Effect of Glass-Fiber Reinforcement and Water Storage on Fracture Toughness (K_{IC}) of Polymer-Based Provisional Crown and FPD Materials

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Purpose: The effect of glass-fiber reinforcement and water storage on the fracture toughness (K_{IC}) of polymer-based provisional crown and fixed partial denture (FPD) materials was investigated. ***Materials and Methods:*** Five unreinforced single-edged, notched control specimens and five test specimens reinforced with unidirectional E-glass fibers (Stick) were fabricated from three dimethacrylate-based provisional materials and one monomethacrylate-based provisional material. The specimens were stored in water at 37°C for 1, 7, 30, or 60 days. Specimens were loaded in three-point bending at a cross-head speed of 0.1 mm/s. Mode I plane-strain K_{IC} was calculated using the maximum load, and results of the two groups were compared. The water storage effect on K_{IC} with time was also evaluated. ***Results:*** The K_{IC} of provisional materials reinforced with glass fibers (range 7.5 to 13.8 $MNm^{-1.5}$) was significantly higher than that of unreinforced materials (range 1.3 to 3.1 $MNm^{-1.5}$), by a factor of 4.4 to 5.5. A small, gradual decrease of K_{IC} in reinforced specimens occurred with aqueous storage, but it was not statistically significant. ***Conclusion:*** The K_{IC} of polymer-based provisional crown and FPD materials was significantly increased when they were reinforced with unidirectional E-glass fibers. Water storage for up to 2 months still left the reinforced materials with K_{IC} values in excess of 7 $MNm^{-1.5}$. Hence, their performance was satisfactory. *Int J Prosthodont 2004;17:318–322.*

Polymer-based provisional crown and fixed partial denture (FPD) materials should have adequate strength to withstand functional loads. One method of enhancing the fracture resistance is the addition of reinforcing materials such as metal wire and glass fiber. Metal wire does not bond chemically with the materials, resulting in stress concentration around the metal wire. The material around the metal wire shrinks away from it, leaving the material with voids that weaken the structure by creating new points of stress concentration. Although several methods, such as silanization of the metal,[1,2] sandblasting of metal wire with Al_2O_3,[3] and metal adhesive resin,[4] have been proposed to improve the adhesion between such materials and metal reinforcements, these enhancements are not sufficient.[5]

Glass fibers are biocompatible, not prone to corrosion, and easy to repair.[6] These fibers also have an excellent esthetic appearance. Reinforcement with glass fibers enhances the mechanical strength characteristics of polymers, including their transverse strength, ultimate tensile strength, and impact strength.[7] This type of reinforcement is superior to metal-wire reinforcement in terms of esthetics and bonding to the resin matrix. These fibers have been used in other dental applications, such as inlay FPDs[8,9] and posts.[10]

Many studies on the fracture strength of fiber-reinforced provisional prostheses have been carried out. Samadzadeh et al[11] studied the effect of a plasma-treated woven polyethylene fiber on the fracture strength of three-unit provisional posterior prostheses made of polymer-based materials. They measured the central compressive load to determine the fracture load of the prostheses and

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Table 1 Materials Investigated

Material	Lot No.	Characteristics
Protemp 3 Garant, 3M/ESPE	646911001	Dimethacrylate-based material
Quicktemp, Schottlander	00040191	Dimethacrylate-based material
Fast Set Temphase, Kerr	003E92	Dimethacrylate-based material
Trim, Bosworth	921900	Monomethacrylate-based material
Stick, Stick Tech	—	Unidirectional E-glass fiber
Stick Resin, Stick Tech	106640	Light-curing adhesive

found that reinforced prostheses made of dimethacrylate-based materials exhibited significantly higher fracture loads than the unreinforced materials.[11] Vallittu[12] investigated the effect of unidirectional glass-fiber reinforcements and woven glass-fiber reinforcements on the fracture resistance of three-unit provisional prostheses. Both types of glass-fiber reinforcements considerably increased the fracture resistance of the provisional prostheses, even though the reinforcements were positioned on the least favorable side of the prostheses.[12]

The material property that represents resistance to fracture is fracture toughness (K_{IC}), an important material characterization parameter required for the prediction of the mechanical performance of structural materials. K_{IC} is considered to be a better measure of fracture resistance assessment than other strength parameters.[13,14] Although previous investigations[11,12] of the fiber reinforcement of three-unit provisional prostheses show enhancement in fracture strength, the effect of glass-fiber reinforcement on K_{IC} of polymer-based provisional materials has not been reported.

The objectives of the present study were to evaluate *(1)* the effect of unidirectional E-glass fiber reinforcement on K_{IC} of polymer-based provisional crown and FPD materials, and *(2)* the effect of water storage over time on K_{IC} of the materials. The null hypotheses were that there would be no difference in K_{IC} between unreinforced and reinforced polymer-based provisional materials with unidirectional E-glass fibers, and that there would be no difference in their K_{IC} values after water storage.

