Ceramic Whisker Reinforcement of Dental Resin Composites

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Abstract. Resin composites currently available are not suitable for use as large stress-bearing posterior restorations involving cusps due to their tendencies toward excessive fracture and wear. The glass fillers in composites provide only limited reinforcement because of the brittleness and low strength of glass. The aim of the present study was to reinforce dental resins with ceramic single-crystalline whiskers of elongated shapes that possess extremely high strength. A novel method was developed that consisted of fusing silicate glass particles onto the surfaces of individual whiskers for a two-fold benefit: (1) to facilitate silanization regardless of whisker composition; and (2) to enhance whisker retention in the matrix by providing rougher whisker surfaces. Silicon nitride whiskers, with an average diameter of 0.4 μm and length of 5 μm, were coated by the fusion of silica particles 0.04 μm in size to the whisker surface at temperatures ranging from 650°C to 1000°C. The coated whiskers were silanized and manually blended with resins by spatulation. Flexural, fracture toughness, and indentation tests were carried out for evaluation of the properties of the whisker-reinforced composites in comparison with conventional composites. A two-fold increase in strength and toughness was achieved in the whisker-reinforced composite, together with a substantially enhanced resistance to contact damage and microcracking. The highest flexural strength (195 ± 8 MPa) and fracture toughness (2.1 ± 0.3 MPa · m$^{1/2}$) occurred in a composite reinforced with a whisker-silica mixture at whisker:silica mass ratio of 2:1 fused at 800°C. To conclude, the strength, toughness, and contact damage resistance of dental resin composites can be substantially improved by reinforcement with fillers of ceramic whiskers fused with silica glass particles.

Key words: ceramic whisker, resin composite, reinforcement, whisker-glass fusion.

Introduction

Dental resin composites are generally composed of a mixture of silanized silicate filler particles with an acrylic monomer system that is polymerized to form a solid restoration. Significant improvements have been made in the microstructure and performance of resin composites, resulting in an enhanced resistance to wear and fracture (Leinfelder, 1988, 1995; Jordan and Suzuki, 1991; Bowen et al., 1992a; Mazer and Leinfelder, 1992; Ferracane and Mitchem, 1994; Suzuki et al., 1996; Lutz, 1996; Christensen, 1996; Ferracane et al., 1997). However, the toughness, strength, and durability of resin composites are still inadequate for their use to be expanded to large stress-bearing posterior restorations that involve the replacement of cusps. Composites "are not recommended for large posterior restorations because of the potential for excessive wear, microleakage or fracture" (Bayne et al., 1994). "In situations where occlusal stresses are concentrated, the current composites...are inappropriate choices," according to Corbin and Kohn (1994). Even the newly improved posterior composites are recommended only for relatively small restorations; none of the composites is accepted for unrestricted use (Wilder et al., 1996).

Considerable effort has been devoted to the improvement of resin composites. These include improved filler packing, higher filler levels, and hybrid filler phases (Cross et al., 1983; Milewski, 1987; Antonucci et al., 1991; Bowen et al., 1992b; Ferracane, 1992; Willems et al., 1992; Bayne et al., 1994; Leinfelder, 1995; Wilder et al., 1996; Anusavice, 1996). The fillers used to reinforce dental resins are mostly silicate glasses and occasionally glass-ceramics. Chopped glass fibers have also been used as fillers for dental resins (Krause et al., 1989; Willems et al., 1992). Recently, branched-fiber fillers, in which each filler particle consists of a porous network of fibers, have been incorporated into dental composites and cements (Bayne and Thompson, 1996; Thompson and Bayne, 1996). Promising results have been shown with these fillers; for example, the flexural strength of a resin composite was improved from 125 MPa to 137 MPa (Bayne and Thompson, 1996). Other studies have developed nano-porous fillers to reinforce dental resins, where the

nano-sized pores on the silica surfaces appear to enhance the interlocking of the fillers in the resin matrix and improve composite wear resistance (Luo *et al.*, 1999).

