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Dental application of various kinds of fibres in heat curing acrylic resin

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The effect of the type of fibres on bending behaviour and impact energy in reinforced acrylic resin was examined. Reinforcing materials such as inorganic glass fibre and cloth and organic polyester and Kevlar fibres were coated with a silane coupling treatment, whereas Co–Cr wire was directly used for the reinforcement. After silane coupling treatment each fibre was added to the heat curing base resin, except polyester fibre. It is concluded that the inclusion of glass linear fibre provides an effective improvement on plain acrylic base resin. An additional way to reinforce the resin matrix was given by the combined use of glass and Kevlar fibres.

1. Introduction

The modification of denture base acrylic resin in order to improve it [1–8] has been attempted as follows: 1. The use of grafted polymethylmethacrylate (PMMA) resins improved flexural strength; 2. Reinforcement by the addition of surface-treated carbon fibres or specially oriented carbon fibres; and 3. The application of inorganic glass fibre as a reinforcing material. As reported in [8–10], the reinforcement due to glass fibre or cloths led to the improvement of flexural properties and impact energy compared with an unreinforced PMMA. As well as silane coupling treatment of fibres, the treatment whereby the fibres were immersed into the methylmethacrylate or multifunctional monomers before curing was effective in the further improvement of their properties [8]. The use of carbon fibres as a reinforcing agent for resins has been generally practiced industrially for decades [2–4].

The results suggest that resins containing layered carbon fibres or oriented alignment of the fibres exhibit enhanced resistance to applied stress. Therefore, other types of fibres available for the improvement of denture base acrylic resin were considered. In this study a silane coupling treatment was tried to obtain better reinforcement. The purpose was to assess the effect of various silane treated fibres as a reinforcing agent on the flexural properties and impact energy, including inorganic and organic fibres.

2. Materials and methods

Bending properties and impact energy were determined as described in ADA specification No. 12 [11] and [8–10]. Specimens having a dimension of 65 mm × 10 mm × 3 mm, with various types of fibres were heat-cured within the gypsum moulds in brass denture flasks. Before testing they were polished to obtain a thickness of 2.5 mm as previously reported [9], and their conditioning was carried out as described in [9, 10]. The denture base acrylic resin was Natural resin (Liquid/Powder ratio; 10 ml/4 g, Nissin Co, Kyoto) for all samples, and five reinforcing materials used were as follows: The inorganic glass fibres (SiO_2–Al_2O_3–CaO system) with shot and linear types, supplied by NITTOBO Co (Tokyo); the organic polyester fibre by Mitsubishi Rayon Co (Linear; Ohtake); organic Kevlar 49 (Linear; Poly-*P*-benzamide) by du Pont (New York, USA) and Co–Cr orthodontic metal wire (diameter 1.0 mm × 55 mm length) by Sankin Ind (Tokyo). The reinforcing fibres used in this study had the following properties: Glass fibre (tensile strength: 3450 MPa and modulus: 72 GPa) in [12], polyester fibre (tensile strength: 980 ~ 1180 MPa, and modulus: 18 GPa) in [13, 14], and Kevlar fibre (tensile strength: 2940 MPa and modulus: 98 ~ 118 GPa) in [14]. In the case of glass shot fibres, both thermoplastic (6 mm length) and thermosetplastic fibres (3 and 6 mm) were used, similar to our study of flexural properties [15]. Maximum strength was calculated by the following equation; $3WL/2bh^2$, where W is maximal load before fracture, L distance between the supports, b and h width and thickness of the specimen. The dimension of 55 mm length was used in each linear fibre within a constant bundle. The reinforced materials for the tests were divided into two for experimental purposes, as described below. In these experiments the fibres which were treated with only a silane coupling were called silane-treated fibres or simply fibres in order to distinguish them from treated fibres with heat-curing base monomer in [8].

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2.1. Experiment I (the use of single fibre and wires)

A control, plain specimen (Cont) was a heat-curing acrylic resin without a reinforcing material, and specimens including five different types of silane-treated reinforcing materials were made. The polyester fibre was treated with the heat-curing methylmethacrylate (MMA) monomer when added to the base resin, because the fibre could not be added with only silane coupling treatment. The other fibres were not treated with MMA monomer.

