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Bond Durability of Resin Cements to Au-Pd-Ag Alloy under Cyclic Impact Load

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The bond durability of resin cements to a 12% Au-Pd-Ag alloy was studied through cyclic impact tests with different loads. A piece of casting alloy was bonded to a cast block with two types of resin cements, Super Bond C&B and Bistite II. A shear load was applied onto a small piece of alloy until debonding of the specimen, using different weights of plungers, 130 g, 230 g, 330 g and 430 g.

The specimen bonded with Super Bond exhibited a higher resistance than that with Bistite II. The fracture modes of the debonded cements were completely different from each other. That is, Bistite II showed a bulk fracture of cement by the crack penetrating through the cement layer. On the other hand, Super Bond showed damages limited to the surface and no bulk fracture. The mode of fracture was dependent not on the loading weight but the types of resin cements used.

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

INTRODUCTION

Recently, the clinical usage of resin cements is becoming popular due to its bonding efficacy to tooth substances and dental alloys. The bond durability of resin cements is a clinical concern when selecting a material brand. Most reliable guides are those obtained from clinical surveys, but they require a long observation period, among other things. Occasionally, brands have disappeared from the market when the data of the clinical surveys were finally collected. Therefore, laboratory tests are usually performed instead of clinical surveys.

In order to investigate the mechanical properties of dental materials, an accelerating method is often adopted in a laboratory test. A cyclic impact loading test is one such method. Htang, Ohsawa & Matsumoto[1] applied this method onto the surface of a resin composite restoration and measured the crack length induced inside the restorations. No cracks were found when the loading weight was light, while a crack length developed in accordance with an increase in the loading weight. Thus, the selection of the loading weight is a crucial factor for evaluating the properties of materials.

It seems that the effect of the loading weight on the durability of a bonded unit with resin cements is not well discussed under the cyclic impact test. In this study, the effect of the loading weight on the durability of a bonded unit with resin cement was studied through the application of different loading weights.

MATERIALS AND METHODS

Preparation of adherend

A casting alloy (Castwell M.C. GC Corp., Tokyo, Lot No.110191) was used as an adherend. This alloy is commonly used in Japanese dentistry for inlays, cast-crowns and brides. The lower part of Fig. 1 shows a specimen assembly. One pair of adherends was a piece of casting alloy (Fig. 1,b), 9.6×6.3×1.6 mm^3 and 1 g in weight, used as delivered from the manufacturer. The other (a) was fabricated by cast. It was approximately 25×10×5 mm^3, had a surface 25×10 mm^2, and was finished flat with a silicone carbide paper #600. The cast block was mounted with a self-curing resin (d) as perpendicular as possible in the plastic ring (c) which facilitates the fixing of the specimen on the testing machine. That is, the flattened surface of the cast block was temporally fixed to another rectangular metal block that was placed on a pedestal, and hung down into the plastic ring.

Preparation of bonded unit

The sandblaster (Microblaster, Comco Corp. U.S.A.) with Al_2O_3 of 50 μm and a pressure of 3.5 kg was used for the surface treatment of both adherends, and frosted surfaces were obtained.

An adhesive tape 0.2 mm in thickness with a hole 4 mm in diameter was pasted on a treated surface of the cast block to regulate the adhesive area. Two types of adhesive resin cements, an MMA-base (Super Bond C&B, Sun Medical, Moriyama City) and a composite type (Bistite II, Tokuyama Dental Corp. Tokuyama City) were used as the bonding cement, according to the manufacturer's instruction. Details of each component and Lot No. are listed in Table 1.

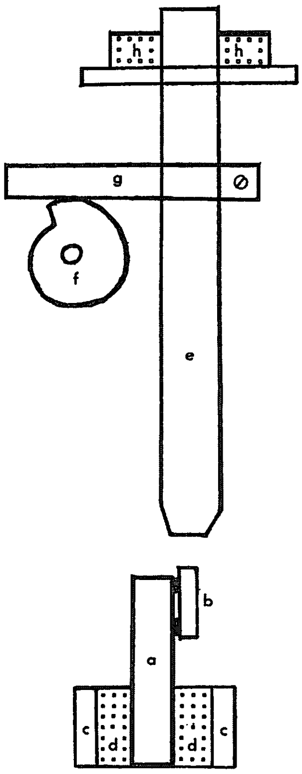

Fig. 1 A schematic drawing of the experimental set-up.
a: Adherend (Cast block)
b: Adherend (A peace of casting alloy)
c: Plastic ring
d: Self-curing resin
e: Plunger
f: Rotating cam
g: Lifting arm
h: Weight (when needed)
The plunger dropped from 3 mm height with a frequency of 4 cycle/min.