Materials and Methods

The materials selected in this study are listed in Table 1. Four polymer-based provisional crown and FPD materials and one unidirectional E-glass fiber (Stick) were selected as reinforcements. Single-edged, notched specimens (n = 5) for each subgroup, conforming to British Standard 5447,[15] were prepared in a polytetrafluoroethylene (PTFE)-lined brass mold that could be split so that no force was required to remove the set specimen from the mold. The overall external dimensions were 3 mm × 6 mm × 25 mm, and a sharp, 3-mm-long notch was made to half the beam height (Fig 1). A sharp central notch of specific length *(a)* was produced by inserting a straight-edged scalpel blade into an accurately fabricated slot at mid-height in the plastic mold; the slot extended down half the height to give $a/W = 0.5$. The blade had a straight cutting edge honed on both sides, with a blade edge radius of less than 0.3 µm. The crack plane was perpendicular to the specimen length.

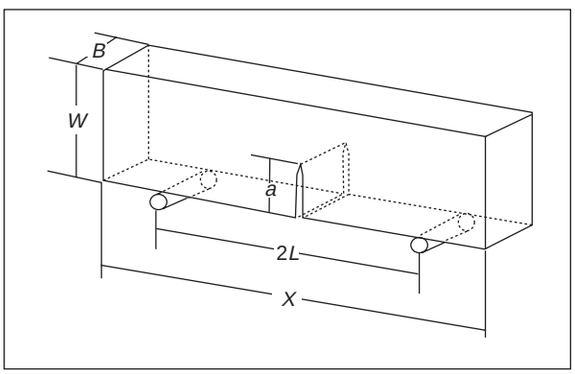

Fig 1 Single-edged, notched specimen used in the study; B = 3 mm; W = 6 mm; X = 25 mm; L = 10 mm; a = 3 mm.

Unreinforced control groups were fabricated according to the following procedure. The materials were mixed according to the manufacturers' instructions and immediately transferred to the mold. The open surface was covered with a plastic matrix strip and a thick glass plate. Specimens were left to polymerize for 30 minutes at 23 ± 1°C. After the material was set, the mold was disassembled and the specimen was removed. The blade was removed from the specimen with great care, and then all flash was eliminated. The specimen was inspected for observable voids and then left to polymerize further at 23 ± 1°C for 1 hour.

For reinforced test groups, all procedures were the same as described above, except for the insertion of one layer of Stick fiber into the material. Stick fiber was removed from the package and cut with sharp-edged scissors into 20-mm-long pieces. To wet the fibers with Stick Resin, the following procedures were followed. The fiber was put into a transparent plastic bag, into which two drops of Stick Resin were placed. The fiber was bent back and forth several

Table 2 Mean (Standard Deviation) K_{IC} Values of Materials Investigated (in $MNm^{-1.5}$)

Material	Water storage (d)			
	1	7	30	60
Protemp 3 Garant				
Unreinforced	3.1 (0.3)	3.1 (0.2)	2.7 (0.1)	2.5 (0.4)
Reinforced	12.8 (1.5)	15.7 (3.0)	12.4 (4.2)	8.9 (3.3)
Quicktemp				
Unreinforced	2.5 (0.1)	2.6 (0.2)	2.6 (0.1)	2.3 (0.2)
Reinforced	13.8 (3.4)	13.8 (2.0)	13.3 (3.8)	13.5 (2.3)
Fast Set Temphase				
Unreinforced	1.9 (0.1)	2.0 (0.2)	2.0 (0.2)	1.8 (0.1)
Reinforced	8.6 (2.0)	11.1 (2.7)	9.4 (1.0)	7.9 (1.8)
Trim				
Unreinforced	1.4 (0.1)	1.4 (0.2)	1.6 (0.1)	1.3 (0.3)
Reinforced	7.5 (1.1)	7.7 (0.7)	7.6 (1.5)	7.5 (0.7)

times for wetting, and it was kept away from all sources of light for 10 minutes. The PTFE-lined brass mold was two thirds full of the resin mixture. The fiber strands impregnated with Stick Resin were removed from the plastic bag and placed, parallel to the long axis of the specimen, into the unset material in the mold. The final resin mixture was added to the fibers to fill the remainder of the mold. A plastic matrix strip and thick glass plate were placed over the mold. For polymerization of the unpolymerized Stick Resin, three different areas of the upper surface of the specimen (center, left, and right) were irradiated with a light-curing unit (Curing Light XL 3000, 3M/ESPE) for 40 seconds each. Specimens were left to polymerize for 30 minutes at 23 ± 1°C. The mold was disassembled and the specimen was removed from the mold. The blade was removed from the specimen with great care, and all flash was eliminated. For further polymerization of the Stick Resin, the light was also applied to the other longitudinal surface of the specimen for the same period. This curing procedure was then extended to additional surfaces. After inspection for observable voids, specimens were allowed to polymerize further at 23 ± 1°C for 1 hour. Each group was also subdivided into four according to the period of storage. Each specimen was placed in a small bottle with distilled water and stored in a cabinet at 37°C. Each subgroup was stored for different lengths of time (1, 7, 30, and 60 days) before testing.

