JOURNAL OF MATERIALS SCIENCE 25 (1990) 3269–3272

Denture base materials reinforced with glass cloths: bending property and adhesivity

J. NITANDA, H. MATSUI, Y. KASAHARA, K. WAKASA, M. YAMAKI
Hiroshima University, School of Dentistry, Department of Dental Materials, 1-2-3 Kasumi, Minami-ku, Hiroshima, 734 Japan

A. MATSUI
Aoi Dental Center, 268-1, Nakashiroyama, Oike-kudaru, Fuya-cho, Nakagyo-ku, Kyoto, 604 Japan

The reinforcement with glass cloth to a heat-curing base resin was studied using 11 kinds of glass cloths. The bending strength was improved by some glass cloths. The scanning electron microscope observation showed that the adhesivity of the interface between glass fibre and base resin was better for the composite resin containing twill weave than for some others with plain weave. The coating treatment of glass cloth tends to give the base resin a stronger bending strength than those of the composite resins composed of non-treated glass cloths.

1. Introduction

Acrylic polymethylmethacrylate as a denture base resin is significantly weak in a bending test, because the bending strength at a maximum load is below 1 000 $Kg\,cm^{-2}$ [1–4]. Some studies were attempted to improve the mechanical and bending properties of acrylic base resin, using various types of fibre reinforcement, e.g., carbon fibre, stainless steel, organic fibres and inorganic glass fibres [3–9]. At some trials small percentages of fibres were manually mixed with an acrylic base monomer before curing. In this reinforcement, enhanced with carbon fibre, the bending strength increased by 14 to 30%, compared to the acrylic base resin [7]. It is suggested that other fibres could be used for reinforcement for an application to base resin matrix [7]. The base resin mixed mechanically together with carbon fibre has been developed and made available commercially as a bone cement, showing a high strength [8]. In a reinforcement with high modulus fibre [7], a failure between the matrix and the fibre interface was observed. As the crack due to a failure of the matrix at a maximum load propagated to the reinforcing layer between them, the delay of failure would appear. Thus, the strengthening of the base resin will be done if a strong bond at a fibre-matrix interface is attained. The increase of bending properties of the fibre-reinforced denture resin was obtained in terms of a silane coupling agent to create a bond between base resin and glass fibre. As a result of the reinforcement with small percentages of large length glass fibres a larger bending strength than the acrylic base resin was obtained [3, 4]. In this study, the authors have shown that the reinforcement with various types of glass cloths increases the bending property when acrylic methylmethacrylate is used as a base resin, and the adhesivity between base resin and glass cloth was examined under the scanning electron microscopy for the composite resin reinforced with glass cloths. On the basis of the result, the relation between the bending properties and impact energy of the composite resins reinforced with coated glass cloths was discussed.

2. Materials and methods

A specimen of 3 × 10 × 65 mm dimension was prepared using heat-curing acrylic resin (Natural resin; Nissin Co., Osaka, Japan), and it was reinforced with glass cloth indicated in Table I. After a silane coupling treatment [3], two types of glass cloths were used. One was coated by the heat-curing base resin monomer and the other not coated by it. The coated and non-coated glass cloths, which were composed of non-alkali $CaO–Al_2O_3–SiO_2$ glass system containing less than 8% as an alkali, were used as a reinforcing material to the base resin (Glass cloths; NITTOBO Co., Tokyo, Japan). The specimen was made using one piece of glass cloth (9.5 × 55 mm rectangle) by setting the glass cloth along a longitudinal direction on an upper surface of the base resin with the dough

TABLE I Glass cloths used

Code	Weave	l/d	V*/V
1 WLB	Twill	10.49 × 10^3	110.0 × 10^{-3}
2 WEB	Plain	6.60 × 10^3	56.0 × 10^{-3}
3 WLC	Plain	10.69 × 10^3	30.0 × 10^{-3}
4 WG	Plain	1.34 × 10^3	28.0 × 10^{-3}
5 WEA	Plain	16.90 × 10^3	14.0 × 10^{-3}
6 KCA	Non	3.82 × 10^3	9.0 × 10^{-3}
7 KCB	Plain	2.54 × 10^3	6.0 × 10^{-3}
8 WLA	Plain	7.33 × 10^3	5.0 × 10^{-3}
9 WKB	Plain	17.88 × 10^3	4.0 × 10^{-3}
10 WF	Plain	11.76 × 10^3	3.0 × 10^{-3}
11 WKA	Plain	13.00 × 10^3	3.0 × 10^{-3}

(l/d, aspect ratio, V^*/V, volume ratio: the glass fibre within the glass cloth is represented by a length (l) and a diameter (d) of the fibre, and V^* and V mean, respectively, a volume of the glass cloth and a volume of the specimen.)

