

Effect of loading weight on bond durability of composite-type resin cement under cyclic impact test (part 2). Loading with light weight of 100–120 g

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The bond durability of composite-type resin cement was evaluated by means of cyclic impact tests using three different loads. In terms of experimental setup, a casting alloy, 12%Au-Pd-Ag, was used as the adherend and bonded to a cast block using a composite-type cement (Bistite II). A shear load — using plungers of three different weights at 100, 110, and 120 g — was dropped from a 3-mm height onto a small piece of the casting alloy until debonding. The cycle numbers that caused debonding were $1756 \pm 680 \times 10^4$ times for 100 g, $1403 \pm 515 \times 10^4$ times for 110 g, and $420 \pm 200 \times 10^4$ times for 120 g, respectively. Therefore, the group loaded with 120 g showed a significantly lower value as compared to the other two groups. On the fracture mode of the cement, it was a bulk fracture regardless of the loading weight employed in this study — the same result obtained in a previous study where heavier weights were employed.

Key words: Cyclic impact load, Adhesive resin cement, Bond durability

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INTRODUCTION

The cyclic impact loading test, one of the accelerated laboratory tests, is often employed to investigate the mechanical properties of restorative materials^{1–6}. Using this test method, a previous study⁶ revealed that at a loading weight of 130 g, the bond durability of a MMA-type adhesive resin cement was 6–7 times higher than that of a composite-type cement. It was thus thought that the loading weight of 130 g could be a bit too heavy for the composite-type resin cement. However, the effect of loading weights less than 130 g on bond durability of composite-type resin cements remains to be clarified.

Therefore, the aim of this study was to investigate the effects of loading weights at 100, 110, and 120 g — applied by means of a plunger — on the bond durability of a composite-type resin cement. Besides, another aim of this study was to confirm whether the fracture mode of the resin cement would differ from that loaded with more than 130g, which was bulk fracture as found in the previous study⁶.

MATERIALS AND METHODS

Preparing the adherends

A casting alloy (Castwell M.C., Lot No. 110191, GC Corp., Tokyo, Japan), $9.6 \times 6.3 \times 1.6$ mm³ and 1 g in weight as delivered by the manufacturer, was used as a small-piece adherend. The other adherend was a block fabricated by casting. Its dimensions were approximately $25 \times 10 \times 5$ mm³, and its 25×10 mm²

surface was finished flat with a #320 silicon carbide paper. The cast block was mounted with a self-curing resin in a direction normal to the bottom surface of the plastic ring, whereby the latter facilitated the fixing of the bonded assembly on the load test machine¹.

Preparing the bonded assembly

The bonded assembly is shown in the lower portion of Fig. 1. A sandblaster (MicroBlaster, Comco Inc., CA, USA) with 0.05-mm Al₂O₃ at a pressure of 0.35 MPa was used for the surface treatment of both adherends, to the effect that frosted surfaces were obtained.

To define the bonding area, an adhesive tape of 0.2 mm thickness with a hole of 4 mm diameter was pasted on the treated surface of the cast block. A composite-type resin cement (Bistite II, Lot No. 37C, 73R, Tokuyama Dental Corp., Tokuyama City, Japan) was used as the adhesive cement, according to the manufacturer's instruction as follows. The alloy surfaces were treated with the accompanying primer (Metaltite, Lot No. 0181). Two pastes of equal lengths were squeezed out onto a paper pad and mixed, and the cement paste applied on both adherends. The small-piece adherend was positioned approximately 1 mm higher than the top end of the cast block. It was pressed on by finger pressure for 10 seconds and then clamped with a paper clip (Double Clip, W: 25 mm, Kokuyo Co. Ltd., Osaka, Japan). The bonded assembly was left at room temperature for 24 hours to secure the curing of the

cement.

