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Original Paper

Effect of Aluminoborate Whiskers on Mechanical Properties of Polycarboxylate Cements

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The mechanical properties of polycarboxylate cements containing 20 mass% of four kinds of aluminoborate whiskers with different fiber lengths and diameters were evaluated. Bending strength of the cements increased with increase in fiber length, whereby the strength of the cement with the longest fiber was about two times greater than that of whisker-free cement. The diametral tensile strengths of the four fiber-reinforced cements were also about two times greater than that of whisker-free cement, but which was not dependent on fiber length. Compressive strength was the same or slightly higher than that of whisker-free cement. SEM observation of fractured specimen after diametral compression test showed high affinity between the cement matrix and the whisker.

Key words: Polycarboxylate cement, Whisker, Mechanical property

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INTRODUCTION

On the mechanical properties of dental and bone cements, it is important to improve not only the compressive strength but also the tensile and bending strengths. When compared to resin cements, polyalkenoate cements offer some upper-hand characteristics such as high fluoride ion release, lower residual monomer content, and reduced irritant action on the pulp. Despite these advantageous features, the failure modes of polyalkenoate cements after tensile testing frequently show cohesive fracture within the cement matrix as well as low adhesive strength between the cement and adherend. Therefore, to improve the performance of polyalkenoate cements, it must be achieved by increasing the tensile strength.

The authors have previously reported that the diametral tensile strength of polycarboxylate cements was excellently improved by the addition of aluminoborate whiskers of 20−50 μm fiber length and 1−5 μm diameter[1]. Conversely, the strength of the cements containing irregular-shaped and spherical fillers — with the same composition as the whiskers — was not improved[1].

The objective of this study was to investigate the effect of whiskers on the bending and tensile strengths of polycarboxylate cement, whereby the latter contained the same composition of whiskers of different lengths and diameters.

MATERIALS AND METHODS

Aluminoborate whiskers

Table 1 shows the four kinds of Alborex G® whiskers (Shikoku Chemicals, Kagawa, Japan) used in this study. Chemical composition of the whiskers was $Al_2(SO_4)_3/H_3BO_3/K_2SO_4$ and the specific gravity of each whisker was 2.93. For Alborex GA (hereinafter referred to as GA), the shortest among the four kinds of whiskers, fiber size was 5 μm in length and 0.2−0.5 μm in diameter. The abbreviations of other whiskers, as shown in Table 1, are GB, GC, and GD. The mean length and diameter of the whiskers in increasing order is GA<GB<GC<GD. Length and diameter of the largest whisker GD were 20−50 μm and 1−5 μm respectively.

Polycarboxylate cements

A commercial polycarboxylate cement (Calron SmFP, Dentsply Sankin, Tochigi, Japan) was used as a basic cement to compare the supplementary effects of the whiskers. An amount of 20 mass% of each whisker was mixed with the polycarboxylate cement powder in a mortar with pestle to avoid the smashing of whiskers. Cement powder (1.08 g) containing the whisker and the cement liquid (0.60 g) were mixed on a mixing pad for 30 seconds using a plastic spatula. The mixed paste was filled into a Teflon mold, whereby rectangular-shaped plates of 30×3×3 mm were prepared as specimens for the bending test.

Specimens for the compression and diametral tensile tests were prepared using the same method. Size of the specimen was 6 mm in diameter and 12 mm in length for the compression test and 6 mm in diameter and 6 mm in length for the diametral tensile test. Codes of the hardened cements prepared from the four whiskers were GA, GB, GC, and GD. Whisker-free cement specimens were prepared as a control. Seventy-five specimens were stored in 37 °C water for two days prior to testing.

Bending, compression, and diametral tensile tests

Bending strength was measured with a universal testing machine (AGS-500, Shimadzu Co. Ltd., Kyoto Japan) using a three-point bending fixture with a span of 20 mm. Crosshead speed of the test was adopted as 0.5 mm/min to facilitate the calculation of the bending modulus. Bending modulus of elasticity was calculated from the straight portion up to the proportional limit of the load-time curve obtained from the bending test.