A unique approach was taken in the present study with the use of ceramic whiskers as fillers for dental resins. The ceramic whiskers are single crystals possessing a high degree of structural perfection and, as a result, strength values ~ 30 GPa (Technical Note, Marketech International, PA, 1996). For comparison, the strength of polished bulk glass is about 0.1 GPa (Lawn, 1993), and that of glass fibers is ~ 3 GPa (ASM, 1988). The fracture toughness of crystalline ceramics (silicon nitride, alumina, zirconia, etc.) ranges from about 2 to 6 MPa $\cdot$ m$^{1/2}$ (Lawn, 1993; Xu *et al.*, 1996), while that of glass is only about 0.8 MPa $\cdot$ m$^{1/2}$ (Lawn, 1993). In addition, the shape of the whiskers is highly elongated (e.g., a diameter of 0.5 μm and a length of 5 μm) with the potential benefit of being more effective in bridging a microcrack and preventing it from propagating (Lawn, 1993) and in resisting dislodgement from the matrix during wear. While extensive studies exist on fiber reinforcement of dentures and retainers (Levenson, 1986; Krause *et al.*, 1989; Altieri *et al.*, 1994; Goldberg *et al.*, 1994), the fibers differ from the whiskers in that the fibers are usually polycrystalline or amorphous, while the whiskers are single-crystalline. The strength of the whiskers is about 10 times that of the fibers, while the size of the whiskers is orders of magnitude smaller. Furthermore, the properties of a fiber composite are usually anisotropic and heterogeneous, depending on fiber size and orientation suitable for structural applications (Xu, 1993, 1994). The whisker composites have relatively isotropic and homogeneous properties and are more suitable for contact and wear applications. In the past, ceramic single-crystalline whiskers have been used to reinforce ceramics (Becher and Wei, 1984; Hirata *et al.*, 1989) and metals (ASM, 1988). Certain plastics have also been reinforced with ceramic whiskers and proposed for use in dentures (Grant and Greener, 1967), electrical applications (Robeson and Harris, 1986), golf club heads (Tominaga and Sasaki, 1987), and orthodontic brackets (Carberry and Negrych, 1992). However, a search of the literature did not reveal any reference to the use of ceramic single-crystalline whiskers for the reinforcement of dental resin composites.

The purpose of the present study, therefore, was to reinforce dental resin composites with ceramic single-crystalline whiskers. It was hypothesized that mixing the whiskers with silica glass fillers would increase the composite strength due to an improved bimodal distribution of fillers in the resin matrix. It was further hypothesized that fusing silica glass particles onto the surfaces of individual whiskers at high temperatures would further improve the reinforcement efficacy, as a result of enhancement in interlocking the roughened whiskers in the resin matrix. Three types of filler treatment were evaluated: (1) Whiskers were silanized and used as fillers for dental resins; (2) whiskers were mixed with silica glass particles for a bimodal distribution, silanized, and used as fillers; and (3) silica glass particles and whiskers were fused at high temperatures, silanized, and used as fillers. Flexural, indentation, and fracture toughness tests were conducted on the composites, together with microscopy examinations.

Materials and methods

Filler powder treatment

Single-crystalline silicon nitride (β-Si$_3$N$_4$) whiskers (UBE Industries, Ltd., Japan) had a diameter ranging from 0.1 μm to 1.5 μm, with a mean of 0.4 μm, and length ranging from 2 μm to 20 μm, with a mean of 5 μm (Hirata *et al.*, 1989). In the present study, these whiskers were treated in one of three ways prior to silanization and incorporation into dental resins.

In the first type of treatment, the whiskers were oxidized by being heated in air to form a surface layer of silicon oxide (SiO$_2$) for the facilitation of silanization. To examine the effect of temperature, we heated equal amounts of whiskers in a furnace in air for 30 min at one of the following temperatures: 150°C, 300°C, 500°C, and 800°C. The whiskers not heated were used for comparison. The whiskers were then silanized and incorporated into dental resins.

In the second type of treatment, the whiskers were heat-treated at 500°C to form a surface silicon oxide layer, then mixed with fumed silica of a particle size of 0.04 μm (Aerosil OX50, Degussa Corp., NJ). The mixing was done by dispersion of the two in ethyl alcohol to form a slurry with 100 mL alcohol *per* 1 g powder. The slurry was stirred with a magnetic stir bar, and the alcohol was slowly evaporated until dry. The dried powder was silanized and incorporated into dental resins. Three mass ratios of whisker:silica were tested: 1:2, 1:1, and 2:1. Mass ratios of whisker:silica of 0:1 and 1:0 were used for comparison.

