Flexural Strengths of Denture Base Resin Repaired with Autopolymerizing Resin and Reinforcements After Thermocycle Stressing

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<u>Purpose</u>: Fracture of an acrylic denture base is a common problem in prosthodontic practice. Although various reinforcement methods have been used, when a fractured denture base is repaired with autopolymerizing resin recurrent fractures frequently occur at the repairing interface or adjacent areas. The purpose of this study was to evaluate the maximum flexural load of denture base resin repaired with autopolymerizing resin and several reinforcement systems after thermocycle stressing.

<u>Materials and Methods</u>: Rectangular $(10 \times 70 \times 3 \text{ mm})$ flexural specimens were fabricated by repairing a pair of heat-cured denture base resin specimens using autopolymerizing resin and a series of reinforcement materials. The materials included 4 metal wires and a woven glass fiber. Each reinforcement was embedded in the center of the specimens. Flexural specimens repaired without reinforcement were prepared as controls. Specimens were subjected to 50,000 thermocycles $(4 \sim 60^{\circ} \text{C}, 1\text{-minute dwell time})$. A 3-point flexural test was carried out by loading the center of the repaired site at 5 mm/minute crosshead speed with 50 mm span jig supports. The load necessary to cause fracture was recorded for each specimen. All data were statistically analyzed using ANOVA and the Bonferroni/Dunn test ($\alpha < 0.05$).

<u>Results</u>: The average load to fracture of specimens repaired with nonreinforced autopolymerizing resin was 68.4 N after 50,000 thermocycles. Specimens reinforced with 1.2 mm diameter stainless steel wire exhibited the highest value (89.8 N). The value for specimens reinforced with 1.2 mm diameter Co–Cr–Ni wire was 86.6 N. These fracture loads were significantly higher than those for specimens without reinforcement (p < 0.05). Low elasticity reinforcement, such as pure titanium wires, woven metal wire, and woven glass fiber were not effective in increasing the load to fracture values of flexural specimens.

<u>Conclusions</u>: Specimens reinforced with 1.2 mm diameter stainless steel wires or Co–Cr–Ni wires resulted in significantly higher loads to fracture as compared to specimens without reinforcement. The use of pure titanium wire, woven metal wire, and woven glass fiber did not improve the fracture loads.

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Many studies have investigated attempts to improve the flexural strength of repaired denture base resin by modifying joint design,³⁻⁵ using pretreatments for the repair surface,^{2,6} and by selection of the repair materials.^{1,5,6} The effects of metal reinforcement⁷⁻¹⁰ and fiber reinforcement¹¹⁻¹⁷ on flexural strength of the denture base have been studied. Even for those reinforcements applied at the time of denture fabrication, denture base fracture in a relatively short period has been experienced clinically. Although some studies^{15,18-21} of the flexural strengths of repaired acrylic base have been reported, the effect of the reinforcement has not been fully clarified. Moreover, although the repaired denture base is usually subjected to severe conditions, including thermal and cyclic flexural stresses during mastication, flexural strengths after fatigue have not been studied.

Therefore, the purpose of this study was to evaluate acrylic denture base maximum flexural load after thermocycle stressing when repaired with autopolymerizing resin utilizing several reinforcement systems.

Materials and Methods

Materials used in this study are listed in Table 1. They included a heat-polymerizing denture base resin and an autopolymerizing acrylic resin as a repairing material. Aluminum oxide particles (50 μ m average diameter) were used for air abrasion to the denture base resin surface. Reinforcement materials included 4 types of metal wire and a woven glass fiber. The metal reinforcements used included experimental stainless steel wires, a Co–Cr–Ni wire, a pure titanium wire, and a

 Table 1. Materials Used

woven metal wire. A woven glass fiber was used as a
nonmetallic reinforcement. Adhesive primer was used
to achieve bonding between the metal reinforcements
and the repairing acrylic.

Specimen Preparation

The denture base resin was prepared by mixing PMMA powder and MMA liquid at a ratio of 100 g to 43 ml. The resin was packed into the stainless steel mold ($10.2 \times 70.2 \times 3.1$ mm) and polymerized in water at 70°C for 60 minutes, followed by 45 minutes at 100°C. The resin specimens were finished to $10 \times 70 \times 3.0$ mm with 600-grit silicon carbide paper under water irrigation and stored in 37°C distilled water until they were watersaturated. To make a place for the reinforcement material, a groove (3 mm wide, 2.1 mm deep, and 36 mm long with circular ends) was prepared on the bottom surface of each denture base specimen using a steel bur (#705HP, Maillefer, Switzerland) under copious water irrigation. Specimens were cut to produce 2 segments separated by a 3 mm space, using a high-speed diamond disc under water irrigation. The denture base specimens were pretreated by air abrasion with 50 μ m alumina oxide particles under a 0.5 MPa pressure. A pair of fragmented specimens was placed in a Teflon mold to maintain a 3 mm space. Each reinforcement material was placed in the center of the groove. The metal wires were air-abraded with 50 μ m alumina oxide particles under 0.5 MPa pressure, followed by treatment with the adhesive primer, then placed in the center of the groove. The glass fiber was immersed in the monomer of autopolymerizing resin for 10 minutes before