0022–2461/90 $03.00 + .12

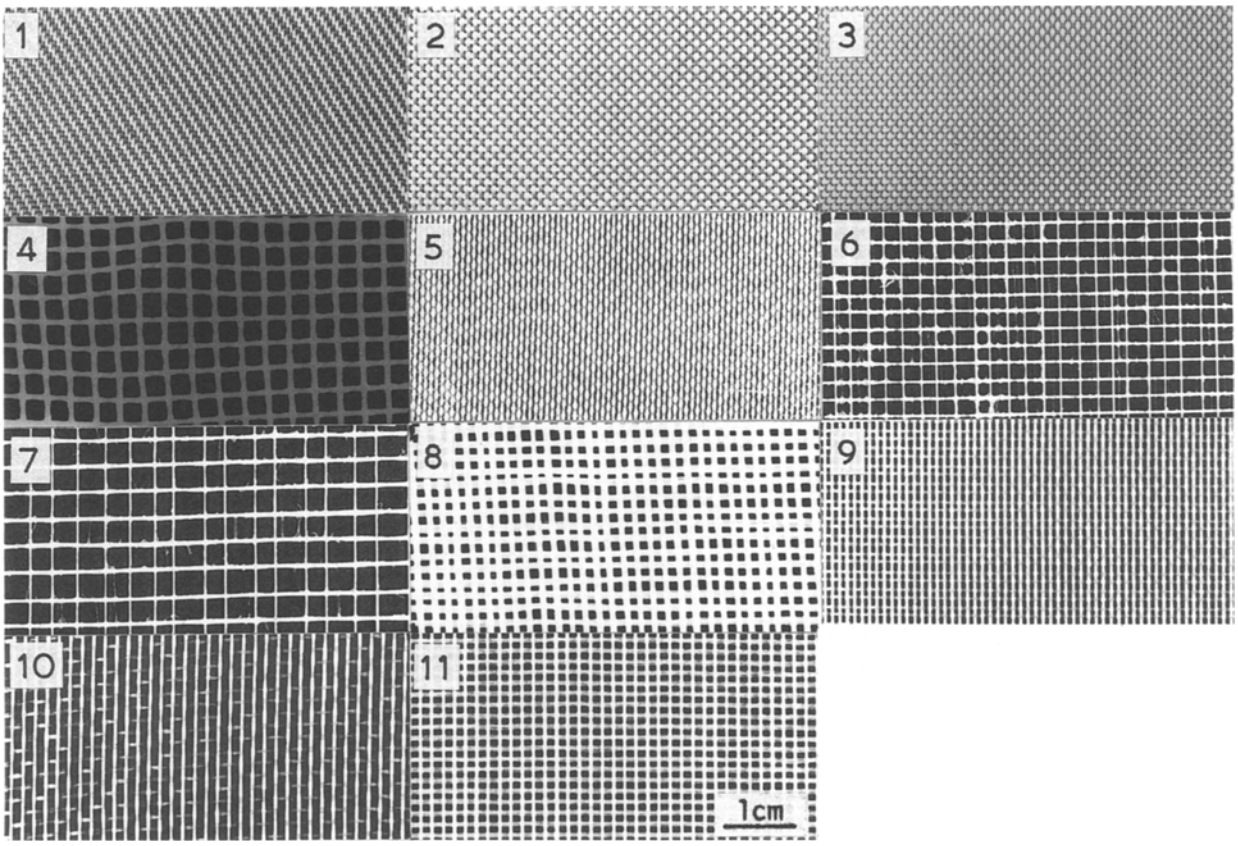

Figure 1 Glass cloths investigated for the reinforcement of the acrylic base resin. (In this study each composite resin is indicated by each code of glass cloth tested. Twill in code 1 and plain mean a glass cloth woven by twill, and plain and non mean a glass cloth without a weave).

