Composite laminate veneers with a continuous inorganic phase comprising microporous sintered glass fiber networks

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Veneers were made from sheets consisting of a three-dimensional network of sintered ultrafine glass fibers. The sheets were molded by a vacuum-pressure technique and then impregnated with a liquid resin. Impregnation was performed with a method that enabled the depth of penetration to be monitored. The resin was cured with UV radiation under N_2 protection. By the use of TiO_2-containing resins the veneer achieved an enamel-like appearance. An in vitro toothbrush dentifrice abrasion test showed a high wear resistance and persisting surface luster. Scanning electron and light microscopy showed fairly smooth and flat light-reflecting glass structures in the surface. □ *Dental materials; composite resin; UV-curing*

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Fractured, badly malformed, or stained permanent incisors and canines are usually restored with crowns. Sometimes a composite resin in combination with the acid etch technique is used when an alternative therapy is desirable. Composite resins have their limitations, however, especially in the difficulty of obtaining good color matching with adjacent teeth and in deterioration due to excessive wear and/or discolorations. Conventional composites often also demonstrate a rough surface after toothbrush-dentifrice abrasion (7). Another useful therapy is to use laminate veneers together with the acid etch technique (5). Modified denture teeth (6) or preformed laminate veneers (1) of unfilled resins have been suggested for such veneers. The resistance to wear of unfilled resins is, however, low, and these veneers may therefore be severely abraded by toothbrushing.

In a previous study reinforcement of resins with a sintered glass fiber network made from ultrafine fibers was suggested (2). Particles comprising such networks have later been used as the inorganic phase in a condensable composite restorative resin (3). This composite material showed a high resistance to in vitro toothbrush-dentifrice abrasion and, compared with a conventional composite, favorable surface characteristics (4). Similar sintered networks may be used as a continuous inorganic phase for the reinforcement of laminate veneers.

The purpose of the present study was to investigate whether it was possible to make thin composite veneers, with favorable surface characteristics and high resistance to in vitro toothbrush-dentifrice abrasion, comprising single blocks of the sintered network.

Materials and methods

Production of veneers

Sheets of sintered three-dimensional glass fiber networks were made in accordance with a method described elsewhere (3). The sheets were then molded by means of a vacuum-pressure technique at approximately the same temperature as previously used for the sintering.

The molds were made by means of a master model comprising upper incisor denture

teeth (Myerson Tooth Corp, Cambridge, Mass., USA). These were mounted in acrylic resin in such a way that the facial surfaces protruded from a surrounding flat acrylic surface. A positive replica was made with a silicone impression material (Xanropren Blue®, Bayer, Leverkusen, FRG) via a dental stone negative. In the facial surface of the positive replica approximately 30 stainless steel wires (diameter, 0.25 mm; length, 30 mm) were inserted, which later would form evacuation channels. To provide a channel for a thermocouple, a wire with approximately the same diameter was placed in the flat surface close to the replicated tooth. A final negative replica was made by pouring an aluminous cement (Secar 250®, Lafarge Aluminous Cement Co., Ltd., England) over the surface without covering the free ends of the wires. The cement was laterally bordered by a wax ring and thereby acquired the shape of a circular disc (diameter, 65 mm). To avoid evaporation of water during setting, the free surface was covered with a plastic foil. After 24 h at room temperature (23 ± 2°C) the wires were removed and the cement mold dried by slowly heating it from 100 to 400°C for 96 h.

Impregnation of the molded sheets was performed with a liquid resin containing 75% wt BIS-GMA (Freeman Chem. Corp., Port Washington, Wisc., USA), 24% wt triethyleneglycol dimethacrylate, a photo-initiator (benzoin methyl ether, 0.8%) and a stabilizer (hydroquinone-monomethyl ether, 0.008%). In some experiments a pigment was also included (TiO_2, 0.001%).