In the third type of treatment, the fumed silica was fused onto the whiskers. The unheated whiskers were mixed with fumed silica at a whisker:silica mass ratio of 2:1 by means of the mixing methods previously described. To fuse the silica particles onto the whiskers, we divided the dried mixture into four equal parts, each heated in air for 30 min at one of the following temperatures: 650°C, 800°C, 900°C, and 1000°C. The heat-treated powders were then silanized and incorporated into dental resins.

Silanization and specimen fabrication

The filler powders from each of the three types of treatment were silanized by being mixed with 3-methacryloxypropyl-trimethoxysilane (MPTMS) in cyclohexane with n-propylamine as a catalyst in a rotary evaporator in a 90°C water bath until dry (Antonucci and Stansbury, 1990). Due to the optical opacity of the whisker composites, two-part chemically initiated curing was performed to make specimens. The silanized powders were manually blended by spatulation with a resin monomer consisting of 1 part by mass Bis-GMA and 1 part by mass TEGDMA (triethylene glycol dimethacrylate) mixed with mass fractions of 0.0005 2,6-di-tert-butyl-4-methylphenol (BHT) and 0.02 benzoyl peroxide (BPO) to form one part of a two-part chemically cured composite. The second part consisted of the same amounts of powders mixed with a resin made of 1 part by mass Bis-GMA and 1 part by mass TEGDMA and a mass fraction of 0.01 N,N-dihydroxyethyl-p-toluidine (DHEPT).

Equal masses of the two pastes were manually blended by spatulation and filled into a mold cavity of 2 mm x 2 mm x 20 mm to make a specimen. For flexural testing, six specimens were made for each condition, with a total of 30 specimens for

Table 1. Effect of whisker oxidation temperature on the properties of resin composites (mean ± SD; n = 6).

Composite Fillers	Filler Level (mass %)	Flexural Strength (MPa)[a]	Work-of-Fracture (kJ/m^2)[b]	Elastic Modulus (GPa)[c]
Whiskers (22°C)	50	90 ± 10	0.9 ± 0.1	5.5 ± 1.3
Whiskers (150°C)	50	103 ± 5	0.9 ± 0.1	6.9 ± 0.5
Whiskers (300°C)	45	117 ± 8	1.6 ± 0.2	5.2 ± 0.5
Whiskers (500°C)	43	133 ± 5	2.4 ± 0.4	5.1 ± 0.6
Whiskers (800°C)	43	122 ± 8	1.5 ± 0.2	6.2 ± 0.3
Fumed silica	50	83 ± 14	1.9 ± 0.6	2.6 ± 0.3
Barium silicate	78	109 ± 13	1.4 ± 0.3	6.0 ± 0.7
TPH	78	120 ± 16	2.4 ± 0.8	4.9 ± 0.8

[a] Flexural strengths of whisker composites with whiskers heated to 500°C or 800°C and TPH are statistically similar (Tukey's Multiple Comparison Test; family confidence coefficient = 0.95). Whiskers at 800°C or 300°C and barium silicate are similar. Whiskers at 22°C or 150°C and barium silicate are similar.
[b] Work-of-fracture of composites with whiskers heated to 500°C, fumed silica, and TPH are similar. Whiskers at 800°C or 300°C, fumed silica, barium silicate, and TPH are similar. Whiskers at 22°C and 150°C are similar.
[c] Elastic moduli of composites with whiskers heated to 22°C, 150°C, or 800°C and barium silicate are similar. Whiskers at 500°C, 300°C, or 22°C and TPH are similar.