state (thickness; 1.5 mm). After that, the base resin was added to the upper part of the specimen. In the curing, 10 g of powder polymer and 4 ml of resin monomer were mixed for heat-curing of the specimen. The glass fibre constituting glass cloth had such properties as 2.54 (specific gravity), 150 $Kg\,mm^{-2}$ (tensile strength), 7400 $Kg\,mm^{-2}$ (elastic modulus) and 4% (elongation) [4]. Before bending and impact tests, the surfaces of the bending test specimen (2.5 × 10 × 65 mm) were polished by a 1200-grit emery paper as a final polishing. The specimens were stored in a desiccator in the laboratory for one day before bending and impact tests. The bending test was performed using a Shimadzu Autograph Tensile Testing machine (Shimadzu Co., Kyoto, Japan; Model DCS-500). During a bending test a constant deformation rate was maintained as 2.0 $mm\,min^{-1}$. For the test, the load-deformation figure was recorded in the built-in X–Y recorder (DATALETTY 401; Shimadzu Co., Kyoto, Japan). In an impact test, a Charpy impact test machine was used (Japan Charpy Co., Tokyo, Japan). The impact load 30 Kg and the hammer arm length 1 m were used to obtain an absorbed impact energy. After setting an initial angle (5 degree), the change of the angle from the initial angle was measured after an impact test.

The fractography of fractured surfaces was done by scanning electron microscopy (Hitachi Co., Tokyo, Japan; Model H-430). Two planes, which were cut perpendicular to the direction of the interface between base resin and glass fibre, were observed for a fractured specimen after a bending test. One plane was the plane cut at 3 mm from the fracture surface, and the other one at 10 mm from the edge of the specimen. The adhesivity was evaluated for the both sides of near fracture plane and edge of the specimen as follows: +, no large size of void at the interface of glass fibre and base resin when observed for two planes; −, when separated interfaces when observed for two planes; ±, a symbol for when + and − were found together for the specimen.

3. Results

Both the bending test and the impact test were done for the composite resins reinforced with glass cloths which were composed of 11 types (Fig. 1). Table I indicates dimensional characteristics of glass cloths woven with different l/d and V^*/V ratios. A ratio of l/d means the size effect for the composite resins reinforced with glass cloths, and a ratio of V^*/V means the volume fraction occupied by glass cloths within the specimen. The length and diameter of glass fibre within glass cloth are indicated by l and d, and V^* and V, a volume fraction of glass cloth and a volume fraction of the specimen tested. For the composite resin reinforced with WLB glass cloth the bending strength was about 1150 $Kg\,cm^{-2}$, indicating a maximum value of the specimens (Table II). The lowest value below 900 $Kg\,cm^{-2}$ was found for the composite resin reinforced with WKB and WG glass cloths in Table I. For the base resin without glass cloth the bending properties were obtained as follows: bending strength as a maximum strength; 968.3 ± 19.7 $Kg\,cm^{-2}$, proportional limit: 554.1 ± 9.8 $Kg\,cm^{-2}$, bending elasticity 271.6 ± 2.4 $Kg\,mm^{-2}$. In Figs 2 and 3, the effect of l/d and V^*/V ratios on bending

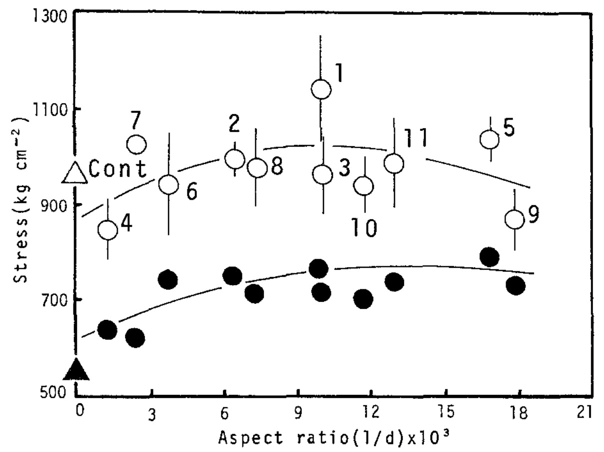

Figure 2 The change of bending strength (○) and proportional limit (●) in the composite resins containing non-coated glass cloths with respect to aspect ratio (For key, see Fig. 1).

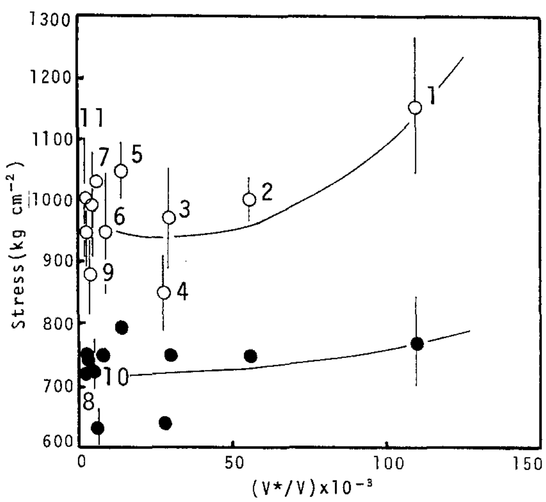

Figure 3 The change of bending strength and proportional limit in the composite resins shown in Fig. 2 with respect to volume ratio (For key, see Figs 1 and 2).

strength at a maxium load (maximum strength) and proportional limit is shown respectively. Their curves were written according to each secondary dimensional equation. The maximum values were found for the composite resin noted by WLB glass cloth with respect to l/d and V^*/V ratios.