2.2. Experiment II (the change of fibre pieces and combination of fibres)

These tests were done as experiment I. As the morphology of glass fibres used was not standardized because the bundle had a linear shape, pieces (2 to 8) of silane-treated fibres in the specimen were added. The reinforced compound, made manually with both fibres of glass fibre (linear) and Kevlar fibre was subjected to flexural and impact tests. Four combined fibres of 55 mm length were set with the same distance between them parallel to the longitudinal direction of the specimen. The weight of combined fibres tested in the specimen was about 160 mg, indicating that Kevlar and glass fibres were, respectively, about 40 and 120 mg.

3. Results

The results in experiment I indicate the effect of various types of silane-treated fibres on the value of impact energy (Table I). The value appeared to be comparable to that of the control heat-cured specimen, indicating that the impact energy increase in the glass fibre (shot) containing 10 wt % ranged from 1.0 to 1.2 times as compared with that in the one containing 5 wt %. Specimens of reinforced resin with 6 mm length of thermoplastic glass fibre (5 wt %) had more impact energy than those with the other shot length of fibre ($p < 0.05$; Fig. 1).

The impact energy in silane-treated glass fibre (linear)-reinforced material with different numbers of fibre is shown in Table II. The values increased with increasing pieces of glass fibres (linear). Typical curves of bending behaviour are shown in Fig. 2, indicating that the maximum strength in the glass fibre-reinforced material was more than in the plain material (Cont) and it was also more rigid than the Cont. The longitudinal alignment of fibres was applied to the other fibres and wire (Table III). The average values of impact energy were about 3.9 J, showing that the fibred specimens were stronger than the Cont specimen or the glass fibre (shot)-reinforced ones, but lower than Co–Cr wire. The metal wire with high impact energy, however, could not be added to the denture, because the design of denture is difficult to make morphology with this. Thus, the other attempts were needed for this study.

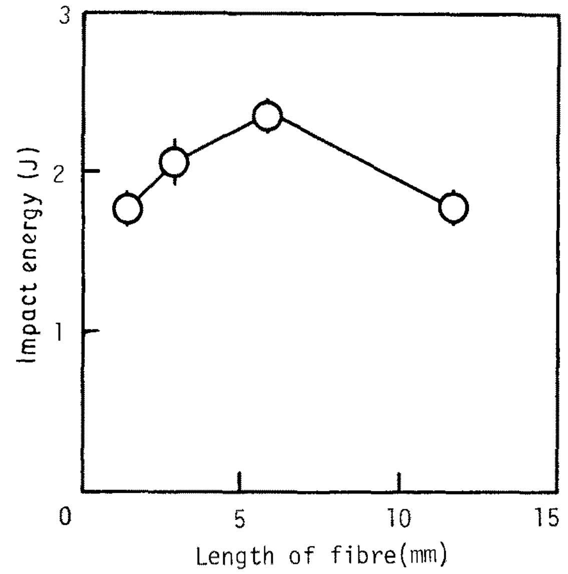

Figure 1 The values of impact energy in silane-treated glass fibre (shot)-reinforced materials which contained 5 wt %, with different lengths of thermoplastic glass (shot) to the acrylic resin.

TABLE I The value of impact energy in reinforced materials with two types of glass fibres (shot), the contents of silane-treated fibres used were 5 and 10 wt % to the plain acrylic base denoted as Cont, sample size = 5

Glass fibre	Content (wt %)	Impact energy (J)
Thermoplastic (6 mm)	5	2.4 ± 0.1
	10	2.6 ± 0.3
Thermosetplastic (3 mm)	5	2.1 ± 0.2
	10	2.4 ± 0.1
Thermosetplastic (6 mm)	5	2.1 ± 0.1
	10	2.1 ± 0.6
Cont (Unfibred)	–	2.1 ± 0.1

Data on experiment II are shown in Figs 3–5 and Table IV. The specimen reinforced with glass fibre (linear) was more rigid than the Cont specimen, and

TABLE II Impact energy in glass fibre (linear)-reinforced materials. The pieces used were 2, 4, 6 and 8 in silane-treated glass linear fibre, sample size = 5