Table 2. Effect of whisker-silica mixing on resin composite properties (mean ± SD; n = 6)

Whisker:Silica Mass Ratio	Filler Level (mass %)	Flexural Strength (MPa)[a]	Work-of-Fracture (kJ/m^2)[b]	Elastic Modulus (GPa)[c]
0:1	50	83 ± 14	1.9 ± 0.6	2.6 ± 0.3
1:2	51	131 ± 18	2.2 ± 0.6	5.3 ± 0.3
1:1	58	131 ± 17	1.9 ± 0.5	6.0 ± 0.6
2:1	53	158 ± 13	2.7 ± 0.5	6.1 ± 0.7
1:0	43	133 ± 5	2.4 ± 0.4	5.1 ± 0.6

[a] Flexural strengths of composites with whisker:silica of 1:2, 1:1 and 2:1 are statistically similar (Tukey's Multiple Comparison Test; family confidence coefficient = 0.95). Composites of ratios 1:2, 1:1, and 1:0 are also similar.
[b] Work-of-fracture of all the five composites are statistically similar.
[c] Elastic moduli of composites of ratios 1:2, 1:1, 2:1, and 1:0 are statistically similar.

the study of whisker oxidation, 18 specimens for the study of whisker-silica mixing, and 24 specimens for the study of whisker-silica fusion. For fracture toughness testing, specimens were made in the same way, but with a razor blade inserted into the mold to produce a sharp notch with a depth of 0.6 mm. Six specimens were fabricated for the composite reinforced with a whisker-silica mixture at a whisker:silica mass ratio of 2:1 fused at 800°C. Each specimen was incubated at 37°C for 15 min for chemical curing and was then demolded. The specimens were immersed in distilled water at 37°C for 24 hrs prior to being mechanically tested.

Three other dental composites were used as controls. For two of these, the silanized fillers and resin were mixed in the present study. The fillers of the first were fumed silica, mean particle size ~ 0.04 μm (Degussa Corp., NJ), and the fillers of the second were a barium silicate glass, mean particle size ~ 12 μm (Specialty Glass, Inc., FL). For these two composites, the matrix resin had the same Bis-GMA-to-TEGDMA ratio, and the specimen mold and mixing procedures were the same as those for the whisker composites. The filler levels were not kept the same; instead, a similar paste working viscosity was maintained by the same

operator. The filler levels are given in Tables 1-3. The third control was a commercial hybrid in the form of a pre-mixed paste filled with 78% by mass of glass particles ~ 0.8 μm in size, and the matrix resin was a urethane-modified Bis-GMA with TEGDMA as the diluent monomer (TPH, Caulk/-Dentsply, Inc., Milford, DE). All control specimens were cured by light (Triad 2000, Dentsply International, Inc., PA) for 1 min on each side of the specimen. For each control composite, six specimens were made for the flexural test and six for the fracture toughness test. All specimens were immersed in distilled water at 37°C for 24 hrs and then tested by the same procedures.

Mechanical testing and sample evaluation

A standard three-point flexural test (ASTM F417-78, 1984) with a span of 10 mm was used to fracture the specimens at a crosshead speed of 0.5 mm *per* min on a computer-controlled Universal Testing Machine (model 5500R, Instron Corp., Canton, MA). The following properties were evaluated: flexural strength, elastic modulus, and work-of-fracture (the energy required to fracture the specimen, obtained from the area under the load-displacement curve normalized by the specimen's cross-sectional area). Fracture toughness was measured by means of the single-edge notched-beam method (SENB, ASTM E399, 1983) in the same three-point flexural machine. A Vickers diamond indenter was used in a microhardness tester (Zwick of America, Inc., CT) to indent the specimens. A load of 10 N was used to make six indentations for each material, and the hardness value was calculated by a standard method (ASTM E384, 1991). Three materials, two conventional composites (fumed silica and barium silicate) and one whisker composite (whisker:silica = 2:1 fused at 800°C), were selected for microscopic examination of the indentations. A scanning electron microscope (SEM, JSM-5300, JEOL, Inc., MA) was used to examine the whiskers and the indentation impressions. An optical microscope with Nomarski interference contrast (Nikon Diaphot, Mager Scientific, Inc., MI) coupled with a video micrometer (model 305, Colorado Video, Inc., CO) was used for examination of the indentation impressions. One-way ANOVA was performed so that significant (α = 0.05) effects of whisker oxidation temperature, whisker:silica ratio, and whisker-silica fusion temperature could be detected. Tukey's Multiple Comparison Procedures were used at a family confidence coefficient of 0.95 for grouping and ranking the measured values.