Table 1 Resin Cements used in this study

Brand Names	Lot No.
Super Bond C&B	81001
V Primer	80402
Monomer	802061
Catalyst	71201
Polymer	
Bistite Ⅱ	
Metalite	010
Paste	81B, 04R

Table 2 Cycle numbers that caused debonding of the specimen ($\times 10^4$)

Plunger Weight (g)	Super Bond	Bistite Ⅱ
130	329.1±168.3	49.8±52.2
	(127～602)	(7～174)
230	15.3±10.0	1.4±1.0
	(8～36)	(1～4)
330	1.9±1.5	1.7±2.2
	(1～6)	(1～8)
430	1.7±0.9	1.1±0.3
	(1～4)	(1～2)

Mean±SD, ( ): Range n=10

The alloy surfaces were treated with the respective primers. Concerning Super Bond, a polymer powder was added into the catalyst-activated monomer liquid in a chilled dish and mixed together. Two pastes of Bistite Ⅱ were squeezed in equal lengths on a paper pad and mixed. The mixed cement was painted on both adherends. The small piece was positioned approximately 1 mm higher than the top end of the cast block, pressed by finger pressure for 10 seconds, and then placed flat. The bonded assembly was left on the bench for two days to secure the curing of the cements.

Testing machine and loading

A cyclic impact loading machine[1] was employed in this study as shown in the upper portion of Fig. 1. This apparatus was designed in such a manner that the plungers (e) were lifted up by rotating cams (f) to a set position (3 mm in this investigation) and dropped freely onto the top of a small piece (b) bonded to the cast block (a). Different weights of plungers, 130 g, 230 g, 330 g and 430 g were employed. The plunger was dropped onto the specimen with a frequency of 4 Hz. Rotation of the testing machine automatically stops after 9,999 ($\fallingdotseq 10^4$) revolutions by means of an electric counter. Debonding of the small piece from the cast block was checked at each stop of the machine. When the specimen broke within the first stop, it was termed that the specimen broke at 10^4 revolutions, and within the second stop at 2×10^4 revolutions. The loading was continued until failure of the specimen. For each weight of loading, 10 specimens were tested. The data was analyzed at the 0.05 level of significance, using the un paired t test between other brand materials, and ANOVA and Post hoc among the same brand materials.

Observation of the fractured surface

Firstly, the fracture mode of the specimens was checked under a stereo microscope $\times 20$. Then, the typical specimens were gold spattered and examined with SEM (S-3500N, Hitachi, Tokyo).

RESULTS

Loading test: Table 2 shows the resultant cyclic number that caused specimen failure. In comparison with Super Bond and Bistite Ⅱ, the specimens bonded with Super Bond showed a statistically significant higher resistance than that with Bistite Ⅱ at 130 g and 230

