Studies on Tensile Bond Strength Tests for Dental Cements*

—Relationship between Measurement Conditions and Tensile Bond Strength—

by

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I. Introduction

In 1968, polycarboxylate cement was developed by D.C. SMITH [1], and after that glass ionomer cement and resin cement having adhesive characteristics were developed [2–5]. There are many reports concerning the bond strength of these cements, but a method of standardization for measuring bond strength has not been established. Therefore, the measuring methods are so full of variety that the obtained results can hardly be compared with one another. Even when using the same measuring method, there are so many differences between the quality of adherends and surface treatment conditions, specimen size, shape and cement film thickness that these results cannot easily be compared with each other.

The authors, in the present study, purposed to establish a base for standardizing the measuring method for bond strength. Therefore, by referring to many reports [6–29], a tensile bond strength test method [7–8] was selected, and by changing film thickness, surface roughness of the specimen, tensile velocity, bond area and the kinds of adherends, they measured bond strength so as to develop a measurement method and conditions showing reproducible bond strength.

II. Experimental Materials and Methods

1) *Materials*

Polycarboxylate cement (CARLON, Sankin Industry, [Powder No. 112-606, Liquid No. 119-010]) was used as a cement. Powder-liquid ratio, mixing time and mixing technique were all in accordance with the manufacturer's instructions.

2) *Experimental methods*

(1) Bond strength test specimen

The adherends (Fig. 1) were made of stainless steel whose peripheral bond surface gutter was treated with a PTFE splay, polished with an abrasive paper and degreased with acetone. The test process was as follows: two minutes after the commencement of mixing, a cement mix, 0.03 g, was placed on the bond surface, and then one adherend was placed on another, under a 2 kg load for 13 minutes using a loading device (Fig. 2). After that, the load was immediately released from

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Fig. 1 Adherends

Fig. 2 Loading device

Fig. 3 The scheme of measurement of film thickness

Table 1 Spacer thickness and film thickness

Space thickness (μm)	Measurement value of film thickness (μm) (S.D.)
15	14.2 (1.1)
30	27.0 (1.1)
45	44.0 (1.1)
60	60.6 (3.0)
75	73.0 (2.1)
90	87.3 (4.0)

the specimen (a set of adherends) and was immersed in the 37°C deionized water and left for 24 hours to measure bond strength.

2) Film thickness

The experimental method for obtaining the fixed film thickness was as follows: A commercial aluminum foil, approximately 15 μm in thickness, was used as a spacer to be piled up 1 to 6 sheets. Film thickness (Fig. 3) was derived from the maximum peak-valley roughness (R_{max}) which was obtained from the profile curve measured with an instrument for surface roughness (Surfcom 2000: Tokyo Seimitsu). Measurement conditions were as follows: a probe radius of 0.800 mm, a traversing length of 2.50 mm, a tracing speed of 0.12 mm/min.. The thicknesses of the spacers and the measurement values of film thickness are shown in Table 1. Hereafter film thickness will be indicated by spacer thickness.

(3) Surface roughness

When stainless steel was used as an adherend, the surface was polished with a silicon carbide abrasive paper (type: DCCS; grit No.: 100, 320, 400, 600, 800; JIS R 6253) and a specimen polishing machine (Marumoto Kogyo). An arithmetic average roughness (R_a) of the surface of the polished specimens was also measured by the Surfcom 2000. The measuring conditions of surface roughness were as fol-

Fig. 4 Adherends (stainless steel)

Table 2 Surface roughnesses of adherends

Adherend	Abrasive paper	Surface roughness (μm)
ST	#100	0.41
	#320	0.17
	#400	0.09
	#600	0.04
	#800	0.03
Pd	#400	0.09
NC	#400	0.09
I	#800	0.10

Stainless steel (ST), 12% Au-Ag-Pd alloy (Pd), Ni-Cr alloy (NC) and Ivory (I).

lows: a probe radius of 0.005 mm, a traversing length of 2.50 mm, a tracing speed of 0.12 mm/min., a cut-off of 0.80 mm. Hereafter surface roughness will be indicated as the grit number of each abrasive paper (for example: #100).