In this study, reasons for obtaining the elasticity modulus from the bending test were as follows: (1) whiskers were added to the cement with a view to improving the latter's bending and tensile strengths; (2) a stress-strain curve from a compression test is unsuitable for obtaining the moduli of brittle materials like this cement.

The compression and diametral tensile tests were carried out using the same testing machine at a crosshead speed of 2.0 mm/min. All data were statistically analyzed by Student's t-test.

SEM observation

To evaluate the affinity between the cement matrix and the whisker, the fractured surface of the specimen after the diametral compression test was observed using a scanning electron microscope (SEM) (JSM-35C, JEOL Ltd., Tokyo, Japan).

RESULTS

Mechanical properties

Table 2 shows the values of the compressive, bending, and diametral tensile strengths, as well as the bending moduli of elasticity. Bending strength of whisker-free cement was 15.2±2.6 MPa, while that of GA was 20.1±2.1 MPa. Therefore, there was a statistically significant difference between GA and the whisker-free cement ($p<0.05$). Bending strengths of GB, GC, and GD were 26.0±2.4, 30.2±2.4, and 32.3±3.5 MPa respectively. The values of GC and GD were about two times greater than that of whisker-free cement and were significantly different from those of GA and GB.

The elastic modulus of whisker-free cement was 3.99±0.35 GPa, and the values of the four cements containing the whiskers — namely GA, GB, GC, and GD — were higher than that of whisker-free cement ($p<0.05$).

The diametral tensile strength of whisker-free

Table 1 Compositions, lengths, diameters, and codes of the various Alborex G® whiskers

Alborex G whisker	Mole ratio of composition $Al_2(SO_4)_3/H_3BO_3/K_2SO_4$#			Length$ (μm)	Diameter$ (μm)
GA	8	2	10	5	0.2−0.5
GB	8	2	20	10−30	0.5−1
GC	8	2	25	20−30	1−2
GD	8	2	30	20−50	1−5

#: Mole ratio of metal

$: Measured by Shikoku Chemicals

Table 2 Mechanical properties of polycarboxylate cements containing 20 mass% of Alborex G® whiskers immersed in water at 37°C for 2 days

Cement	Compressive strength (Mpa)	Diametral tensile strength (Mpa)	Bending strength (Mpa)	Elastic modulus& (Gpa)
Control	63.8 (7.4)^{a}	5.28 (0.8)^{a}	15.2 (2.6)^{a}	3.99 (0.35)^{a}
GA	74.1 (1.5)^{b}	9.88 (1.3)^{b}	20.1 (2.1)^{b}	4.86 (0.27)^{b}
GB	72.0 (5.8)^{ab}	10.3 (1.2)^{b}	26.0 (2.4)^{c}	5.41 (0.40)^{c}
GC	69.2 (5.9)^{ab}	11.0 (1.1)^{b}	30.2 (2.4)^{d}	4.91 (0.26)^{b}
GD	64.3 (5.3)^{a}	10.8 (1.3)^{b}	32.3 (3.5)^{d}	5.39 (0.60)^{bc}

&: Bending modulus of elasticity

Mean value (SD)

Same superscript letters denote no significant difference test between groups ($p>0.05$)

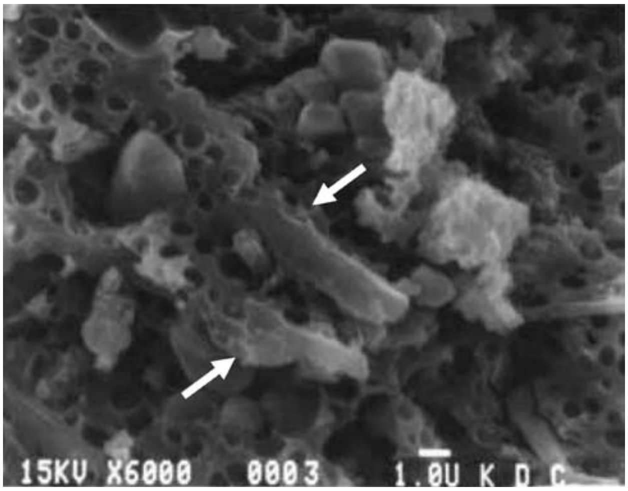

Fig. 1 SEM photograph of the fractured surface of cement specimen containing the GB whisker after diametral compression test. *: White arrows indicate the cement matrix adhering to the whisker.