Results

Table 1 shows that the composite filled with whiskers heated to 500°C had a flexural strength significantly higher (family confidence coefficient = 0.95; Tukey's Multiple Comparison Test) than that of those filled with whiskers heated to 300°C, 150°C, and 22°C and the composites filled with fumed silica and barium silicate, but not significantly different from that of the TPH. The work-of-fracture of the composite filled with whiskers heated to 500°C was significantly larger than that of the other composites except the TPH and the composite filled with fumed silica. The fumed silica composite had the lowest elastic modulus; those of the other materials were similar to each other. The filler levels for the barium silicate composite and the TPH were higher than those for the whisker composites.

Table 2 shows that the flexural strength of the composite reinforced with a whisker-silica mixture at a whisker:silica mass ratio of 2:1 was significantly higher (family confidence coefficient = 0.95; Tukey's Multiple Comparison Test) than that with ratios of 0:1 and 1:0, but not significantly different from that having ratios of 1:2 and 1:1. The composite elastic modulus value at 1:0 ratio was significantly smaller than the values at all the other ratios, which were not significantly different from each other. The composite filler levels at whisker:silica mass ratios of 1:1, 1:2, and 2:1 were higher than those having ratios of 0:1 and 1:0 while maintaining a similar working viscosity for the resin pastes.

In Table 3, the whisker:silica mass ratio was kept constant at 2:1. The flexural strength of the composite reinforced with the whisker-silica mixture fused at 800°C was significantly larger than that heated to 1000°C, 650°C, and 22°C, but not significantly different from that fused at 900°C (family confidence coefficient = 0.95; Tukey's Multiple Comparison Test). Compared with the control composites, the composite reinforced with the whisker-silica mixture fused at 800°C had an average flexural strength 2.3 times that of the fumed silica composite, 1.8 times that of the barium silicate composite, and 1.6 times that of the TPH composite. Microscopic examination of the fused whisker-silica powders revealed silica particles on the whisker surfaces, an example of which is shown in Fig. 1 with fumed silica glass particles (G) fused onto the silicon nitride whiskers (w) at 800°C.

The fracture toughness and hardness values are listed in Table 4 for the composite reinforced with a whisker-silica mixture at a whisker:silica mass ratio of 2:1 fused at 800°C,

Table 3. Effect of whisker-silica fusion on resin composite properties (mean ± SD; n = 6).

Whisker-Silica Fusion Temperature	Filler Level (mass %)	Flexural Strength (MPa)[a]	Work-of-Fracture (kJ/m²)[b]	Elastic Modulus (GPa)[c]
22°C (whisker-silica mixture not fused)	53	158 ± 13	2.7 ± 0.5	6.1 ± 0.7
650°C	53	158 ± 19	2.8 ± 0.8	6.2 ± 0.2
800°C	57	195 ± 8	3.9 ± 0.5	7.1 ± 0.5
900°C	53	182 ± 33	3.6 ± 1.1	6.6 ± 1.1
1000°C	53	139 ± 17	2.4 ± 0.4	5.6 ± 1.1

[a] Flexural strengths of composites with fusion temperatures of 800°C and 900°C are statistically similar (Tukey's Multiple Comparison Test; family confidence coefficient = 0.95). Composites at 900°C, 650°C, and 22°C are also similar.

[b] Work-of-fracture of composites at 650°C, 800°C, and 900°C are similar. Composites at 22°C, 650°C, 900°C, and 1000°C are similar.

[c] Elastic moduli of composites at 800°C, 900°C, and 22°C are similar. Composites at 1000°C, 900°C, 650°C, and 22°C are similar.

Table 4. Fracture toughness and hardness of resin composites (mean ± SD; n = 6)

Composite	Whisker Composite	Fumed Silica	Barium Silicate	TPH
Fracture toughness (MPa · m$^{1/2}$)[a]	2.1 ± 0.3	0.9 ± 0.1	1.3 ± 0.1	1.4 ± 0.2
Vickers hardness (GPa)[b]	0.66 ± 0.03	0.44 ± 0.01	0.68 ± 0.04	0.65 ± 0.02

[a] Fracture toughnesses of barium silicate and TPH composites are statistically similar (Tukey's Multiple Comparison Test; family confidence coefficient = 0.95).

