

H.H. K. Xu\*, J.B. Quinn,  
and A.A. Giuseppetti

Paffenbarger Research Center, American Dental Association Foundation, National Institute of Standards and Technology, Building 224, Room A-153, Stop 8546, Gaithersburg, MD 20899-8546, USA; \*corresponding author, hockin.xu@nist.gov

*J Dent Res* 83(12):930-935, 2004

## ABSTRACT

Resin composites must be improved if they are to overcome the high failure rates in large stress-bearing posterior restorations. This study aimed to improve wear resistance *via* nano-silica-fused whiskers. It was hypothesized that nano-silica-fused whiskers would significantly improve composite mechanical properties and wear resistance. Nano-silicas were fused onto whiskers and incorporated into a resin at mass fractions of 0%-74%. Fracture toughness (mean  $\pm$  SD;  $n = 6$ ) was  $2.92 \pm 0.14$  MPa·m<sup>1/2</sup> for whisker composite with 74% fillers, higher than  $1.13 \pm 0.19$  MPa·m<sup>1/2</sup> for a prosthetic control, and  $0.95 \pm 0.11$  MPa·m<sup>1/2</sup> for an inlay/onlay control (Tukey's at 0.95). A whisker composite with 74% fillers had a wear depth of  $77.7 \pm 6.9$   $\mu$ m, less than  $118.0 \pm 23.8$   $\mu$ m of an inlay/onlay control, and  $172.5 \pm 15.4$   $\mu$ m of a prosthetic control ( $p < 0.05$ ). Linear correlations were established between wear and hardness, modulus, strength, and toughness, with  $R = 0.95-0.97$ . Novel nano-silica-fused whisker composites possessed high toughness and wear resistance with smooth worn surfaces, and may be useful in large stress-bearing restorations.

**KEY WORDS:** dental resin composite, nano-silica-fused whiskers, wear, mechanical properties, mechanisms.

# Wear and Mechanical Properties of Nano-silica-fused Whisker Composites

## INTRODUCTION

The performance of a dental composite depends on filler type, resin composition, filler-matrix bonding, and cure conditions (Wendt, 1987; Pallav *et al.*, 1989; Ferracane *et al.*, 1998; Watts and Hindi, 1999; Lim *et al.*, 2002). Short fibers (Krause *et al.*, 1989) and networked fibers (Ruddell *et al.*, 2002) have been used to reinforce composites, resulting in modest strength increases. The degree of polymerization conversion has been increased *via* heat-treatment, leading to moderate strength increases (Loza-Herrero *et al.*, 1998). Further improvements are needed for composites to overcome brittle fracture and high failure rates in large stress-bearing restorations (Tyas, 1990; Christensen, 1999; Donly *et al.*, 1999), especially those that involve the replacement of tooth cusps.

Recently, nano-silica-fused whiskers were incorporated into resins, resulting in strength increases (Xu, 2000; Xu *et al.*, 2000). Silica was fused onto whiskers to enhance silanization and retention in the resin by roughening the whisker surfaces. Besides strength, occlusal wear resistance is also a major requirement for the longevity of restorations (Pallav *et al.*, 1989; Bayne *et al.*, 1992; Peutzfeldt and Asmussen, 1992; Wassell *et al.*, 1994; Leinfelder and Suzuki, 1999; Manhart *et al.*, 2000). Wear tests have been developed to simulate *in vivo* wear (DeLong *et al.*, 1985; Sakaguchi *et al.*, 1986; Suzuki *et al.*, 1996; Lim *et al.*, 2002). Three-body wear with artificial food slurries produced data that corresponded well with clinical results (de Gee *et al.*, 1996; Condon and Ferracane, 1997; Leinfelder and Suzuki, 1999; Xu *et al.*, 1999). Filler levels, filler treatments, and degrees of cure have been shown to influence wear (Condon and Ferracane, 1997; Lim *et al.*, 2002). Fine fillers have been used to reduce inter-particle spacing to improve wear resistance (Pallav *et al.*, 1989; Bayne *et al.*, 1992).