(4) Tensile velocity

Tensile bond strength was measured by a universal testing machine (Auto Graph IS-5000: Shimadzu) with cross-head speeds of 1.0, 1.5, 2.5, 5.0, 10.0, 15.0 and 20.0 mm/min.

(5) Bond area

The set of stainless steel adherends shown in Fig. 4 was prepared as a specimen. Bond areas were 1.54 cm² (14 mm in diameter), 0.79 cm² (10 mm in diameter) and 0.38 cm² (7 mm in diameter).

(6) Kinds of adherends

Four kinds of adherends — stainless steel, (ST, hereafter), shown in Fig. 1, 12% Au-Ag-Pd alloy (Pd, hereafter), Ni-Cr alloy (NC, hereafter) and ivory (I, hereafter) — were prepared. Bond strength was measured at the combination of ST-ST, ST-Pd, ST-NC and ST-I. When metal was used as an adherend, the surface roughness was #400. In the case of I, when the roughness was #800 (Table 2), it indicated almost the same roughness as that of metal, so bond strength was measured at this roughness.

Measurements of the above-mentioned (1)~(6) were carried out at $23 \pm 1^\circ\text{C}$ and at $50 \pm 5\%$ in relative humidity. Ten specimens were tested for each com-

Fig. 5 The relationship between film thickness and bond strength (#400, 10 mm/min., 0.79 cm²)

bination of conditions. Comparisons of tensile bond strength values were made by using Student's *t*-test (at the 5% level of significance).

III. Experimental Results and Discussion

(1) Film thickness and bond strength

The relationship between film thickness and bond strength is given in Fig. 5. The conditions are as follows: the adherend: ST-ST, surface roughness: #400, tensile velocity: 10 mm/min., bond area: 0.79 cm².

When film thickness was 45 μm, the bond strength was 191.0 kg/cm². That was the largest value with the smallest standard deviation compared with the other cases. This was followed by 15 μm and 30 μm, and their tensile bond strengths were large values of 185.9 and 187.3 kg/cm². These three means were not significantly different. When film thickness was over 60 μm, tensile bond strength showed small values. They were not significantly different. In the range of 15~45 μm, it showed a cohesive destruction of the cement layer, but over 60 μm, it showed interfacial destruction. It seems that the reason why bond strength decreased considerably over 60 μm in film thickness is that as the layer becomes thicker, voids in the cement increase and some defects will occur in the set cement.

(2) Surface roughness and bond strength

The relationship between surface roughness and tensile bond strength is shown

Fig. 6 The relationship between surface roughness and bond strength (45 μm , 10 mm/min., 0.79 cm^2)

Fig. 7 The relationship between surface roughness and bond strength (45 μm , 10 mm/min., 0.79 cm^2)

in Figs. 6 and 7. The experimental condition is the same as (1) in tensile velocity, bond area, adherend and film thickness (45 μm).

In the combinations of the same surface roughness adherend (Fig. 6), tensile bond strength showed the largest value, 191.0 kg/cm^2 , at #400. This mean showed significant differences from the means obtained at other roughnesses. In the case of #600 and #320, tensile bond strength showed 182.0 and 179.0 kg/cm^2 and there was no significant difference between them. In the case of #100 and #800, it showed a small value and also showed no significant differences.