Material	Product Name	Manufacturer	Code
Heat-polymerizing denture base resin	Acron	GC International Corp. (Tokyo, Japan)	
Autopolymerizing resin	Metafast	Sun Medical Corp. (Moriyama, Japan)	
Adhesive primer	Cesead II opaque primer	Kuraray Co., Ltd. (Osaka, Japan)	
Stainless steel wire ($\emptyset = 1.2 \text{ mm}$)	_	Nissin Corp. (Kyoto, Japan)	St1.2
$(\phi = 1.4 \text{ mm})$	-	Nissin	St1.4
$(\phi = 1.6 \text{ mm})$	_	Nissin	St1.6
$\dot{C}o$ - Cr - Ni wire ($\phi = 1.2$ mm)	Sancobalt clasp wire	Sankin (Tokyo, Japan)	CoCr
Pure titanium wire ($\emptyset = 1.2 \text{ mm}$)	Rematitan-Draht	Dentaurum JP Winkelstoeter KG (Ispringen, Germany)	Ti
Woven metal wire	Strengthener (medium)	Sankin	WM
Woven glass fiber	Fiber-Splint (#5978)	Polydentia (Cary, NC)	WG

-: experimental material.



Figure 1. Schematic drawing of bending specimen.

being placed into the groove. Autopolymerizing resin was filled into the space and the groove by brush-on procedure, and cured for 1 hour at room temperature. Resin excess was ground flat with #600 silicon carbide paper under water irrigation and finished to a $10 \times 70 \times 3$ mm rectangular flexural specimen (Fig 1). Specimens with autopolymerizing resin and stainless steel wires of 1.2, 1.4, and 1.6 mm diameters were designated as groups St1.2, St1.4, and St1.6, respectively. Depths of the grooves for the groups St1.4 and St1.6 were adjusted to 2.2 and 2.3 mm, respectively, to allow placement of the wires in the center of the specimen. For groups CoCr, Ti, WM, and WG, Co-Cr-Ni wire, pure titanium wire, woven metal wire, and woven glass fiber were used, respectively. Group A specimens (control) received no groove preparations and were repaired by filling the interspecimen space with autopolymerizing resin. Additionally, heat-polymerizing resin block (group HB) and autopolymerizing resin block (group AB) were also fabricated into the same size (10×70) \times 3 mm) and used as comparative specimens.

Three-Point Flexural Test

Ten specimens were fabricated for each of the 10 groups, and stored in 37°C distilled water for 24 hours. Three-point flexural tests were performed for 5 of 10 specimens in each group to deter-

mine the load to fracture 24 hours after fabrication. The loads of the remaining 5 specimens were determined after subjecting them to 50,000 thermocycles. Thermocycling was carried out by soaking the specimens alternatively into 4°C and 60°C water baths with a 1-minute dwell time at each temperature. The flexural test was carried out using a universal testing machine (AGS-5kNG, Shimadzu Co., Kyoto, Japan) at a 5.0 mm/minute crosshead speed. The specimens were supported on the jigs with a 50 mm span. Load was applied to the center of the repaired site. Linear contact was obtained between the specimen and both supporting and loading levers (Fig 1). Stress-strain curves were recorded on a chart throughout the flexural tests. The maximum flexural load during fracture was determined from the chart and recorded as a fracture load in N (Newton). Specimen size was strictly maintained with 0.05 mm accuracy. Deflections during fracture were calculated with the following equation:

deflection = a * l/b

where a is the crosshead speed (5 mm/minute), b is the chart speed (100 mm/minute), and l is the actual traveling distance of a loading lever (mm), as determined from the chart.

The loads to fracture and deflections during fracture of each group were analyzed with 2-way analysis of variance (ANOVA) using the existence



Figure 2. Loads required to cause fracture of each flexural specimen. The same letters on columns of the same thermo-cycle number indicate no significant difference (p > 0.05). Columns connected with horizontal bars are not significantly different (p > 0.05).

of thermocycling and reinforcement groups as factors. To analyze the difference between each group, multiple comparisons were carried out by Bonferroni/Dunn tests at a 95% confidence level. The data with and without thermocycling were compared for each reinforcement, and those among each reinforcement were compared with and without thermocycling.