The aim of this study was to investigate the effects of nano-silica-fused whisker filler level on composite wear, and to examine the relationships between wear and mechanical properties. It was hypothesized that increasing nano-silica-fused whisker fillers would increase the composite wear resistance, and that wear rate would decrease when composite mechanical properties (hardness, elastic modulus, flexural strength, and fracture toughness) were increased.

## MATERIALS & METHODS

### Specimen Fabrication

Nano-silica (Degussa, Ridgefield, NJ, USA) having particles of 60 nm to 120 nm (mean = 80 nm) was used. Silicon nitride whiskers (UBE, New York, NY, USA), with diameters ranging from 0.1  $\mu$ m to 2  $\mu$ m (mean = 0.4  $\mu$ m) and lengths from 2  $\mu$ m to 30  $\mu$ m (mean = 5  $\mu$ m), were used. Whiskers were mixed with silica at a whisker:silica mass ratio = 2:1 by being stirred in ethyl alcohol on a hot plate until dry. The mixed powder was heated in a furnace at 800°C for 30 min to fuse the nano-silica onto the whiskers (Xu, 2000). The powder was

Received July 28, 2003; Last revision September 24, 2004;  
Accepted September 28, 2004

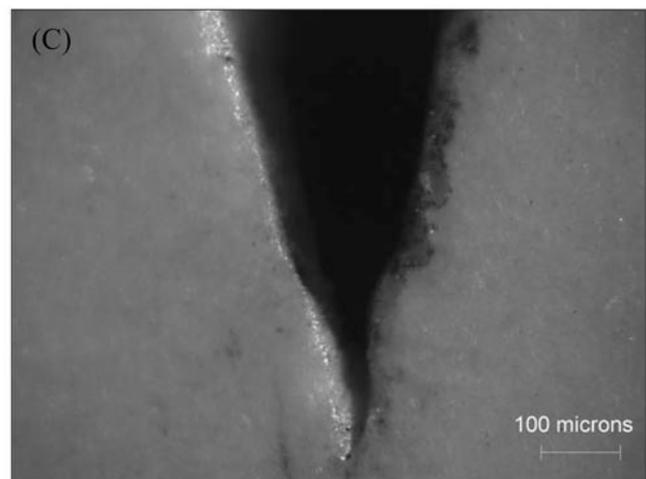
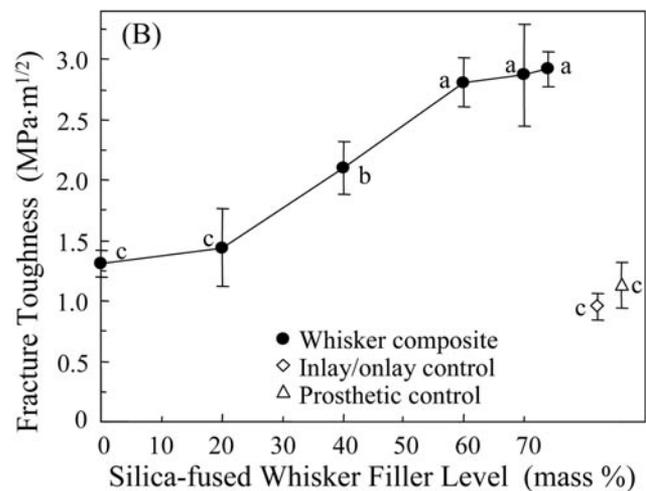
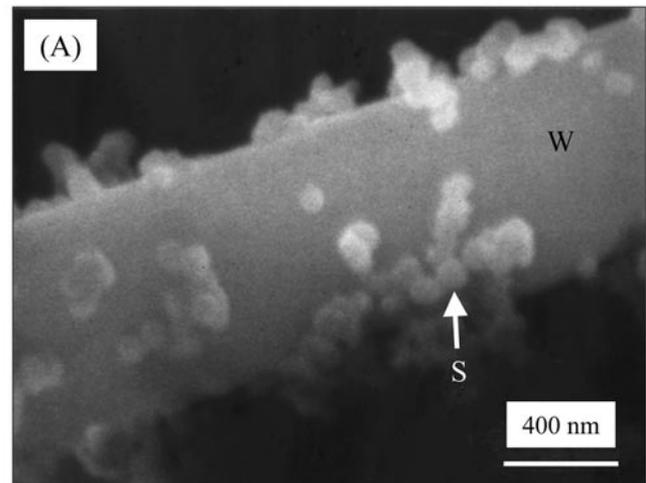
silanized with mass fractions of 4% 3-methacryloxypropyltrimethoxysilane and 2% n-propylamine in cyclohexane in a rotary evaporator. The silanized powder was mixed with a resin monomer of mass fractions of 48.965% of an oligomeric urethane derivative of Bis-GMA (Caulk/Dentsply, Milford, DE, USA), 48.965% triethylene glycol dimethacrylate (TEGDMA), 2% benzoyl peroxide, and 0.07% 4-methoxyphenol. Filler mass fractions (%) of 0, 20, 40, 60, 70, and 74 were used following recommendations in previous studies (Xu, 2000; Xu *et al.*, 2000). A previous study showed that composite with 70% and 74% fillers possessed high strengths, while the paste with 79% fillers was dry and the specimens had a lower strength (Xu, 2000). For fracture toughness, the paste was placed into steel molds of  $2 \times 2 \times 25 \text{ mm}^3$  and heat-cured at  $120^\circ\text{C}$  for 30 min for indirect applications, because the specimens were too opaque to be light-cured. For wear-testing, the paste was placed in molds of 4 mm diameter and 3 mm depth and cured in the same manner.