After a bending test, the composite resin fractured was observed as shown in Figs 4a and b. Near fracture surface and near edge of the specimen, the observation at cross-section was done at the interface between base resin and glass fibre within the glass cloths. In Table III better adhesive interface indicated as + was obtained for WLB-, WEB-, WEA-, KCB-, WLA-, and WKA-reinforced composite resins than the other ones reinforced with WLC, WG, KCA and WF glass cloths which were noted as ± and −. For WEB, WEA, KCB, WLA, WKB and WKA glass cloths-reinforced composite resins, the values of maximum strength were below 1 050 Kg cm^{-2}. It is deduced that the increase of the strength is obtained by a twill weave (WLB-composite resin) rather than a plain weave.

The bending strength and impact energy of the composite resins containing coated glass cloths are shown in Fig. 5. The impact energy in the composite resin reinforced with WLB was about 1.4 times that compared with the base resin (Cont) (such composites resins as WG, KCA, KCB and WKA having lower bonding strength were not carried out in a Charpy impact test). The relationship between bending strength and impact energy showed a linear relation for the glass-reinforced composite resins when the glass cloth was coated or not coated by a resin monomer.

TABLE II Bending properties of maximum strength and bending elasticity in the composite resins reinforced with non-coated glass cloths indicated by code 1 to 11

Code	Maximum strength (kg cm^{-2})	Bending elasticity (kg mm^{-2})
1 WLB	1152.0 ± 110.4	295.5 ± 33.6
2 WEB	1000.5 ± 35.8	257.3 ± 17.9
3 WLC	970.2 ± 88.2	326.0 ± 10.0
4 WG	848.8 ± 63.9	313.7 ± 21.0
5 WEA	1049.2 ± 48.3	292.0 ± 7.5
6 KCA	945.6 ± 107.0	268.8 ± 4.2
7 KCB	1031.7 ± 12.1	283.7 ± 17.7
8 WLA	987.7 ± 86.4	272.7 ± 17.0
9 WKB	879.3 ± 67.1	283.3 ± 1.2
10 WF	948.7 ± 54.7	264.3 ± 5.7
11 WKA	995.6 ± 95.6	276.9 ± 5.0

4. Discussion

In the strengthening of the resin matrix due to reinforcing with various kinds of glass cloths, the impact energy of the reinforced composite resins did not show a large change as compared with the base resin (Cont). In containing a small amount of glass fibres, the value of bending elasticity was about 1.1 times that of the base resin because of glass fibres which were distributed at random. In addition, the increase of glass fibres led to the increase of bending strength and proportional limit of reinforced composite resin [10]. This means that the strengthening is given in terms of larger amounts of glass fibres in the base resin. Therefore, the use of glass cloths which showed a high content as indicated in Table I and II was made in this study, because the quantitative amount of glass fibres were limited for the operation of mixing. The bonding of the interface between non-coated glass fibre and base resin was tight when some glass cloths were used, showing higher bending strength than the base resin noted by Cont (Table III). In coating glass cloths the bending property was improved (Fig. 5), and it is deduced that the interface between glass fibre and base resin is better than before coating. The coating

TABLE III Adhesivity evaluated by scanning electron microscopy for the composite resins reinforced with non-coated glass cloths

Code	Weave	Adhesive
1 WLB	Twill	+
2 WEB	Plain	+
3 WLC	Plain	−
4 WG	Plain	−
5 WEA	Plain	+
6 KCA	Non	±
7 KCB	Plain	+
8 WLA	Plain	+
9 WKB	Plain	+
10 WF	Plain	±
11 WKA	Plain	+

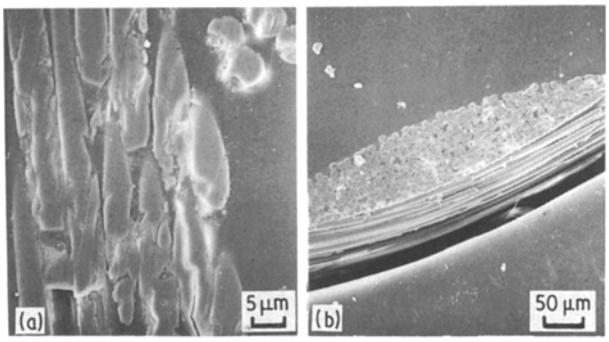

Figure 4 Scanning electron micrographs of the composite resins with non-coated glass cloths. (a) Near fracture plane for the composite resin (code 2: Glass cloth; WEB). (b) Near edge of the specimen (code 3; WLC glass cloth).