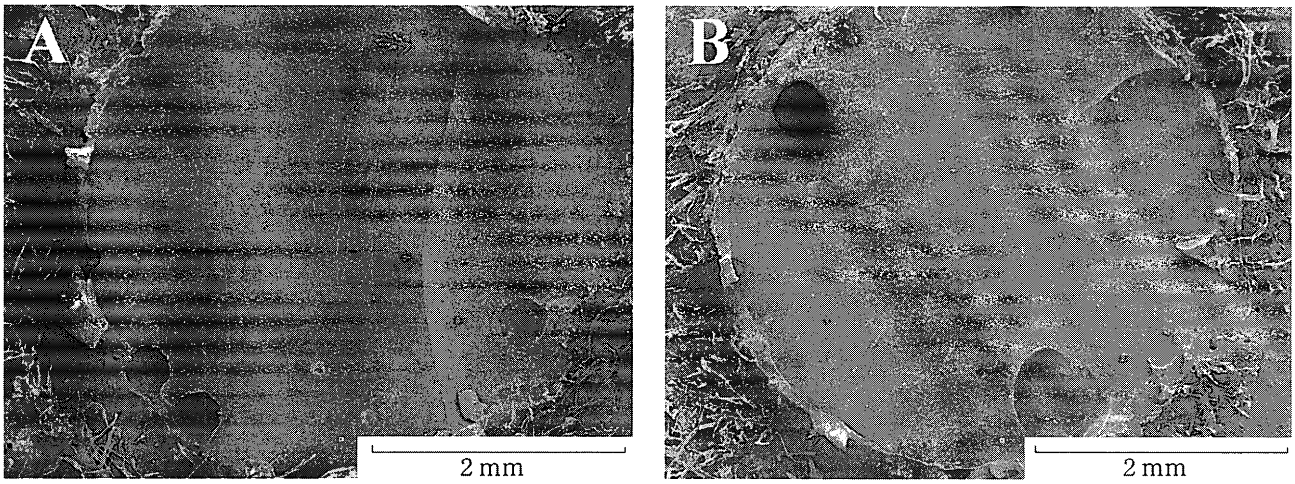

Fig. 2 SEM picture of debonded surface of Bistite II.
A: fractured surface with 130 g plunger
B: fractured surface with 430 g plunger

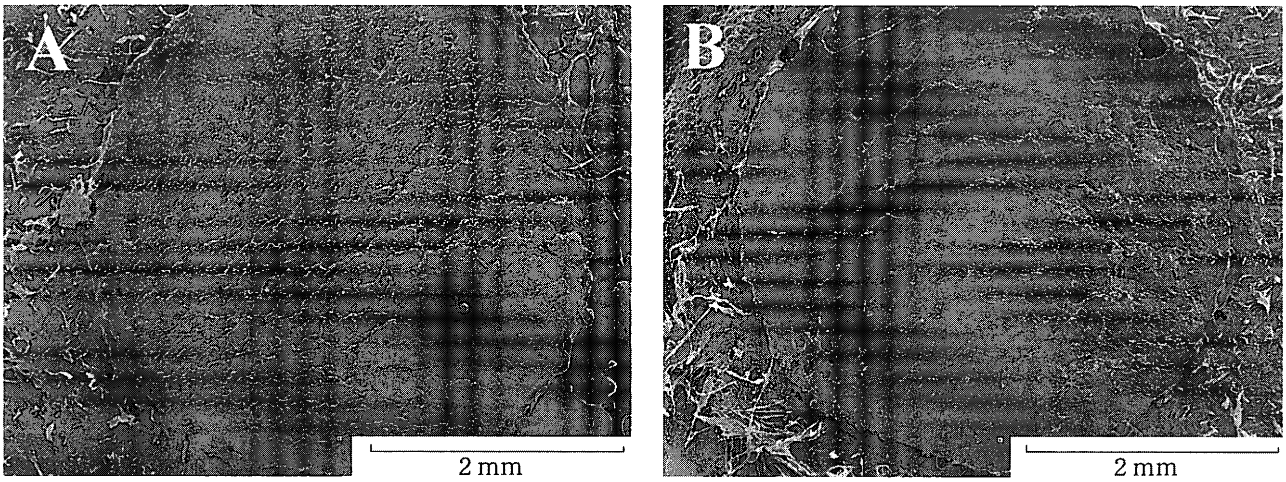

Fig. 3 SEM picture of debonded surface of Super Bond.
A: fractured surface with 130 g plunger
B: fractured surface with 430 g plunger

g weight loading. There were no statistically significant differences between the two groups at 330 g and 430 g weight loading.

Concerning Super Bond, when the plunger of 130 g was applied, the specimen resisted more than 100 $\times 10^4$ times loading. When, the plunger weight was increased up to 230 g, the specimen broke around 15 $\times 10^4$ times loading, resulting in a statistically significant decrease in loading numbers. There were no statistically significant difference between the two groups loaded with 330 g and 430 g.

For Bistite II, when the 130 g plunger was applied, the specimen broke around 50×10^4 times loading. There was no statistically significant difference among the three groups when a plunger weight of more than 230 g was loaded.

Examination of fractured surfaces: All specimens were fractured at the interface between the resin cement and the cast block, resulting in the cement layer remaining on the small casting alloy piece. Fig. 2 shows the SEM picture of the debonded surfaces of Bistite II. Long cracks are penetrating through the cement layer, resulting in a bulk fracture of the cement. On the other hand, Fig. 3 shows the specimen with Super Bond. A bulk fracture was not seen and the damage seemed to be limited on the surface of the cement. The fracture mode seemed to be independent of the loading weight but was peculiar to the type of cement, composite-type or MMA-base.