An indirect inlay/onlay composite (Concept, Ivoclar, Amherst, NY, USA), referred to as inlay/onlay control, was cured in the Concept Heat-Integrated Processor at  $120^\circ\text{C}$  for 10 min under a pressure of 0.6 MPa. Concept consisted of 53-56% mass fraction of 40 nm silica and 20% radiopaque fillers, for a total of 73-76% in a urethanedimethacrylate resin. An indirect prosthetic composite (Artglass, Heraeus Kulzer GmbH, Wehrheim, Germany), referred to as prosthetic control, was cured in a Dentacolor-XS photo-curing unit for 90 sec. Artglass contained 70% barium-aluminum-silicate (mean particle size =  $1 \mu\text{m}$ ) in a resin with tetra- and hexa-functional groups and conventional bifunctional methacrylates.

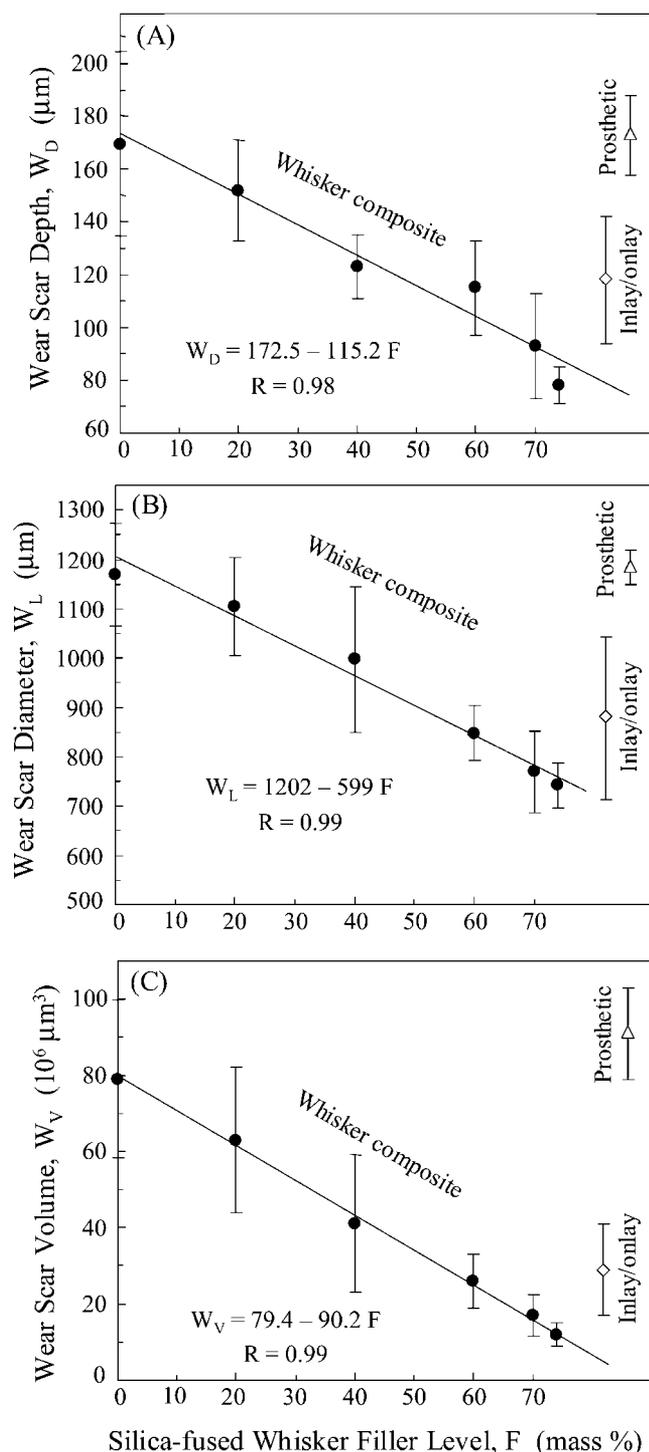
### Testing

Fracture toughness was measured by the use of a single-edge-V-notched beam method that has been extensively studied in a 'round robin' commissioned by the Versailles Project on Advanced Materials and Standards (VAMAS) and is currently an ISO draft (Kübler, 1999). This method has been used to measure the fracture toughness of dental materials (Scherrer *et al.*, 1998; Quinn *et al.*, 2003). A notch depth of approximately  $500 \mu\text{m}$  was machined into a specimen by means of a  $150\text{-}\mu\text{m}$ -thick diamond blade. Diamond paste of  $3 \mu\text{m}$  was placed into the notch tip, and a sharp blade was used to cut the notch further to a total depth of  $700\text{-}800 \mu\text{m}$  with a relatively sharp notch tip. We cut 5 specimens simultaneously by mounting bars side by side, sandwiched between 2 bars of the same material (dummy bars). We used the dummy bars to avoid chipping at groove entry and exit points, and