For the molding experiments sintered sheets of various thicknesses and densities were used. They were placed on the flat cement surface covering the concave mold. As much fine-grained quartz sand (art. 7536, Merck, Darmstadt, FRG) as could be hosted was then poured on top (Fig. 1). The cement disc was placed on a stainless steel tray provided with a thermocouple that had contact with the sintered glass through the previously mentioned channel. The tray was then placed in a furnace with a temperature constantly held at 800°C and was kept there until the temperature of the glass, after 15 min, reached 720°C. The disc was then transferred

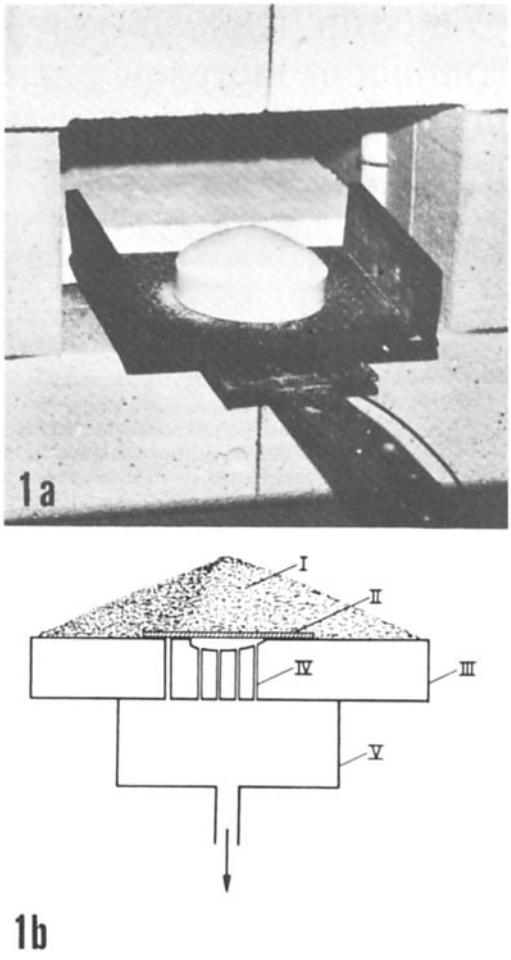

Fig. 1. Cement disc (mold) placed on tray provided with a thermocouple (a). The disc placed on a mouthpiece for evacuation (b); I = quartz sand, II = sintered sheet, III = cement disc, IV = evacuation channels, and V = mouthpiece connected to vacuum pump.

to a mouthpiece connected to a vacuum pump (Fig. 1b), which allowed a residual pressure of 0.1 atm to be momentarily established in the mouthpiece. The pressure was constant for 60 sec. Because of the reservoir of hot air in the quartz sand, the temperature, in spite of air leakage, was high enough for the few moments required to mold the sheets. Subsequent cooling by the air prevented further compaction of the sheet.

Before impregnation the molded sheet was treated with silane ('adhesion promoter') as previously described (3). Impreg-

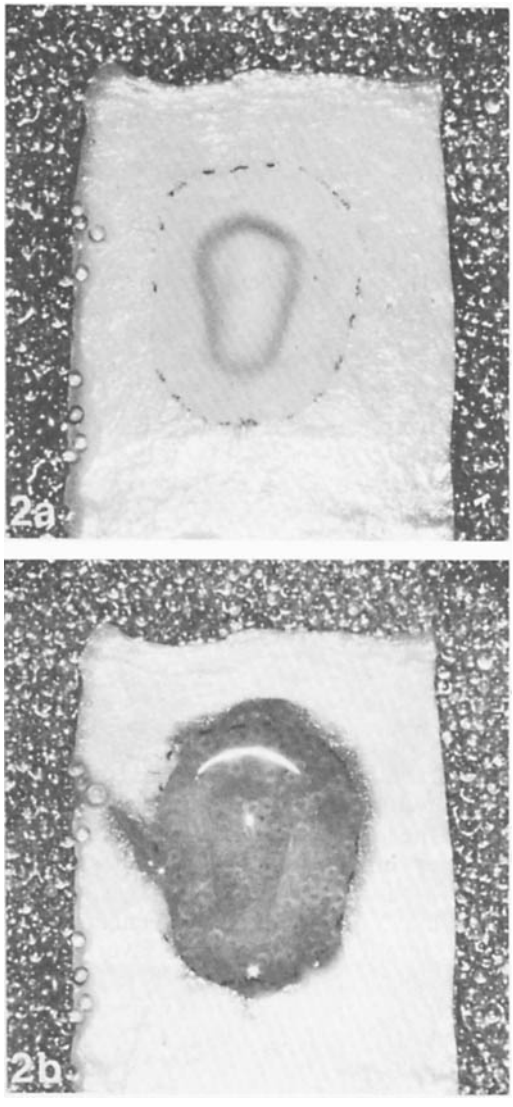

Fig. 2. Molded sheet, partly covered by a polyester film, on a bed of glass pearls (a). After impregnation (polyester film removed) with liquid resin (b).

nation was performed in a vacuum mouthpiece comprising a beaker filled to the rim with glass pearls (diameter, 1 mm). The sheet was placed on the glass pearls and covered with a thin polyester film, which also covered the rim of the beaker. When air was evacuated from the bed of glass pearls, the polyester film was firmly pressed into place, and an opening could easily be cut to expose the veneer part of the sheet (Fig. 2a).