Fibre	Pieces used	Impact energy (J)
Glass fibre (linear)	2	2.6 ± 0.2
	4	2.9 ± 0.3
	6	3.5 ± 0.6
	8	3.5 ± 0.3

TABLE III Impact energy in materials reinforced with polyester, Kevlar and Co–Cr wire. The polyester fibre was treated with heat-cured MMA monomer after a silane coupling treatment, but the latter two were not treated with MMA monomer, sample size = 5

Fibre	Treatment	Impact energy (J)
Polyester	Treated[a]	3.8 ± 0.2
Kevlar	Not-treated	4.1 ± 0.1
Co–Cr	Not-treated	5.9 ± 0.1

[a] MMA monomer coat.

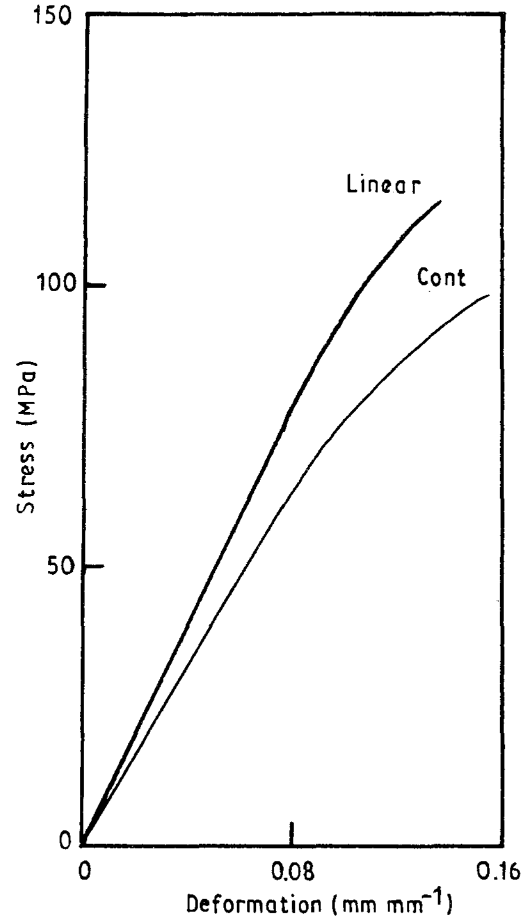

Figure 2 Typical example of the bending behaviour of a glass fibre (linear)-reinforced material (linear and Cont).

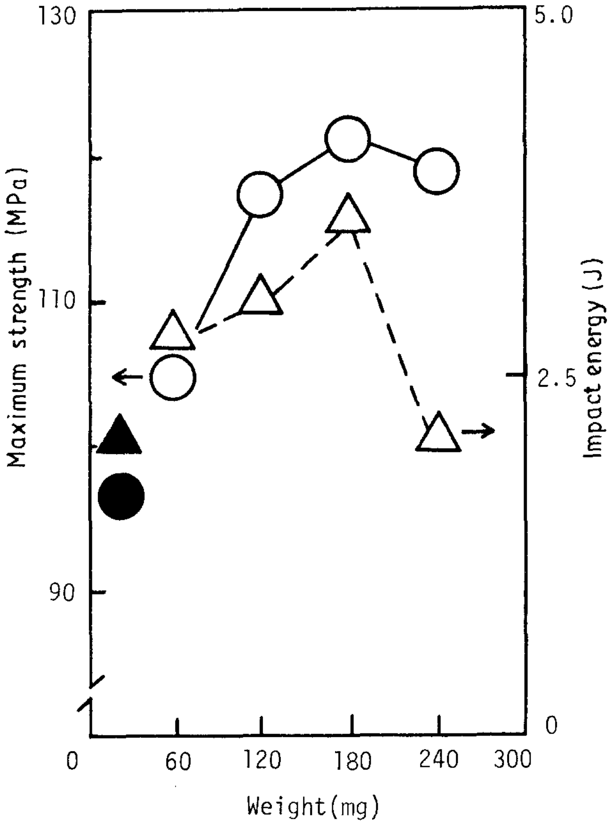

Figure 3 The average values of maximum strength and impact energy at various weights of glass fibre (linear) in the acrylic base resin: (●) maximum strength; (▲) impact energy for a Cont plain resin; (○) maximum strength and (△) impact energy for reinforced specimens.