Load test machine

The cyclic impact loading machine¹⁾ employed in this study is shown in the upper portion of Fig. 1. The test setup was such that that the plunger was lifted to a set height of 3 mm by a rotating cam and then dropped freely onto the small-piece adherend bonded to the cast block. To investigate the effects of different loading weights, plungers of three different weights at 100, 110, and 120 g were employed in this study.

To fabricate a light-weight plunger, an aluminum tube with a steel pin was used. The plunger then dropped on the bonded assembly at a frequency of 4 Hz. By means of an electric counter, the load test machine automatically stopped its rotation after 9,999 ($\approx 10^4$) revolutions. Debonding of the small-piece adherend from the cast block was checked each stop the machine stopped. The loading would continue until the failure of the bonded assembly occurred. For each loading weight, eight specimens were tested.

Cycle number data were analyzed at 0.05 level of significance using ANOVA and *post hoc* Scheffé's test.

Fracture surface analysis

Using a stereomicroscope, the fracture modes of the specimens were examined under $\times 20$ magnification. Following which, typical specimens—with the fractured cement surface remaining on a piece of casting alloy—were gold-sputtered and examined using SEM (Model S3500N, Hitachi, Tokyo).

RESULTS

Loading test

Table 1 shows the cycle numbers that caused the failure of the bonded assembly at each loading weight. For the group loaded with 120 g, it exhibited a significantly low cycle number value as compared to the groups loaded with 100 g and 110 g. For the groups loaded with 100 g and 110 g, there was no significant difference between their loading numbers.

Examination of fractured surfaces

Debonding of the bonded assembly occurred at the interface between the resin cement and the cast block, resulting in a large part of the cement layer remaining on the small piece of casting alloy. This was the same finding that was obtained in the previous study⁶⁾ where heavier loading weights were used.

Figure 2 shows the SEM picture of the debonded surface of resin cement at low magnification. Long

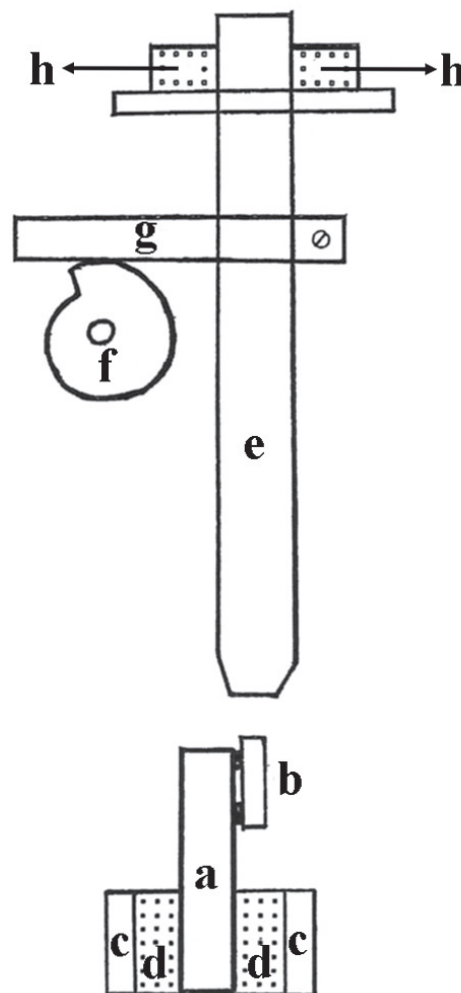


Fig. 1 A schematic drawing of the experimental setup. a: Adherend (cast block); b: Adherend (small piece of casting alloy); c: Plastic ring; d: Self-curing resin; e: Plunger; f: Rotating cam; g: Lifting arm; h: Weight (when needed)

Table 1 Cycle numbers that caused the failure of bonded assembly ($\times 10^4$)

Plunger weight (g)	Mean \pm SD (range)
100	1756 \pm 680 ^a (842–2514)
110	1403 \pm 515 ^a (601–2016)
120	420 \pm 200 ^b (181–751)

n=8

Same letter indicates no significant difference.