When the surface roughness in #400 was combined with the other adherends (Fig. 7), the combination of #400-#600 showed the largest bond strength of 107.0 kg/cm^2 and in the other cases it showed the range of 92.9~101.8 kg/cm^2 . There were no significant differences between the combinations #400-#100 and #400-#320, but in the other cases significant differences were recognized. There are many reports concerning surface roughness and bond strength, and it was pointed out that bond strength was considerably influenced by cement wettability with the adherend. When air and moisture exist between the cement and adherend interface, bond strength decreases [30-32]. Whether the mechanism of bonding is chemical or mechanical it is clear that the cement completely wets the entire surface of the adherend and is the main factor in making bond strength increase. However, when surface roughness is large, the surface invariably contains innumerable small hills,

Fig. 8 The relationship between tensile velocity and bond strength
(#400, 45 μm , 0.79 cm^2)

valleys, and crevices, and the bonding area increases as compared with small surface roughness, but because the cement does not reach deep into the valleys and crevices finely, air pockets occur and wettability decreases, so that chemical as well as mechanical bonding decreases. Surface imperfections are always a potential hazard in attaining an adhesive bond [32]. Also, as film thickness becomes thicker, the production of stress at the cement-adherend interface increases. Stress caused by thermal changes and mechanical stress concentrate to cause internal flaws such as air spaces and structural defects [32], and bond strength decreases. In this case, when the surface roughnesses of adherends are different from each other, surface energy is also different, and mechanical stress concentrates to cause imperfections. It is clear that bond strength decreases.

When the surface roughness was small, bond strength decreased. In this case the reason was thought to be that both the bonding area and mechanical interlocks decreased.

(3) Tensile velocity and bond strength

When the conditions of surface roughness, bond area and adherend are the same as (1) and those of film thickness are the same as (2), the relationship between tensile velocity and bond strength is as shown in Fig. 8.

The largest bond strength of 191.0 kg/cm^2 was shown at 10 mm/min. and its value was recognized as a significant difference from the values of the other cases. Next were the cases of 15.0 mm/min. and 5.0 mm/min. and they showed 179.2 and

Student's *t*-test0.38cm²

	15	30	45	60
15				
30			*	*
45			*	*
60				*

0.79cm²

	15	30	45	60
15				*
30			*	*
45			*	*
60				*

1.54cm²

	15	30	45	60
15			*	
30			*	
45				*
60				*

* : to > t (φ, .0.05)

15μm

	0.38	0.79	1.54
0.38		*	*
0.79		*	*
1.54			*

30μm

	0.38	0.79	1.54
0.38		*	*
0.79		*	*
1.54			*

45μm

	0.38	0.79	1.54
0.38		*	*
0.79		*	*
1.54			*

60μm

	0.38	0.79	1.54
0.38		*	*
0.79		*	*
1.54			*

Fig. 9 The relationship between bond area, film thickness and bond strength (#400, 10 mm/min.)

176.6 kg/cm² and there was no significant difference between them. Contrarily, the smallest bond strength was shown in the case of 1.0 mm/min.

Thus, it is apparent from the experimental results that the largest bond strength was shown in the case of 10 mm/min. in tensile velocity. In the cases with both lower and higher speeds than 10 mm/min. tensile bond strength decreased. The reason for this decrease in bond strength in the lower speeds was inferred to be a twist-like stress caused by tension propagated to the adherends and the concentration of stress due to the mechanism of the measurement machine. On the other hand, in the higher speeds, because the impact was added to the specimens by instantaneous initial velocity, it appears, inherent strength was not obtained. But it will be necessary to discuss these matters in the future.

(4) Bond area, film thickness and bond strength

When the conditions of surface roughness, tensile velocity and adherend are the same as (1), the relationship between bond area, film thickness and bond strength is as shown in Fig. 9. Furthermore, the relationship in the case of bond area 0.79 cm², replotted from Fig. 5, is also shown in Fig. 9 for comparison.

When the bonding area was 0.79 cm², the largest bond strength was shown and in this case whichever film thickness was selected, a bond strength larger than in the other cases was shown. In the case of 0.38 cm², when film thickness was 30 and 45 μ m it was 163.0 and 170.1 kg/cm² respectively. These means are significantly different. In the case of 1.54 cm², compared with other cases, bond strength in any film thickness showed a small value. Thus, in any bond area when film thickness was 45 μ m, a large bond strength was shown. Also, between the cases of 0.79 cm², 45 μ m, and the other cases of bond area, and of film thickness, a significant difference was recognized.