to aid in maintaining an even notch depth (Kübler, 1999). It took several hours of alternating cutting and checking under an optical microscope for each group of specimens. A new blade was used for each specimen group. The sharpness of the notch was deemed sufficient when the tip was less than  $20 \mu\text{m}$  in diameter (Kübler, 1999). For each specimen, the notch length was measured on both sides and averaged. The notched specimen was fractured on a computer-controlled Universal Testing Machine (model 5500R, Instron, Canton, MA, USA) in three-point flexure with a 10-mm span at 1 mm/min cross-head speed (Xu, 2000). Forty-eight specimens were tested for the 6 filler levels and 2 controls with 6 repeats each.

Wear specimens were tested in a four-station apparatus (Caulk/Dentsply, Milford, DE, USA) (Suzuki *et al.*, 1996; Xu *et al.*, 1999). Each specimen was surrounded by a brass ring filled with a water slurry, 63% of which was comprised of polymethyl methacrylate (PMMA) beads (mean particle size =  $44 \mu\text{m}$ ). A carbide steel pin with 3-mm tip diameter was loaded onto the



**Figure 1.** Nano-silica-fused whisker, fracture toughness, and a notch. In (A), the nano-silica particles (S) are indicated by the arrow, and 'W' designates the whisker. In (B), each value is the mean of 6 measurements, with the error bar showing 1 standard deviation (SD) (mean  $\pm$  SD;  $n = 6$ ). Dissimilar letters indicate values that are significantly different (Tukey's multiple-comparison test; family confidence coefficient = 0.95). In (C), optical micrograph shows an example of a notch in unfilled resin showing the sharpness of the notch tip produced by the single-edge-V-notched beam method.