The opening was covered by the liquid resin which then was heated *in situ* by means of a jet of hot air (65 ± 5°C) to reduce viscosity, thereby promoting rapid impregnation. Evacuation was stopped when the sheet had achieved a translucency that made the glass pearls below completely visible (Fig. 2b). Curing was effected by exposing the sheet to UV radiation (Nuva-Lite®, L. D. Caulk Co., Canada) for 60 sec on each side. During this time the sheet was held in a stream of nitrogen to prevent inhibition of polymerization due to the presence of oxygen.

Abrasion test

Resistance to toothbrush-dentifrice abrasion was determined by the use of a method described elsewhere (4). The specimens were made from veneers with an average density of the sintered glass of 1.5 g/cm^3. Heat-cured poly(methyl methacrylate) (Plexiglas, Röhm u. Haas, Darmstadt, FRG) and UV-cured unfilled resin specimens of the same monomer formulation as used for impregnation served as controls. The specimens were ground flush with the holder on carborundum paper 600 and were subsequently polished on polishing linen with an aqueous suspension of corundum powder (particle size designation, 0.3 μm).

After being polished, the specimens were brushed for 1 h in 3 dl dentifrice slurry (paste to water ratio, 1:3 by weight) by the use of a soft nylon toothbrush (TePe, Eklund & Pettersson AB, Malmö, Sweden). Brush load was 9 N, stroke length 20 mm, and stroke rate 200 strokes/min. At half of the total running time the specimen holder was rotated 180° to reduce a tendency to channeling. On this occasion the slurry was also stirred to minimize sedimentation. In addition to the previously used commercial dentifrice (Sensodyne®, Stafford-Miller Ltd., England) a standard dentifrice formulation was used. This consisted of 20% (wt) precipitated calcium carbonate (Fisher Scientific Co., USA), 2% fluor of pumice (J. Mayer Co., USA), 2% methylcellulose (A4M, Dow Chemical Co., USA), and 76% deionized water. The resulting height loss

was measured for five specimens of each kind.

The brushed surfaces were studied in the light microscope with incident light. After being coated in vacuum with an approximately 200-Å-thick layer of a palladium–gold alloy (palladium 40%, gold 60%), the same surfaces were studied with the scanning electron microscope (SEM) (Cambridge Stereoscan Mark 11:a, Cambridge Instrument Co. Ltd., England).

Table 1. Mean thickness of the sintered sheet before and after the molding procedure

Sheet		Veneer thickness (mm)
Density (g/cm^3)	Thickness (mm)	
0.3	2.0	1.0
0.3	1.0	0.5
0.3	0.35	0.15
0.5	2.0	1.2
0.5	1.0	0.7
0.5	0.35	0.22

Results

The vacuum molding process resulted in a satisfactory adaption of the sheet to the mold surface. Coarse details in the surface were reproduced but not the openings of the evacuation channels. There was a reduction of sheet thickness, especially for low initial sheet densities (Table 1). Impregnation with the liquid resin resulted in a fairly translucent material. On addition of TiO_2 pigment to the resin an enamel-like appearance was achieved. The veneers were also enamel-like with respect to brittleness and required careful handling when being trimmed by the use of rotating instruments. Partial impregnation was possible by regulating the supply of hot air. The impregnation process could thereby be monitored and interrupted at a fairly well-defined depth of penetration.

Toothbrush-dentifrice abrasion is shown in Table 2. Abrasion rates of the same magnitude were found for the two dentifrice slurries. In both cases the controls were abraded considerably more than the experimental veneer. By the use of the commercial paste, the poly(methyl methacrylate) wore at a rate approximately 17 times higher than the veneer.

The surface after toothbrushing had a glossy appearance. Light-reflecting parts seen in the microscope were identified in the SEM as glass structures in a characteristic pattern (Fig. 3).

Discussion

The vacuum pressure method made it possible to mold sheets of sintered glass and thereby to predetermine thickness and density of the molded material. With subsequent polymer impregnation it was possible to leave a thin unimpregnated layer. Such a layer may, on bonding the veneer to the tooth, retain an intermediate layer of resin by mechanical interlocking. The molding and impregnation techniques were fairly simple and may well be practiced in a dental laboratory with the necessary facilities.