Specimens were tested in three-point bending with a Howden Universal Testing Machine (RDP Howden) at 23 ± 1°C. The specimens were placed on the testing machine, using supports that consisted of two parallel 2-mm-diameter stainless steel rods with a fixed span width of 20 mm. A mechanical load was applied on the center of each specimen at 90 degrees to the specimen axis through a 2-mm-diameter stainless steel rod. By movement of the cross-head at 0.1 mm/s, the load was increased until specimen fracture. Peak load to fracture was recorded, and specimen deflection was recorded as load/deflection curves. After each experiment, the initial notch length–to–specimen height ratio was rechecked with an optical microscope. Mode I plane-strain K_{IC} was calculated using the following equation[16]:

$$(3PL/BW^{3/2})Y$$

where P = peak load at fracture; L = length; B = width; W = height; and Y = calibration functions for given geometry, $(1.93[a/W]^{1/2} - 3.07[a/W]^{3/2} + 14.53[a/W]^{5/2} - 25.11[a/W]^{7/2} + 25.80[a/W]^{9/2})$.

The mean values and standard deviations of the results were computed. The independent sample t test was used to compare K_{IC} values between reinforced and unreinforced specimens of each material. One-way analysis of variance (ANOVA) and multiple comparisons Scheffé test at the .050 significance level were used to compare K_{IC} values of the materials at each storage time and evaluate the effect of different storage times in water. Linear regression analysis was also applied for each material. SPSS software (version 10.1, SPSS) was used for these statistical analyses.

Results

In unreinforced control specimens, the mean K_{IC} values obtained from the dimethacrylate-based materials (1.8 to 3.1 $MNm^{-1.5}$) were significantly higher ($P < .001$) than those of the monomethacrylate-based material (1.3 to 1.6 $MNm^{-1.5}$) (Table 2). Linear regression of all materials except Trim exhibited negative values, which means that K_{IC} values tended to decrease with time when the materials were stored in water. However, ANOVA showed that K_{IC} values of the materials were not significantly decreased ($P > .050$) after 2 months of water storage compared with the values at 1 day of water storage (except for Protemp 3 Garant; $P = .043$).

When the specimens were reinforced with Stick fiber, the mean K_{IC} values of the dimethacrylate-based materials ranged from 7.9 to 13.8 $MNm^{-1.5}$, whereas the monomethacrylate-based material showed lower values

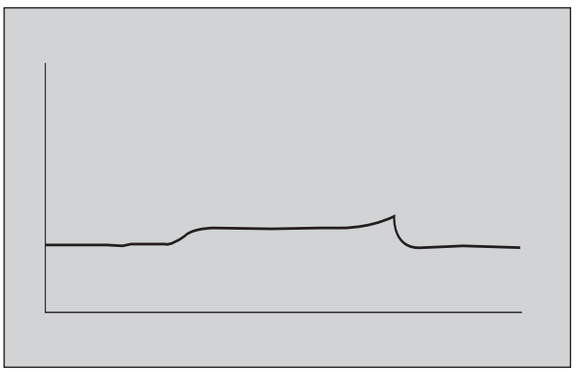

Fig 2a Load/deflection curve: unreinforced specimen.

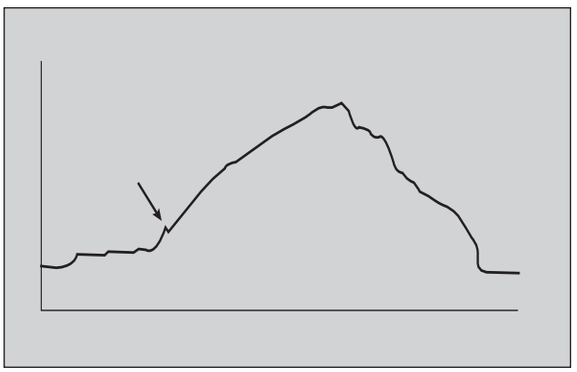

Fig 2b Load/deflection curve: specimen reinforced with Stick fibers; *arrow* = crack interruption.