[b] Vickers hardnesses of whisker composite, barium silicate, and TPH are similar.

in comparison with values of the controls. The fracture toughness of the whisker composite was significantly higher than that of all the controls (family confidence coefficient = 0.95; Tukey's Multiple Comparison Test). The hardness of the whisker composite was significantly higher than that of
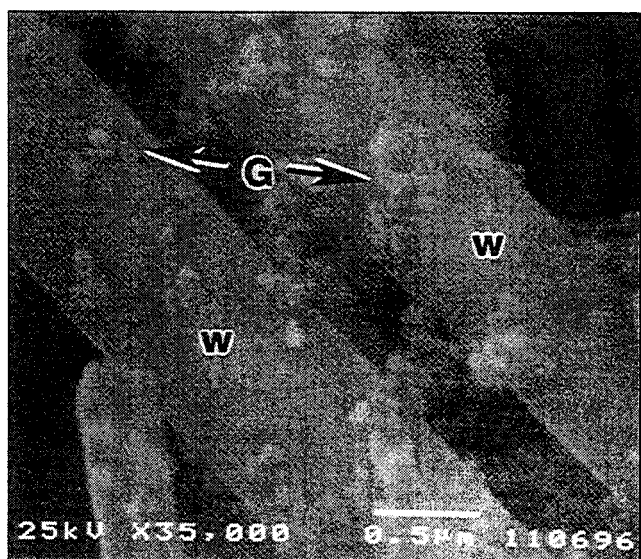

Figure 1. SEM micrograph of fumed silica glass particles "G" fused onto the silicon nitride whiskers "w" by being heated at a temperature of 800°C.
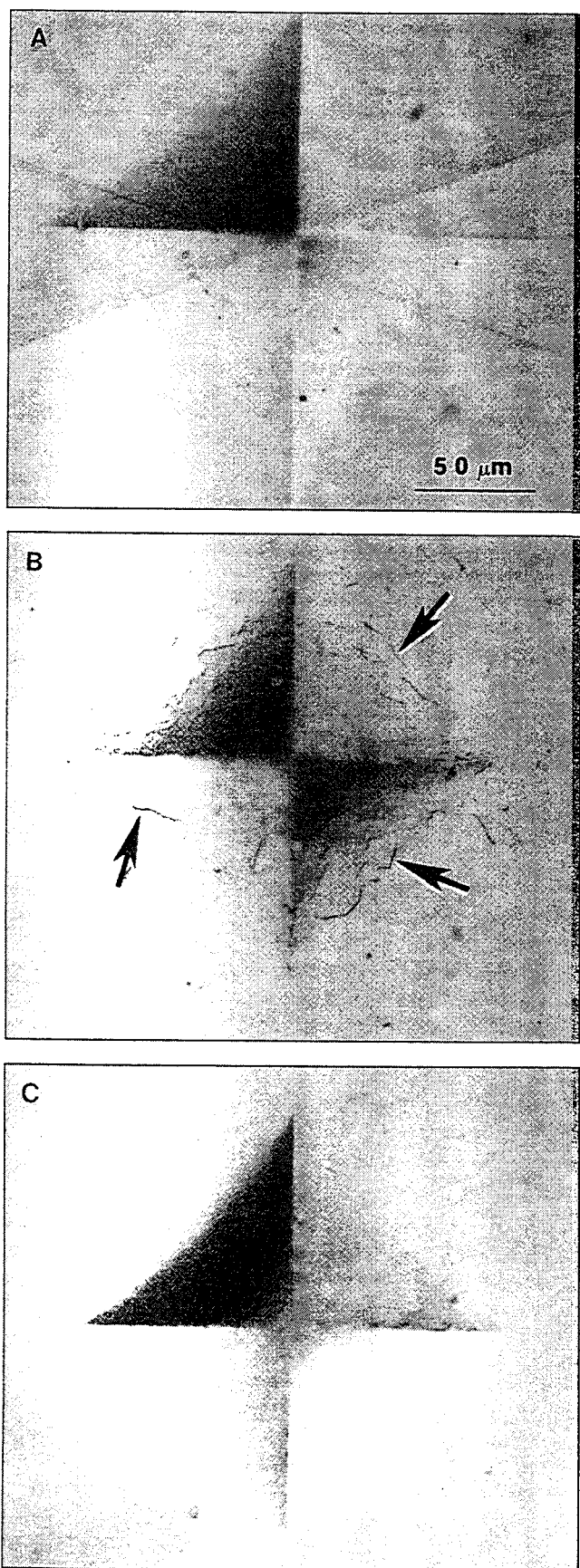
the fumed silica composite, but not significantly different from that of the barium silicate and the TPH.