cement was 5.28±0.80 MPa. The values of the four cements containing the whiskers were about two times greater than that of whisker-free cement. The compressive strength of GA was 74.1±1.5 MPa — the highest value among the four cements — was significantly different from that of whisker-free cement ($p<0.05$). On the other hand, there were no significant differences between the whisker-free cement and the other three cements, GB, GC, and GD.

SEM observation

Figure 1 shows a SEM photograph of the fractured surface of GA after diametral compression test. It was observed that the matrix of the cement adhered to the rod-like whisker, indicating high affinity between the whisker and the matrix (white arrows in Fig. 1).

DISCUSSION

Previously, researches with a view to improving the mechanical strength of polyalkenoate cements have been conducted using powders composed of different types of particles. Kerby and Bleiholder[2] reported that the mechanical properties of glass ionomer cements were reinforced by the addition of stainless steel or silver powder. In the same vein, Tsuchihashi *et al.*[3] reported that the strength of glass polyalkenoate cements was improved by about 30% with the addition of a powder composed of irregular-shaped particles. However, Walls *et al.*[4] and Irie and Nakai[5] reported that the addition of silver powder hardly improved the bending strength of glass ionomer cements.

On adding fibers to reinforce glass ionomer cements, Kobayashi *et al.*[6,7] reported that the tensile and bending strengths of glass ionomer cements with short glass fibers of CaO-P_2O_5-SiO_2-Al_2O_3 were increased by about two times as compared to the cement free of the short fibers. In this study, the strength of the cements with Alborex G® whiskers was obviously improved. On the contrary, cements containing particulate fillers of the same composition as Alborex G® whisker were not improved in diametral tensile and bending strengths[1]. It seemed that with particular filler as a reinforcement agent, improvement in the mechanical properties of glass ionomer cements was largely dependent on the shape of the added powder particles. On the other hand, a general improvement in the strength of glass ionomer cements could be discerned with the addition of whiskers.

The bending strength of the cements with whiskers increased in the order of GA<GB<GC<GD. In particular, the bending strengths of the cements with whiskers GC and GD were two times greater than that of whisker-free cement. Compressive strength was slightly improved by the addition of whisker GA, but not so with the addition of whiskers GB, GC, and GD. The diametral tensile strengths of the four cements with whiskers GA to GD were about two times greater than that of whisker-free cement, and that there were no significant differences among the four cements with whiskers.

Compressive strength was not remarkably improved in this study with the addition of whiskers. It was thus thought that the matrix of the cement, comprising the metal salt of polyacrylic acid, was the weak layer in the cement. On the other hand, the increase in the bending and diametral tensile strengths of the cements could be ascribed to the dynamic friction in the drawing process of the whiskers on bending or tensile stress. From the result of each test, it seemed that the addition of whisker GB reinforced the cement with optimum toughness. This could be ascribed to the aspect ratio of the whisker, where the aspect ratio values of GA, GB, GC, and GD calculated from the mean lengths and diameters of the whiskers were 14, 26, 13, and 12 respectively.

On the SEM photograph of the fractured surface of GB after the diametral compression test, it was observed that the matrix of the cement adhered to the rod-like whisker (white arrows in Fig. 1). Hirata *et al.*[8] reported that polyacrylic acid showed a good affinity for alumina powder surface. As Alborex G® whiskers contained alumina in the composition, it was thought that the good mechanical properties of whisker-reinforced cements might be due to the high affinity between the whisker and the matrix.

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