We have few reports concerned with bond area and bond strength, but GENT [33] indicated an equation of its area and its strength, and he described that the changes in bond strength depended on elasticity of the bonding agent, its thickness, the stress concentration factor, critical strain energy in the edge regions, and disc radius and that there is no proportional relationship between bond area and bond strength. Although it was not evident that in the case of 0.79 cm² a large bond strength was shown, in this present cement, it was thought that in the case of 1.54 cm², a larger bond area, a small value was shown in comparison with 0.79 cm² because of increasing stress concentration and strain energy.

(5) Kinds of adherends and bond strength

When the adherends were ST, Pd, NC, the condition of surface roughness was #400. It was #800 in the case of I and the conditions of tensile velocity and bond area were the same as (1). When ST-Pd, ST-NC and ST-I were prepared as the specimens the relationship between film thickness and tensile bond strength was as shown in Fig. 10. As film thickness increased, 15 μ m to 45 μ m, whatever the combination of the adherends, bond strength tended to be large, though in 60 μ m, bond strength decreased. Thus the largest bond strength was shown in 45 μ m, and in the cases of ST-Pd and ST-NC it was 65.0 and 63.7 kg/cm². In the case of ST-Pd, whichever film thickness, 30, 45 or 60 μ m, there were no significant differences between them. But in the case of ST-NC, whatever the film thickness, there were

Fig. 10 The relationship between film thickness and bond strength (ST-NC, ST-Pd, ST-I) (10 mm/min. 0.79 cm^2)

no recognized significant differences. In the case of ST-I, at $45 \mu\text{m}$, $49.0 \text{ kg}/\text{cm}^2$ was shown, but there were no recognized significant differences between $30 \mu\text{m}$ and $45 \mu\text{m}$.

Many reports concerned with the relationship of adherends and bond strength have been published and the bonding mechanism that occurred in the cases where metal was used as an adherend is almost inferred [34–37]. That is, the bonding of metal and polycarboxylate cement is changed by surface potential, oxide film thickness and surface tension of the metal. It is well known that bond strength increases in the case of base metal, whose surface forms a thin and durable cover of oxide film. Especially, on ST and NC, the oxide film formation presented chemical passivity [11] and bond strength was large. Also in Pd, as it contains base metal elements like Cu and Zn, its bond strength becomes comparatively large [11].

On the other hand, the bond strength of ST-I was lower than the bond strengths of the ST-metals in any film thickness. The reason why bond strength decreased was thought to be that I contains water [31–33], and that dimensional changes due to thermal change on the ST and cement are greatly different from those of I. It is well known that bond strength considerably decreases when I is immersed in water. Surface roughness of I polished with #400 showed $0.30 \mu\text{m}$, that is three times that of ST, NC and Pd, and bond strength was smaller than that of #800.

In other words, it was clear that when surface roughness of both sides was about the same level, a large bond strength was shown.

IV. Conclusion

This present study purposed to establish a base for standardization of the measuring method of bond strength in polycarboxylate cement. Therefore, a tensile bond strength test method was applied in order to obtain the method and conditions for a large and stable bond strength. To do this the conditions of film thickness, surface roughness of adherends, tensile velocity, bond area and kinds of adherends were changed variously and examined. The experimental results are as follows:

1. When film thickness was 45 μm , bond strength showed the largest stable value.
2. In the cases of ST-ST, ST-Pd, ST-NC, bond strength was large when both surfaces were polished with silicon carbide abrasive paper #400. Significant differences were recognized in comparison with the other cases.
3. In the case of a tensile velocity of 10 mm/min. in cross-head speed, bond strength was obviously the largest.
4. Bond strength was the largest in the case of bond area 0.79 cm^2 (10 mm in diameter), followed by 0.38 cm^2 (7 mm in diameter). But in the case of 1.58 cm^2 (14 mm in diameter), bond strength was small.
5. Whatever the various bond areas, when film thickness was 45 μm , bond strengths were the largest.

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