Evaluation of Flexural Property of Metal-Reinforcement Materials

For each metal-reinforcement material, 30 mm wire specimens were subjected to flexural loading until reaching breaking stress. Flexural load was applied to the center of a series of metal reinforcements at a crosshead speed of 5 mm/minute with 25 mm jig supports. Stress-strain curves were recorded with 100 mm/minute chart speed during the tests.

Results

The results of 2-way ANOVA demonstrated that there were significant differences in both fracture loads and deflections between with thermocycling and without thermocycling (p < 0.001) and among the reinforcement modes (p < 0.001). In addition, there was a significant interaction between the thermocycling and the reinforce mode (p = 0.0017 for fracture loads and p < 0.0001 for deflections).

The loads and deflections of the specimens with and without thermocycling are presented in Figure 2 and Table 2. Typical fracture patterns are presented in Figure 3.

Table 2. Deflection at Maximum Flexural Load

	Specimen									
Thermocycles	A	St1.2	CoCr	Ti	WM^*	WG^*	St1.4*	St1.6*	HB^*	AB
0 50,000	$\begin{array}{c} 4.15^{\rm a} \\ (0.29) \\ 2.92^{\rm d} \\ (0.18) \end{array}$	3.90^{a} (0.21) 3.42^{c} (0.34)	$\begin{array}{c} 4.25^{\rm a} \\ (0.26) \\ 3.43^{\rm c} \\ (0.26) \end{array}$	$\begin{array}{c} 4.17^{\rm a} \\ (0.35) \\ 2.71^{\rm d} \\ (0.19) \end{array}$	3.10^{b} (0.25) 2.84^{d} (0.23)	3.06^{b} (0.18) 2.86^{d} (0.23)	3.15^{b} (0.46) 2.84^{d} (0.21)	2.66° (0.29) 2.45 (0.22)	6.78 (0.26) 6.80 (0.21)	8.99 (0.34) 5.19 (0.43)

*Not significantly different with and without thermocycling (p > 0.05).

Mean values with the same superscript letters on the same thermocycle number indicate no significant difference (p > 0.05). Deflection are given in millimeters; the values in parentheses are standard deviations.



Figure 3. Fracture mode after flexural test. Note that fracture occurred at the center on (A) but at the ends of the wire on (B) and (C).

The values of the load for groups A, St1.2, CoCr, Ti, WM, and WG after thermocycling were significantly lower (p < 0.05) than for those without thermocycling. The values of groups St1.2 (89.8 N) and CoCr (86.6 N) were significantly higher (p < 0.05) than group A values (68.4 N). Deflection values after thermocycling for both St1.2 and CoCr groups were not significantly different than those for group A (p > 0.05). For groups Ti, WM, and WG, the load and deflection after thermocycling were not significantly different from those of group A (p > 0.05).

Fractures occurred between heat-polymerizing resin and the autopolymerizing resin interface, and the crack propagated into autopolymerizing resin for groups A, St1.2, CoCr, Ti, WM, and WG. Although the specimens were not broken into pieces, permanent transformations were observed, except for in group A (Fig 3A).

For groups St1.4 and St1.6, the loads and deflection between specimens with and those without thermocycling were not significantly different (p > 0.05). There were no significant differences in the loads among groups St1.2, St1.4, and St1.6 (p > 0.05); however, deflections of groups St1.4 and St1.6 were significantly lower than for of group St1.2 (p < 0.05).

Fracture of group St1.2 specimens after thermocycling occurred at the heat-polymerizing resin and autopolymerizing resin interface, and the crack propagated into the autopolymerizing resin (Fig 3A). Two (40%) of 5 St1.4 specimens and 4 (80%) of 5 St1.6 specimens fractured at the end of reinforcement after thermocycling, and the specimens separated into pieces (Fig 3B and 3C).



Figure 4. Typical stress-strain curve for each metal reinforcement.

The fracture load recorded for group HB after thermocycling was 115.8 N, which was not significantly different from the fracture load for the nonthermocycled group (p > 0.05). The fracture load of group AB after thermocycling was 90.0 N and was significantly lower than that of the nonthermocycled group (p < 0.05). The fracture load of group AB (90.0 N) after thermocycling was not significantly different compared to groups St1.2 (89.8 N) and CoCr (86.6 N) after thermocycling.

Stress-Strain Curve of Reinforcements

Stress–strain curves for St1.2, CoCr, Ti, WM, St1.4, and St1.6 specimens under flexural loads are presented in Figure 4. St1.2 and CoCr showed similar elasticity under flexural loads. The elasticity of Ti and WM was lower than that of St1.2 or CoCr, but St1.4 and St1.6 showed higher fracture loads.