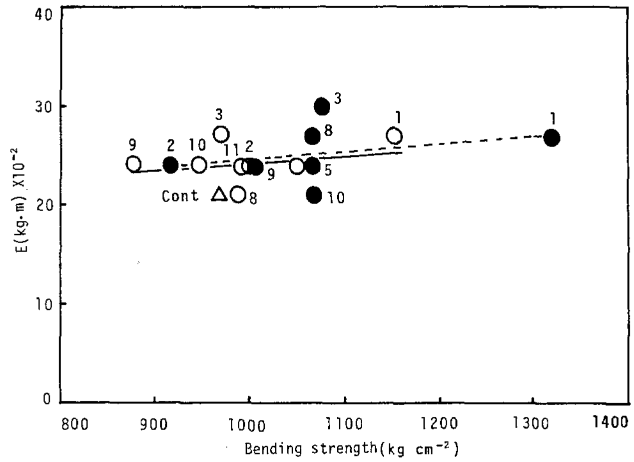

Figure 5 Relation between bending strength and impact energy for the composite resins containing coated glass cloths (●) and non-coated ones (○) (For key, see Fig. 1).

treatment thus tried to obtain high bond and good adhesivity between glass cloth and base resin (Fig. 5). Compared with the composite resins reinforced with non-coated glass cloths, the impact energy of the composite resins was almost the same value as the composite resins with coated glass cloths. On the contrary, the bending strength increased from 1 150 to 1 320 Kg cm^{-2} for the composite resin (WLB; glass cloth) as shown in Fig. 5, by coating glass cloths with a base resin monomer. If the coating treatment was done for the WLC (code 3) and WF (code 10) composite resins, the value of bending strength increased in comparison with the composite resins containing non-coated glass cloths (Fig. 5). With the exception of the composite resin containing WEB glass cloth (code 2), the other composite resins had a larger value than the base resin without glass cloth (Cont). As a result the coating treatment was effective for the increase of bending strength rather than impact energy. Thus the stronger bonding of the interface between glass fibre and base resin rather than the coating treatment in this study, is necessary for the improvement of adhesivity and the increase of their properties.

In summary the reinforcement of the methylmethacrylate base resin with glass cloth was studied by 11 different types of glass cloths. A better adhesivity between glass fibre and base resin was observed for some glass cloths. Higher bending strength than the other reinforced composite resins was found in the composite resin reinforced with twill weave described as WLB. The bending strength increased to 1 320 Kg cm^{-2} as a maximum value by coating a base resin monomer to glass cloth. As a future study the improvement of adhesivity between the interface could be done by other base resin monomers, such as bis-GMA.

References

1. A. REYTON and D. H. ANTHONY, *J. Prosthet. Dent.* **13(2)** (1963) 269.
2. J. B. WOELFEL, G. C. PAFFENBARGER and W. T. SWEENEY, *J. Amer. Dent. Assoc.* **67** (1963) 489.
3. J. NITANDA, H. MATSUI, A. MATSUI, Y. KASAHARA, K. WAKASA and M. YAMAKI, *J. Mater. Sci.* **22** (1987) 1875.
4. G. D. STANFORD and R. W. HANDLEY, *Br. Dent. J.* **125** (1968) 337.
5. S. K. SCHREIB, *Br. Dent. J.* **130** (1971) 29.
6. A. M. H. GRAVE, H. D. CHANDLER and J. F. WOLFAARDT, *Dent. Mater.* **1** (1986) 185.
7. D. L. GUTTERIDGE, *Br. Dent. J.* **164** (1988) 177.
8. S. SAHA and S. PAL, *J. Biomed. Mat. Res.* **20** (1986) 817.
9. J. NITANDA, H. MATSUI, A. MATSUI, Y. KASAHARA, K. WAKASA and M. YAMAKI, *J. Mater. Sci.* **22** (1987) 1879.

Received 20 March
and accepted 4 September 1989