(7.5 to 7.7 $MNm^{-1.5}$) (Table 2). The K_{IC} values of the reinforced materials significantly increased ($P < .001$) compared with the unreinforced specimens. The highest value for the reinforced materials was shown at 1 week of water storage. As with the unreinforced specimens, linear regression showed negative values. All materials except Trim showed a slight decrease in K_{IC} after water storage for 2 months. The K_{IC} decrease was highest in Protemp 3 Garant but was not significant ($P > .050$).

The load/deflection curves showed that the unreinforced specimens were associated with smaller and smooth curves (Fig 2a), whereas the reinforced specimens exhibited bigger, jagged curves (Fig 2b). Fracture processes between the reinforced and unreinforced specimens were different. In the reinforced specimens, load increased until the first peak, then dropped (Fig 2b) and increased again. The peak load was higher in the reinforced specimens than in the unreinforced specimens.

Discussion

K_{IC} depends on the type of polymer and reinforcement.[17] In the present study, K_{IC} varied depending on the type of matrix polymer. K_{IC} of the monomethacrylate-based material was lower than that of the dimethacrylate-based materials before and after reinforcement. The dimethacrylate-based materials have a three-dimensional network structure, resulting in higher mechanical strength than the monomethacrylate-based material. The K_{IC} values for the unreinforced materials were higher than those previously reported for provisional materials, which may be explained by differences in test methods.[17–19]

All test materials showed a statistically significant increase in K_{IC} after reinforcement with unidirectional E-glass fibers, regardless of the material's chemical composition. Thus, the null hypothesis that there would be no difference in K_{IC} between unreinforced materials and reinforced materials was rejected. K_{IC} increased by a factor of 4.4 to 5.5 compared to the control group. This is in accordance with a previous study on the fracture resistance of polyethyl methacrylate and polybutyl methacrylate provisional materials reinforced with fibers.[20]

The K_{IC} value defines the critical stress intensity level at which catastrophic failure occurs. When the crack propagation from the crack tip was interrupted at the first apex, the load dropped slightly (Fig 2b). As the fibers resisted the fracture, the load increased. As the load increased, the specimens appeared to deform elastically. The crack propagated from the crack tip through the specimen in the tension side during flexure, but did not propagate through the compression side because the fibers did not stretch or fracture. Fibers in the specimen have been observed to deflect crack propagation, and some fibers were stretched and fractured individually. Some fibers also bridged the crack and resisted opening and propagating, thus exerting a closure force on the crack. The modes of failure are believed to include transverse splitting, compressive failure caused by fiber kinking, interfacial shear failure, and brittle tensile failure.[21] Fibers reduced the net stress intensity at the crack tip, significantly enhancing the toughness of the specimen.[22] The fracture process was intermittent and irregular. The trace of roughness in the last half of the load/deflection curve showed that no catastrophic failure occurred in the reinforced specimens. Fracture of the fibers caused complete failure of the test specimen. The fibers thus controlled the toughness of the specimen.

The oral cavity contains saliva that affects the mechanical properties of biomaterials; the mechanical properties of fiber-reinforced materials can be influenced by water.[23,24] Vallittu et al[25] studied the effect of long-term water storage on the flexural properties of woven E-glass fiber-reinforced denture base resin and concluded that the ultimate transverse strength decreased by 14% after 48 weeks of water storage. Water absorption in the resin is possible because the water molecule is smaller than the

interchain distance in the resin.[26] Water diffuses through the resin and leaches the surface of the fibers, resulting in their deterioration.[25] Any poorly impregnated regions in the resin will increase water absorption and weaken the bond between fiber and resin matrix, reducing the mechanical strength.[25–27]

Water absorption is also dependent on the amount of filler particles, volume percent of fibers, monomeric composition of the resin, voids in the resin, and degree of silanization of the fiber surface.[26,27] The data for reinforced materials in the present study suggested a maximum K_{IC} at 1 week. This may be explained by the probable competing effects of network post-cure of the material and gradual water degradation. All samples showed small negative linear regression. Such gradients can be explained by changes in the hardness of the material caused by water sorption, resulting in softening of the material that, in turn, decreased the toughness. These changes were most apparent in reinforced Protemp 3 Garant. The K_{IC} values of reinforced materials stored for up to 2 months were still in excess of 7 $MNm^{-1.5}$. However, more studies are required to evaluate the effect of longer water storage on K_{IC}.

Conclusion

Polymer-based provisional crown and FPD materials exhibited a significant increase in K_{IC}, by a factor of 4.4 to 5.5, when reinforced with unidirectional E-glass fibers. With cross-linked matrices, maximum K_{IC} was seen at 1 week and decreased slightly thereafter. Nevertheless, storage up to 2 months still left the reinforced materials with K_{IC} values in excess of 7 $MNm^{-1.5}$, a satisfactory performance.

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