Optical micrographs of typical indentation impressions are shown in Fig. 2 for three materials: (A) the fumed silica composite; (B) the barium silicate composite; and (C) the composite filled with a whisker-silica mixture at a whisker:silica mass ratio of 2:1 fused at 800°C. The whisker composite had a white-grey color and was relatively opaque. The fumed silica composite showed a large impression, and hence a low hardness. Numerous indentation-induced microcracks were present in the barium silicate composite (arrows in Fig. 2B). SEM examination revealed that these cracks either cut through the glass particles (crack I, Fig. 3) or propagated in the resin matrix around the glass particles (crack II, Fig. 3). No cracks could be found in the whisker composite (Fig. 2C) and the fumed silica composite, as confirmed by SEM examination at magnifications up to 30,000x.

Discussion

The three types of filler treatment proposed in this study progressively enhanced the composite properties. Compared with unheated whiskers, heating the whiskers to 500°C prior to silanization significantly improved the composite mechanical properties. This improvement was probably a result of silicon oxide formation on the surfaces of silicon nitride whiskers which enhanced the whisker bonding to the silane and hence to the matrix resin. Previous studies (Andrews and Riley, 1989; Hirata *et al.*, 1989) found that heat treatment produced a thin film of silicon oxide on the silicon nitride surface. Smaller flexural strength values at temperatures ≤ 300°C were probably due to insufficient silicon oxide formation on the whiskers. Further studies are required if we are to understand the silicon oxide formation mechanism and if the heat-treatment process for the whiskers is to be optimized.

Whisker entanglement is a commonly encountered problem in composite manufacturing (Hood and Pickers, 1984; Hirata *et al.*, 1989). Mixing the whiskers with silica particles has the potential benefit of (1) dispersing the whiskers from each other to minimize whisker entanglement and (2) forming a bimodal distribution with improved filler packing. A whisker:silica mass ratio of 2:1 not only significantly improved the composite mechanical properties, but also increased the filler level from 43% to 53%, which might have reduced the composite polymerization shrinkage. Further studies should address the optimization of whisker:glass ratio and glass particle size, and the effect of whisker-glass bimodal distribution on polymerization shrinkage.

A key challenge in whisker reinforcement is improving

the whisker-matrix bonding. Fusing silicate glass particles onto the individual whiskers has the benefits of (1) facilitation of silanization regardless of the whisker composition (*e.g.*, sapphire, zirconia, mullite); and (2) enhancing whisker retention in the matrix by providing rougher whisker surfaces. Fusing silica particles onto silicon nitride whiskers at 800°C significantly improved the composite mechanical properties. Fusion at 650°C did not improve the composite properties, perhaps due to insufficient softening of the silica particles. The composite properties became inferior at 1000°C, probably a result of whisker degradation at this temperature. The mechanisms of whisker-glass fusion are worthy of further exploration.

When probed by indentation, a material susceptible to contact damage and wear usually shows a big indentation impression due to its low hardness, and/or microcracking because of its brittleness (Lawn, 1993). The fumed silica composite showed a large impression. The barium silicate composite exhibited cracks that either cut through the glass fillers or propagated around the filler particles, where the reinforcing effect of the filler was lost. In contrast, the whisker composite exhibited no cracks. The whiskers possess extremely high strength and toughness and are less likely to be cut through by cracks. The indentation test suggested an enhanced resistance of the whisker composite to contact damage and wear. Further studies are needed to examine the actual wear characteristics, the abrasiveness of the composite to enamel, and, if needed, the veneering of the whisker composites with conventional composites (Xu and Eichmiller, 1997).