**Figure 2.** Composite wear rates vs. nano-silica-fused whisker filler level. (A) Wear scar depth  $W_D$ , (B) wear scar diameter  $W_L$ , and (C) wear scar volume  $W_V$ . Data for the prosthetic and inlay/onlay composite controls were included in each plot near the right axis. Each value is the mean of 6 measurements, with the error bar showing 1 standard deviation (mean  $\pm$  SD;  $n = 6$ ). The line through the data for the whisker composites was the linear best fit with the equation and the correlation coefficient  $R$  shown in each plot.

specimen submerged in the PMMA slurry. The pin was pressed down against the PMMA particles on the specimen and rotated 30°. Upon reaching a maximum load of 76 N, the pin was counter-

rotated during unloading and moved upward back to its original position. Each specimen was subjected to 400,000 wear cycles. Forty-eight specimens were tested for the 6 filler levels and 2 controls with 6 repeats. The sizes and depths of the wear scars were measured with the use of a computer-controlled profilometer (Mahr, Cincinnati, OH, USA) with a 5- $\mu\text{m}$  diamond stylus. For each wear scar, profilometric tracings were made at intervals of 50  $\mu\text{m}$  in 2 directions perpendicular to each other, with the unworn surface as baseline. The maximum values in the 2 perpendicular directions were averaged to yield the maximum depth and diameter for each wear scar (Xu *et al.*, 1999).

The worn specimens were gold-coated and observed in a scanning electron microscope (SEM, JSM-5300, JEOL, Peabody, MA, USA). The data were analyzed by one-way ANOVA and Tukey's Multiple Comparison procedures ( $\alpha = 0.05$ ).

## RESULTS

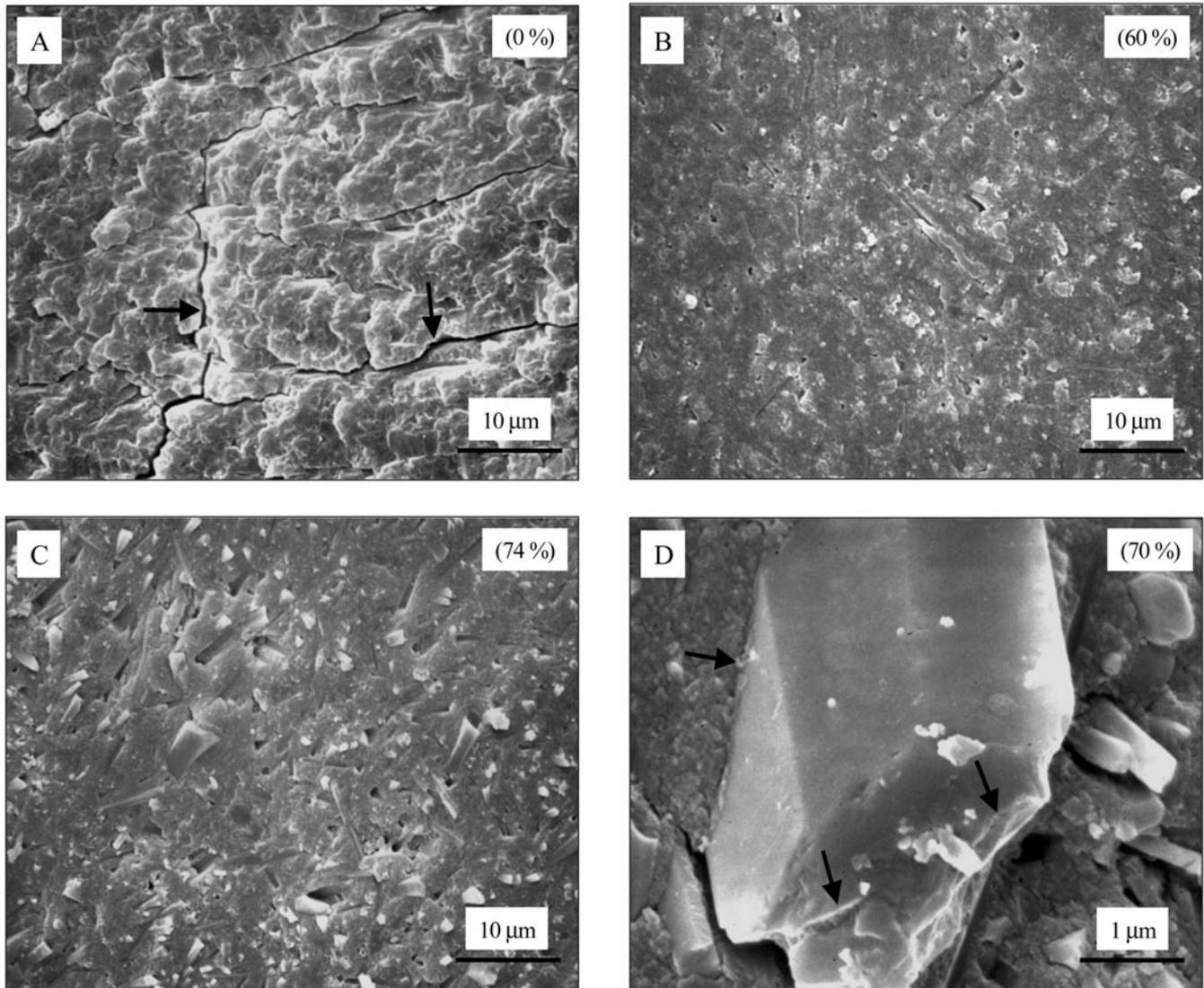
Nano-sized silica particles were fused onto the whiskers at 800°C (Fig. 1A). Fracture toughness increased with filler level (Fig. 1B). Fracture toughness (mean  $\pm$  SD;  $n = 6$ ) at 74% silica-fused whiskers was  $(2.92 \pm 0.14)$  MPa·m<sup>1/2</sup>, significantly higher than those from 0% to 40%,  $1.13 \pm 0.19$  MPa·m<sup>1/2</sup> for the prosthetic control, and  $0.95 \pm 0.11$  MPa·m<sup>1/2</sup> for the inlay/onlay control (Tukey's at 0.95).