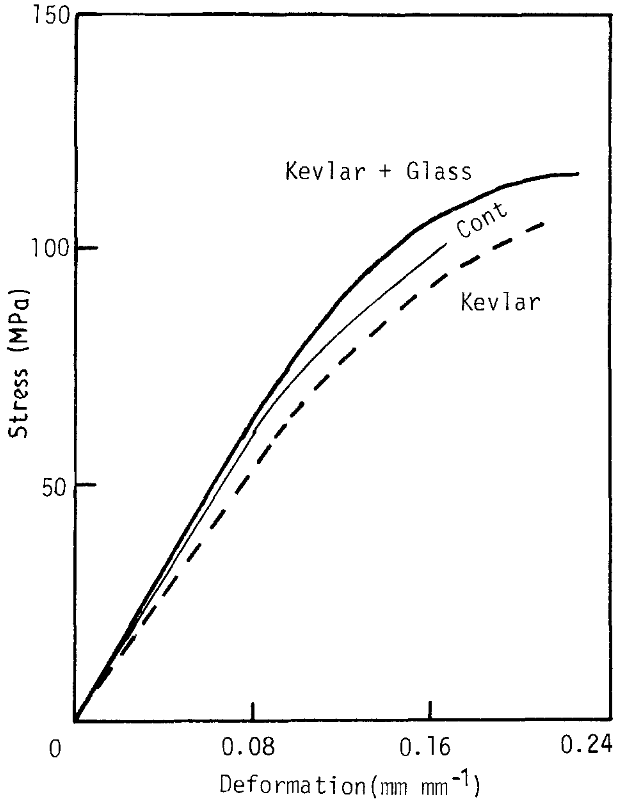

Figure 4 The bending behaviour in reinforced materials with Kevlar fibre, solely and combined Kevlar and glass fibres (linear), which are denoted as Kevlar, and Kevlar and Glass. The Cont specimen is also shown.

the increase of weight due to the addition of longitudinally oriented silane-treated fibres had the maximum values at 180 mg (Fig. 3; maximum strength and impact energy). The significant difference between unfibred resin and the fibred resin was then found ($p < 0.01$). The use of Kevlar fibre gave more ductility than that in glass fibre-oriented specimens, comparing Figs 2 and 4. The decrease in impact energy done to Kevlar fibre alone would suggest that the coating method to its fibre has to be considerable. The combination of glass fibre (linear) and Kevlar fibre resulted in the increase of the deflection value (Fig. 4), where the total weight of fibres was 160 mg. In Table IV the combined fibres appeared to have more rigidity and greater permanent deformation as compared with Cont or the other fibred resins.

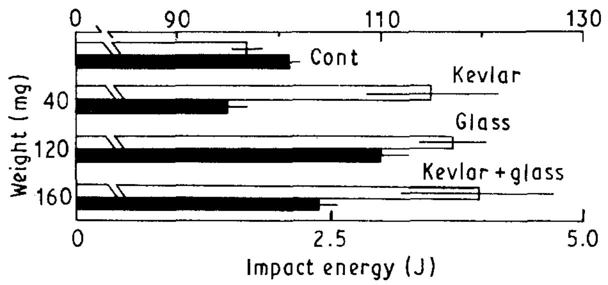

Figure 5 Maximum strength (□) and impact energy (■) in reinforced materials indicated in Fig. 4 and glass fibre (linear)-reinforced resin (each weight corresponds to that in each fibre tested).

TABLE IV Maximum strength, modulus and impact energy in materials reinforced and a Cont unfibred specimen, the silane-treated reinforcing fibres were glass, Kevlar and combined fibres of Kevlar and glass fibres with a linear type, sample size = 5

Fibre	Morphology	Maximum strength (MPa)	Modulus (GPa)	Impact energy (J)
Glass fibre	Linear	118.6 ± 4.2	2.8 ± 0.1	3.5 ± 0.6
Kevlar fibre	Linear	111.4 ± 1.4	2.7 ± 0.1	4.1 ± 0.1
Kevlar + Glass fibres	Linear (Compound)	117.2 ± 7.2	3.1 ± 0.2	2.4 ± 0.2
Cont (unfibred)	–	94.9 ± 1.9	2.7 ± 0.2	2.1 ± 0.1