Discussion

The fracture load was described in N (Newton), but not calculated into MPa in the current study because it was impossible to calculate with a conventional mathematical formula, due to the complexity of the reinforced specimens consisting of acrylic resin and reinforcement material.

Within the reinforcement methods used in this study, fracture loads of groups St1.2 and CoCr were significantly higher than that of group A, but lower than group HB before thermocycling. Polyzois et al^{18,21} have reported that a reinforcement using a round metal wire showed a significant effect when it was compared with intact denture base strips. Results of the current study disagree with these reports, as their results were obtained using a longer metal wire for the reinforcement compared to the length used in this study. Groups St1.2 and CoCr showed significantly higher loads and greater deflections than those of group A after thermocycling. These 2 reinforcements showed similar elasticity. Groups WM and WG demonstrated lower fracture loads even before thermocycling. Nagai et al²⁰ reported that a woven metal wire and a woven glass fiber had significant reinforcing effects compared to nonreinforced specimens, and that woven glass fiber had a significant reinforcing effect compared to the intact denture base strips. The results of the current study disagree with these findings as well. The reason for the disagreement is probably due to different placement manner of reinforcement. Nagai et al placed the reinforcement at the tension side of the specimen, while it was placed at the middle of the specimen in the present study. Loads and deflections of groups Ti, WM, and WG had no significant difference compared to group A after thermocycling. Therefore, St1.2 and CoCr are considered to be adequate materials for denture base reinforcement. The fracture loads of groups St1.2 (89.8 N) and CoCr (86.6 N) after thermocycling were not significantly different from the fracture loads of group AB (90.0 N). They were 78% and 75% of the fracture load of group HB (115.8 N). Compared to those materials, Ti, WM, and WG did not achieve effective reinforcement due to their lower rigidity.

Fracture load increase after thermocycling was anticipated for group AB, because of the further polymerization occurring in the 60°C water bath during thermocycling, but this did not occur. This result can probably be attributed to the porous structure associated with air-bubble involvement during packing. This phenomenon can be explained by the results of group A. The fracture load decrease of group A after thermocycling may be caused by the water absorption and thermal stress, in addition to the presence of a porous structure. These results agree with earlier reports²²⁻²⁴ claiming that the decrease of flexural strengths after repair is caused by mere immersion in water. The fracture load of group A after thermocycling (68.4 N) was 59% of group HB (115.8 N) and 76% of group AB (90.0 N).

The fracture loads of groups St1.2 (89.8 N) and CoCr (86.6 N) after thermocycling were significantly higher than that of group A (68.4 N); however, they were equivalent to that of group AB, and were 78% and 75% of the fracture load of group HB (115.8 N). This suggests the need for improvement in the mechanical properties of autopolymerizing acrylic resin.

When comparing the results of fracture loads among different thicknesses of stainless steel wires, there were no significant differences between the groups; however, deflections using thicker wires were significantly smaller than that of the thinnest wire. The observation that there is a high possibility of fractures at the wire's end, but not at the loading area, suggests that an unfavorable stress concentration occurs because of the discrepancy in flexibility between the metal wire and the repairing resin. Stress-strain curves demonstrated that St1.6 showed the highest rigidity among the metal reinforcements tested. This stress concentration will be aggravated by the loss of some of the thickness of the repairing resin when a thicker wire is used.

This *in vitro* study evaluated the fracture loads of repaired acrylic denture base by 3-point flexural tests after thermocycle stressing. This study design has limitations for simulating the clinical situation, as the specimen tested was different from actual denture configuration. This study also failed to simulate repetitive mechanical stressing during mastication, which is inevitable with repaired dentures. Further investigations under more closely simulated clinical conditions are necessary.

Conclusion

The maximum load to fracture for heatpolymerizing resin repaired with autopolymerizing resin and a series of reinforcements after 50,000 thermocycles was evaluated. The following conclusions were drawn.

1. The value of the fracture load for heatpolymerizing resin blocks was constant regardless of whether thermocycling occurred.

- The fracture load of specimens repaired with autopolymerizing resin without reinforcement was 59% of the heat-polymerizing resin block and 76% of the autopolymerizing resin block.
- 3. Specimens reinforced with a 1.2 mm diameter stainless steel wire or Co–Cr–Ni wire provided significantly higher maximum flexural loads compared to specimens without reinforcements. These values were equivalent to that of the autopolymerizing resin block.
- 4. Titanium wire, woven metal wire, and woven glass fiber were not effective materials for reinforcing the denture base repair.
- 5. It is possible to create another fracture at the end of the reinforcement when highly rigid metal wire is used.

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