Wear scar depth, diameter, and volume decreased significantly (one-way ANOVA;  $p < 0.001$ ) with increasing filler level (Fig. 2). In (A), whisker composite with 74% fillers had a wear depth of  $77.7 \pm 6.9$   $\mu\text{m}$ , significantly less than the depth of  $118.0 \pm 23.8$   $\mu\text{m}$  for the inlay/onlay control and  $172.5 \pm 15.4$   $\mu\text{m}$  for the prosthetic control (Tukey's at 0.95). In (B), wear scar diameter of a whisker composite with 74% fillers was  $742 \pm 46$   $\mu\text{m}$ , not significantly different from that of  $878 \pm 165$   $\mu\text{m}$  for the inlay/onlay control; both were less than that of  $1184 \pm 34$   $\mu\text{m}$  for the prosthetic control ( $p < 0.05$ ). We estimated wear volume in (C) by assuming a parabolic shape for the wear scar (Xu *et al.*, 2004). Wear volume for the composite with 74% whiskers was  $12.1 \pm 3.2$  ( $10^6 \mu\text{m}^3$ ), not significantly different from that of  $29.3 \pm 11.9$  ( $10^6 \mu\text{m}^3$ ) for the inlay/onlay control; both were less than that of  $91.2 \pm 11.8$  ( $10^6 \mu\text{m}^3$ ) for the prosthetic control ( $p < 0.05$ ).

We examined SEM micrographs of the worn surfaces inside the wear scars for unfilled resin and for whisker composites with 60% and 74% fillers (Figs. 3A, 3B, 3C). Whisker composites (Figs. 3B, 3C) had smooth surfaces free of the cracks seen in unfilled resin. Such cracks were also absent in composite with 20% and 40% whiskers. Long protruding whiskers, observed in fracture surfaces (Xu, 2000), were not seen in the worn surfaces. Instead, the whiskers were worn down by microfracture even with the composite surface, with the whisker tip showing signs of wear by microfracture (lower arrows in Fig. 3D).