4. Discussion

The fibre-reinforced resins showed an increase in maximum strength, modulus and impact energy (Table IV). The bending behaviour as shown in Figs 2 and 4 was better than that in a Cont plain specimen. On the contrary, the random dispersion of glass fibre (shot) gave an impact energy below 2.9 J as indicated in Table I and Fig. 1, which was about 1.2 times that of a Cont specimen. There was no significant difference between the reinforced resins with 5 and 10 wt % fibre contents. The reinforcement was done by the present studied factors, such as the mass fraction of fibres, the types of fibres and the combination of glass and Kevlar fibres. The increase from 5 to 10% in the weight percentage of glass (shot) fibre led to a small change in the value of impact energy (Table I), and glass fibre (linear) also gave a larger increase in the value of impact energy. In the combined fibre the reinforcement by fibres could be achieved by each fibre of glass and Kevlar fibre with a linear morphology (Table IV and Figs 3–5). The increase in impact energy means that the interfacial bonding between fibre and resin matrix became stronger than those in randomly dispersed glass fibres (shot). Then, the hand-mixing of glass (shot) would make the portions incur a void, leading to the lower value of impact energy. The shape of the fibres, such as glass (linear) and Kevlar fibres, might contribute to an additional bond strength between fibre and resin matrix. The use of combined glass (linear) and Kevlar fibres made more deformability and maximum strength than those in glass or Kevlar fibre solely (Figs 2 and 4). From these results it is deduced that the fibre-reinforced resin is not obtained with the treatment due to heat-curing monomer, but with high strength or high modulus fibres. Namely, the deformation with a bending test might relax at the interface between fibre and resin, suggesting that the fibre accepts a part of the bending deformation and the deformation is released at the interface or toward the resin base.

The result of this study suggests that the bending properties and especially impact energy are significantly improved with the combined inorganic glass fibre and organic Kevlar fibre reinforcement. It becomes evident that the bending behaviour is better than those in a Cont specimen and glass (shot) or solely used fibres. This is probably because of the improvement in the interface between fibre and resin matrix in the final product of acrylic resin. This preparation of reinforced material has an added advantage in that the fibres were set longitudinally at the centre of the heat-cured specimen. These procedures to make the denture base resin including reinforcing materials could be applied to the dental field.

References

1. D. E. SMITH, *Brit. Dent. J.* **110** (1961) 257.
2. C. K. SCHREIBER, *ibid.* **130** (1971) 29.
3. E. P. JOHNSTON, J. I. NICHOLLS and D. E. SMITH, *J. Prosthet. Dent.* **46** (1981) 478.
4. J. DE BOER, S. G. VERMILYEA and R. E. BRADY, *ibid.* **51** (1984) 119.
5. A. M. H. GRAVE, H. D. CHANDLER and J. F. WOLFAARDT, *Dent. Mater.* **1** (1985) 185.
6. H. FUKUDA and H. MIYAIRI, *Shika Zairyou Kikai* **5** (1986) 10.
7. S. HIGUTI, *ibid.* **9** (1990) 1.
8. J. NITANDA, K. WAKASA, H. MATSUI, Y. KASAHARA, M. YAMAKI and A. MATSUI, *J. Mater. Sci.* **25** (1990) 3269.
9. *Idem., ibid.* **22** (1987) 1875.
10. *Idem., ibid.* **22** (1987) 1879.
11. J. W. STANFORD, in "Guide to Dental Materials and Devices" 7th Edn (American Dental Association, Chicago, 1974) p. 203.
12. H. MAKI and A. SHIMAMURA, in "Fukugo Zairyo Gizyutsu Shuusei" (Sangyou Gizyutsu Center, Tokyo, 1976) p. 198.
13. Mitsubishi Rayon, *Bulletin No. 1* (1987).
14. T. OHNISHI, in "Shinsozai, Zairyou Shuusei" (Gizyutsu Shiryou Center, Tokyo, 1986) p. 118.
15. J. NITANDA, K. WAKASA, H. MATSUI, Y. KASAHARA, M. YAMAKI and A. MATSUI, *J. Mater. Sci. Mater. Med.* in press (1990).

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