In previous techniques for making veneers there seems to have been no way of combining a high resistance to toothbrush-den-

Table 2. Toothbrush-dentifrice abrasion expressed as height loss in μm/h (no. = 5)

Material	Commercial paste		Standard paste	
	$\bar{X}$	±SD	$\bar{X}$	±SD
PMMA	76.7	14.1	77.3	14.3
Experimental resin	45.4	11.2	48.4	8.4
Experimental veneer	4.6	1.9	3.0	2.1

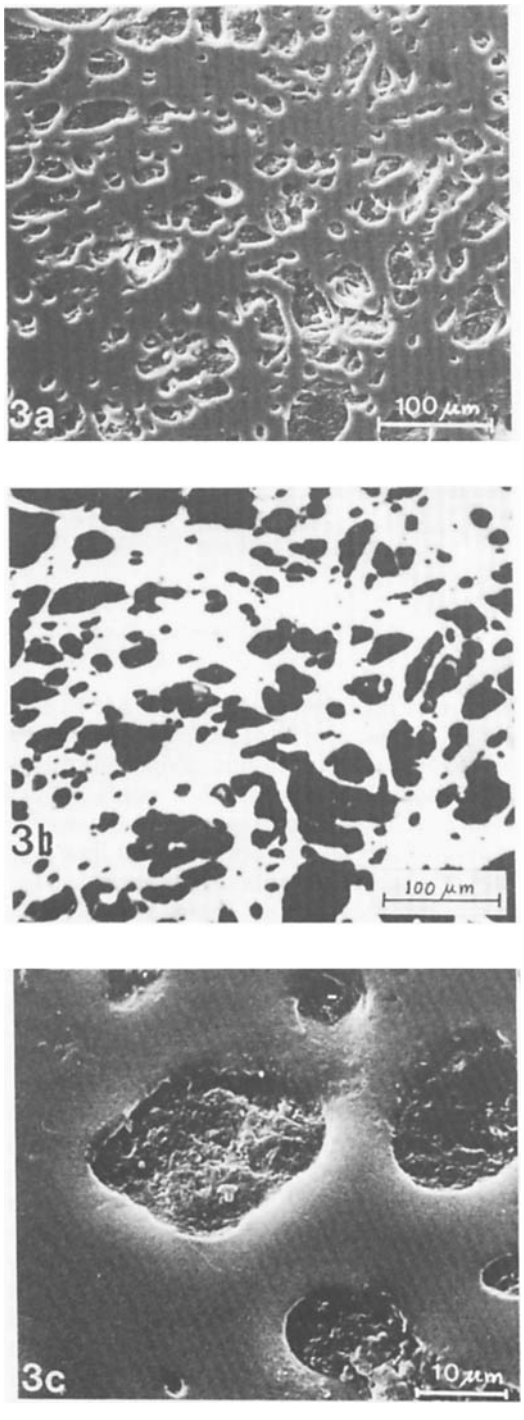

Fig. 3. Scanning electron (a and c) and light (b) micrographs, showing the same surface area of a brushed experimental veneer. Part of 3a is enlarged in c. Note level and surface appearance of the resin.

tifrice abrasion, as in regular composites, with surface smoothness and gloss, as in unfilled or 'microfilled' resins. In dental composites the inorganic filler is dispersed in an organic, continuous phase. In resins with conventional filler particles (1–100 µm) wear will expose particles in the surface which then will be rough and dull. In the experimental composite veneer the structural build-up was different, with continuous, interpenetrating, and interlocking inorganic and organic phases.

The glass exposed in the surface gave the veneer a lustrous appearance. In a previous study (4) it was found that luster was favored by the presence of large continuous areas of glass rather than the same amount of glass divided into smaller areas. The luster therefore is expectedly dependent on molding features such as initial sheet density and molding temperature.

The toothbrush-dentifrice abrasion was low in comparison with the heat-cured poly(methyl methacrylate) and the UV-cured BIS-GMA-based resin. The high wear resistance is therefore mainly ascribed to the inorganic network in the experimental veneer. The wear rate was of the same magnitude as previously found for a conventional and an experimental condensable, composite resin (4).

Summing up, it was shown that enamel-like, preformed laminate veneers can be made in the form of a composite in which the organic and inorganic phases are continuous and interpenetrating. These laminates had a high resistance to in vitro toothbrush-dentifrice abrasion and received a lustrous appearance after such wear.

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