## DISCUSSION

Increasing the nano-silica-fused whisker filler level improved the composite wear resistance. This is consistent with previous studies on wear and filler level (Bayne *et al.*, 1992; Condon and Ferracane, 1997; Lim *et al.*, 2002). The cracks in the wear scars of unfilled resin were also consistent with previous observations (Baran *et al.*, 1998). Whisker composites with fillers from 20% to 74% had relatively smooth worn surfaces, free of cracks like those in unfilled resin. The transition from cracking to non-cracking likely occurred between filler levels of 0% and 20%. A



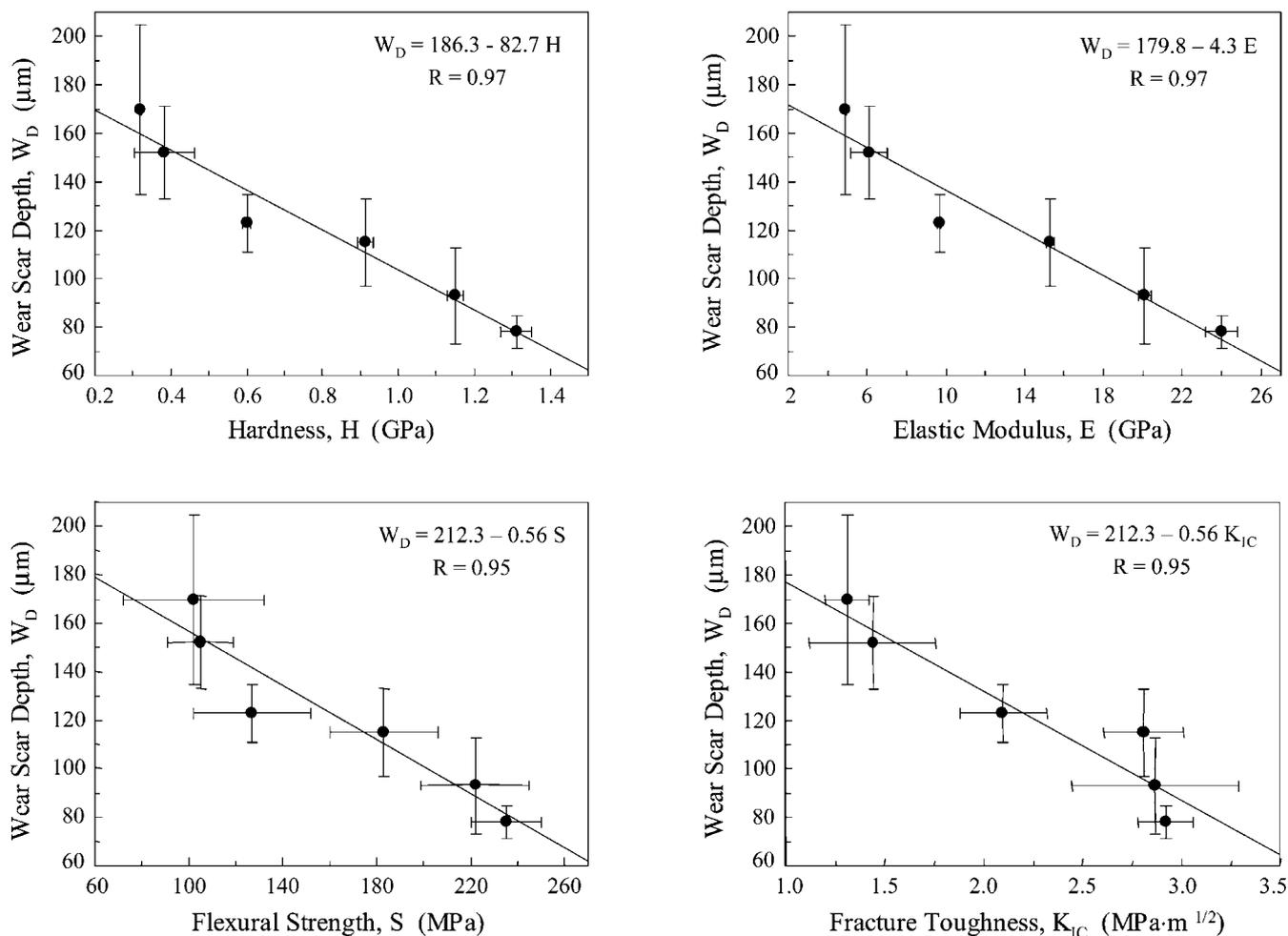
**Figure 3.** Worn surfaces. SEM micrographs inside wear scars after 400,000 cycles for (A) unfilled resin (0%), and (B) and (C) whisker composites at filler levels of 60% and 74%, respectively. Arrows in (A) point to microcracks in the worn surface of the unfilled resin. The whisker composites in (B) and (C) had relatively smooth surfaces inside the wear scars, free of cracks like those in the unfilled resin. A higher magnification in (D) for whisker composite at the 70% filler level shows a worn-down whisker with the tip exhibiting signs of wear by microfracture (lower arrows). The left arrow in (D) indicates that the whisker was firmly embedded in the resin matrix.