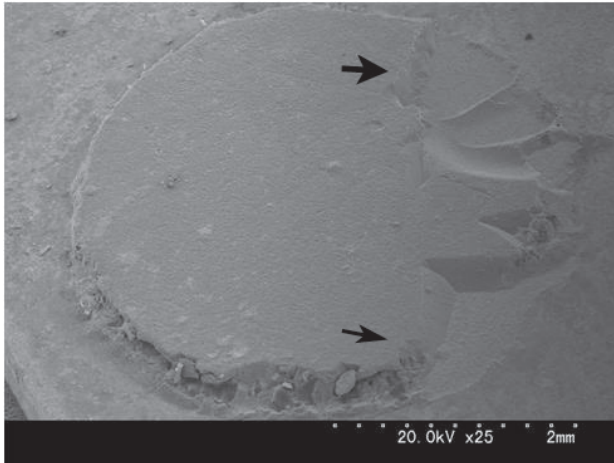


Fig. 2 Low magnification view of the fractured surface with 110 g at 1948×10^4 . Arrows indicate the fracture lines.

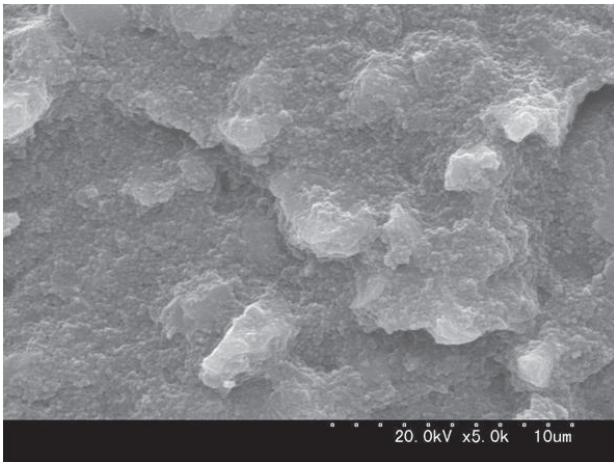


Fig. 3 High magnification view of the same specimen.

cracks penetrating through the cement layer could be seen, resulting in a bulk fracture of the cement. At a higher magnification, Fig. 3 shows that small fillers were exposed at the fractured surface. This indicated that fracture lines developed along the interface between the fillers and the matrix resin.

DISCUSSION

In this study, it was investigated how the bond durability of a composite-type resin cement would be influenced by loading weights under cyclic shear loading, with plunger weights ranging from 100 g to 120 g. Based on the results obtained, it was observed that the cycle numbers increased with decrease in plunger weight. Cycle number yielded by the group loaded with 120 g was significantly lower than those

loaded with 110 g and 100 g. Within the limitation of this study, it was noted that there was a discrepancy in the tolerance of resin cement between loading with 120 g and 110 g. Therefore, compared to loading with 100 g and 110 g, loading with 120 g induced an approximately four-fold stress which then led to the debonding of the bonded assembly.

Fatigue fracture is caused under repetitive stress, although the stress is very much smaller in magnitude than the fracture strength of the material. This is because fatigue is induced by small stresses accumulated at weak areas such as the boundary between filler particles and matrix resin. Initiation of flaws leads to crack formation when stress reaches a certain level. Subsequently, the crack propagates through to another part of the material⁷. By the same token, a long period of stress loading is needed to induce fatigue fracture, as shown in this study *via* the loading of 100 g and 110 g which resisted more than 10^7 times of loading. Indeed, the cycle number — exhibited by 100 g and 110 g loading were counter to our expectation for loading less than 130 g. This was against the background whereby bond durability exhibited at 130 g loading was not so high.

On the mode of fracture obtained in this study, it was almost the same as that obtained in the previous study⁶) where heavier loading weights were employed. In the context of the present study, the fracture mode of composite-type cement was consistently bulk fracture, regardless of the loading weight.

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