The silicon nitride whisker-reinforced composites were relatively opaque, with a white-grey color. Future improvements may be made on the composite translucency by the use or development of single-crystalline whiskers and dental resins with matched refractive indices. This pilot study showed the use of whisker reinforcement for dental composites only. The methods for bonding particulate fillers onto the elongated fillers to control interfacial chemistry and to improve the filler retention in the matrix should be applicable to other resin and resin-polyacid matrices, as well as to metal matrices and ceramic matrices. The elongated fillers can be single-crystalline ceramic whiskers as well as chopped fibers of ceramics, glass, polymers, and metals. The particulate fillers can be glass, ion-releasing fluorosilicate glass, and various types of ceramics, metals, and polymers. The bonding of particulate fillers onto the elongated fillers can be achieved by temperature-assisted fusion as well as by other techniques of bonding, cementing, chemical reaction, sputtering, and deposition (Xu and Eichmiller, 1997).

This pilot study has demonstrated the promise of single-crystalline ceramic whisker reinforcement for improving the mechanical properties of dental resin composites. The clinical performance of a composite is a complicated issue that involves not only the mechanical properties of the composite, but also factors such as polymerization shrinkage, isolation during placement, and wear of composite and opposing enamel. The strength and toughness appear to be a controlling factor, since the currently available resin composites are not recommended for use in large stress-bearing posterior applications, especially those that involve
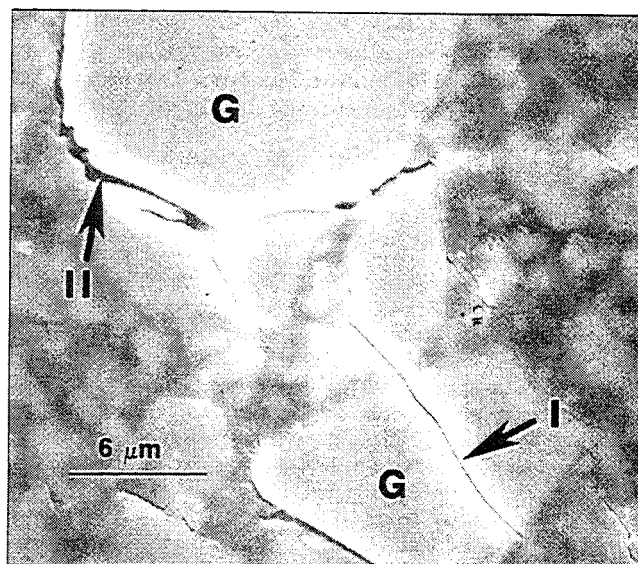

Figure 3. SEM micrograph showing failure mechanisms in the conventional barium silicate resin composite: crack I that cut through the glass filler particle (G) and crack II that propagated around the round-shaped filler particle. In both cases, the reinforcing effect of the filler is lost.

cusp replacement. In the present study, dental composites reinforced with silica particle-fused silicon nitride whiskers possessed flexural strength and fracture toughness values approximately 1.6 to 2.3 times those of conventional composites reinforced with glass particles only. Other properties—including elastic modulus, work-of-fracture, and resistance to contact damage and microcracking—were also significantly improved. Preliminary wear studies with composite pin against enamel flat in three-body wear showed that the wear of a whisker composite with a filler mass fraction of 55% and the opposing enamel was comparable with that of a hybrid composite. Ongoing studies showed that the roughness of clinically polished surfaces of the whisker composite was similar to that of a hybrid composite, likely a result of the small size of the whiskers (Xu *et al.*, 1996). The reinforcing methods developed in the present study are expected to be applicable to other resin-, ceramic-, and metal-based composites for biomedical and engineering applications.

Acknowledgments

Disclaimer

Certain commercial materials and equipment are identified in this paper to specify the experimental procedure. In no

instance does such identification imply recommendation or endorsement by the National Institute of Standards and Technology or the ADA Health Foundation or that the material or equipment identified is necessarily the best available for the purpose.

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