DISCUSSION

The cyclic impact load is a useful method as an accelerating laboratory test and often used in Japan[2-5]. This method loads a cyclic dynamic stress on the specimen that simulates an occlusal force. Thus, this test method is considered to be close to the clinical situation rather than other methods such as bond strength measurement. Htang *et al.*[1] have utilized this method on a resin composite placed in a cavity prepared on bovine teeth. They discussed the suitable filler content against the cyclic impact load by examining the crack length induced inside the resin composite. As a preliminary study of their research, the propagation of the crack was dependent on the loading weight. That is, no crack was observed after 10^5 times loading with a plunger weight of 250 g, but two out of 5 specimens exhibited cracks with a plunger weight of 350 g. This would mean that there is a critical weight which induces cracks during limited loading times. In this study, a shear load ranging from 130 g to 430 g, each differing 100 g, was applied onto the specimen. Almost all of the specimens failed within the first loading cycle when the loading weight exceed the critical point, that is, 330 g for Super Bond and 230 g for Bistite II. This indicates that an optimal loading weight must be selected for this kind of test procedure. And then, applying with a light loading for a long duration would cause a fatigue fracture. A cyclic impact test by shear loading seems to be a more severe method than that by vertical loading onto the bonded unit. When a vertical loading weight of 330 g was added at a 6×10^5 cycle to the specimen to which a casting alloy pair was bonded with resin cement, no crack was found under SEM observation, even though composite type cement was used[5].

Fig. 4 shows a kind of S-N curve for Super Bond and Bistite II. Super Bond showed a much higher resistance against the impact load than Bistite II. Super Bond consists of an MMA-base resin without any filler, while Bistitte II consists of a dimethacrylate resin and filler (77wt%). Therefore, a coefficient of elasticity of Bistite II is much higher than that of Super Bond, resulting in Bistite II being more brittle than Super Bond. Furthermore, stress is apt to accumulate at the interface between the filler and matrix of the composite material. This would be one of the reasons why the long cracks developed in Bistite II. The single-phase composition, not containing the filler, seemed to be much more resistant against the impact loading.

The mode of the fractured surface was dependent on not the loading weight but the types of cements used. When a light weight plunger was used, many loading times were actually needed to induce failure of the bonded units, and the pattern of the fractured surface resembled that caused by the lesser loading time with a heavier weight plunger. This would mean that debonding occurs when the damages at the bonded interface accumulated and reached a certain level. Therefore, the fracture mode of the bonded interface was dependent on the types of cement used.

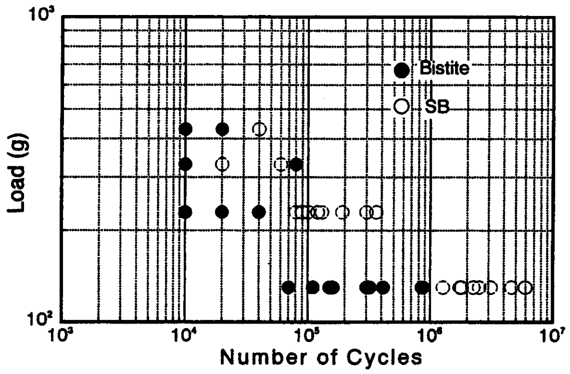

Fig. 4 Load - cycle number relationship.
X and Y axes are expressed in logarithm
Each group has 10 specimens but some figures are in heaps

There are many air bubbles in the mix of cement as shown in the SEM picture (Fig. 2 and 3). It is inevitable to avoid an inclusion of air bubbles in the cement mix because mixing is essential for both cements, paste/paste for Bistite II, powder/liquid for Super Bond. It was thought that the frequency of the air bubbles in the cement mix would be one of the reasons causing a wide scatter of the data obtained. Oxygen in the air bubble hinders the conversion of the double bond[6] of resin cement and lowers the mechanical properties of the cement. Also, the existence of air bubbles causes a concentration of stress and initiates cracks.

Concerning the effect of the film thickness of cements on the retentive strength of restorations, both opinions, "yes"[7] or "no"[8,9], have been reported in previous studies. Our study[10,11] revealed that the retentive strength of the inlays were almost comparable to each other in spite of the film thickness of the adhesive cements, for which class I metal inlays were bonded into the cavity prepared on the bovine teeth with different cement thickness over 400 μm expressed by the sum of both sides. The other study[12] reported that a thicker film thickness (200 μm) brought a higher fracture toughness than the thinner specimen (20 μm). The thinner layer of resin induces greater stresses at polymerization than the thicker one[13]. Therefore, the author believes that it does not have an influence on the bond strength of bonded units with a film thickness of adhesive cement within 0.2 mm on one side.

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