounding silicone matrices. The force was statistically significantly higher than the other groups after 200 hours of aging (p < 0.05). This increase in the interfacial bond strength suggests further polymerization of silicone elastomer by the simulated environmental conditions of the aging chamber; however, further aging for longer intervals degraded bond strength.

Silicone specimens were constructed by pouring silicone in stone flasks, and curing them for 1 hour at 100°C. It is reported that silicones are not completely polymerized in stone molds.^{31,32} Furthermore, the degree of cure of maxillofacial silicones was reduced by the introduction of pigments, contaminants, and by curing in dental stone molds.³¹ Accordingly, silicones after aging for 200 hours were further polymerized. exhibiting higher bond strength than that of unaged specimens; however, aging for longer periods (increase of irradiation time) has been reported to reduce silicones' elasticity and tear strengths and increase their hardness.^{18,25} Such changes caused the silicone specimens to be less elastic when pulling the fibers out of them, so that the bond strength with the glass fibers was weakened and decreased in value. Aging for 600 hours greatly weakened the interfacial strengths with the embedded fibers, suggesting that longer durations of aging adversely affected silicones and altered their properties to a greater extent.^{24,33}

The reduced elasticity of silicone specimens is caused by the increased cross-linking between polymer chains activated by heat produced from the Xenon lamp during aging. The degree of cross-linking between polymer chains of silicone elastomers is one of three main factors that influence the properties of silicones.^{34,35} A silicone elastomer with a very high cross-link density produces an inelastic brittle material, whereas a material with very low cross-link density would produce a very weak material with very low tear and tensile strengths.^{34,35}

Failure in bond strength between fiber bundles and silicone elastomers was noticeable by the force drop after debonding (Fig 3). Aging specimens for 200 hours caused the statistically significant largest drop. This can be explained by enhanced elasticity of silicones after 200 hours.

Pull-out work is the work needed to extract the whole embedded fiber from silicone when the interfacial bond strength is broken. It was greater at the baseline and less after 200 hours of aging; however, pull-out work increased after subsequent aging. A significant difference in pull-out work was evident between aging at 200 hours and at 600 hours (p < 0.05). This might be due to the increased light aging duration (600 hours), as it induces structural modifications in the distribution of the polymer molecular masses, intensifying cross-linking between chains and monomers, and producing denser and harder elastomeric structures.^{36,37}

The lowest mean value of pull-out force (10.37 N) was exhibited when the specimens were aged for 600 hours. Forces expected to influence the bond integrity between fibers and silicones are likely to generate when the patient grips the silicone to dislodge the prosthesis from the magnetic retentive sites or bars, or during cleaning of the prosthesis.³⁸ The forces of attraction between the retentive components of magnets are reported

to be 7.2 N,³⁹ and 3 N to 10 N as in the Magnacap attachment system (Magnacap attachments, Technovent, Leeds, UK), and they range from 5 N to 7 N for retentive bars.⁴⁰ Although retentive attraction forces are lesser than bond strengths of glass fibers to silicone elastomer, greater dislodging forces can disrupt the bond integrity leading to delamination of silicones from the framework. Accordingly, patients should be advised to gently treat their prostheses during daily routine actions of gripping and cleaning the prosthesis. Furthermore, mild detergents should be used, as acidic or alkaline cleaning solution affect silicone properties and its bonding integrity.

Bending strength of FRC framework

Statistics showed that after exposing silicone specimens with the glass fiber bundles embedded in them to artificial daylight, the bending strength of glass fibers statistically significantly improved after 200 and 400 hours of aging, likely due to the prolonged exposure to artificial daylight, as the material is photo-polymerized. Prolonging the polymerization time and increasing the light intensity increased the degree of conversion of the polymer matrix of FRC.⁴¹ Furthermore, the increased cross-linking density increased the number of covalent bonds between polymer backbones and lowered the amount of residual monomers that would plasticize the polymer matrix.⁴²

This study established a baseline of pull-out bond strengths of fiber-embedded maxillofacial silicone prostheses after 600 hours of artificial daylight aging. In the literature, it has been reported that a fiber-embedded maxillofacial prosthesis restoring a large midfacial defect overcame disadvantages associated with traditionally fabricated prostheses, whereby a silicone body is attached to acrylic baseplate that retains the prosthesis over the defect site. The glass framework was lightweight, the margins of the prosthesis were compliant and adaptable to facial changes during facial expressions, and the prosthesis was comfortable and more psychologically acceptable for the patient. Furthermore, FRC framework's high fatigue resistance against bending extended the serviceability of this glass fiberembedded silicone facial prosthesis up to 2 to 3 times that of the patient's former prosthesis with an acrylic resin base, as it was easier to adjust and repair.8

The authors are currently incorporating different protocols to enhance the bond strength integrity between glass fiber and silicone elastomers, including the usage of different types of bond primers.

Conclusions

Within the limitations of this in vitro study, it can be concluded that after 200 hours of exposure to artificial daylight and moisture conditions, bond strength between glass fibers and heat-cured silicones is optimal, and the bending strength of the glass fiber bundles is enhanced.

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