similar transition from brittle behavior to a more tough behavior was observed in a previous study (Xu, 1999). The unfilled resin cracked more readily, creating a flat surface from fast fracture (Fig. 2B in Xu, 1999). In contrast, the whisker composite exhibited toughening and crack deflection, with accompanying fracture steps on the fractured surfaces at filler levels starting from 10% (Fig. 2C in Xu, 1999). In addition to increased fracture resistance, the whisker composite with 70% and 74% fillers exhibited wear depths about half those of the unfilled resin. The whisker composite was also more wear-resistant than commercial glass-filled controls at similar filler levels.

Comparison can also be made with dental amalgam, which is known for its resistance to occlusal wear and is taken as the standard by which newer restorative materials are judged. A previous study, in which the same operator used the same equipment, subjected amalgam (Dispersalloy, Dentsply, Milford,

DE, USA) to 400,000 cycles of three-body wear, and measured a wear scar depth of  $134 \pm 54 \mu\text{m}$  and a diameter of  $778 \pm 270 \mu\text{m}$  (Xu *et al.*, 1999). The whisker composite with 74% fillers had a wear scar depth of  $77.7 \pm 6.9 \mu\text{m}$  and a wear diameter of  $742 \pm 46 \mu\text{m}$ . Regarding the correlation between these *in vitro* wear values and clinical wear, a previous study reported on results with use of the same type of wear machine, compared with *in vivo* data (Leinfelder and Suzuki, 1999). These investigators found that the 400,000-cycle *in vitro* wear values agreed with the *in vivo* wear values over a three-year period. This is consistent with results from another study showing that a wear depth of 100-160  $\mu\text{m}$  occurred for amalgam in 2-3 yrs (DeLong *et al.*, 1985).

Wear of dental materials is a complex process involving fatigue, erosive, adhesive, abrasive, and corrosive components. Nevertheless, wear occurs *via* microfracture and material removal; hence it is inherently related to mechanical properties.



**Figure 4.** Relationships between wear and mechanical properties. To understand the mechanisms of three-body wear of dental composites, we established linear best-fits between wear depth  $W_D$  and (A) hardness  $H$ , (B) elastic modulus  $E$ , (C) flexural strength  $S$ , and (D) fracture toughness  $K_{IC}$  of the nano-silica-fused whisker composites at filler level mass fractions ranging from 0% to 74%. Each value is the mean of 6 measurements, with the error bar showing 1 standard deviation (mean  $\pm$  SD;  $n = 6$ ).

McKinney *et al.* (1987) suggested that "wear does not necessarily vary in a manner consistent with the hardness." Pallav *et al.* (1989) found "the absence of a relationship between wear" and hardness or diametral tensile strength. Hardness of the nano-silica-fused whisker composites has been measured by nano-indentation (Xu *et al.*, 2000). Contrary to previous reports, we found a good correlation between wear depth and hardness for whisker composites, with a correlation coefficient of  $R = 0.97$  (Fig. 4A).

Tyas (1990) found some correlation, but not significant correlation, between wear and elastic modulus. Peutzfeldt and Asmussen (1992) concluded that no correlation was established between wear and modulus of elasticity. For the whisker composites, with the wear depth measured here and elastic modulus from a previous study (Xu *et al.*, 2000), a linear correlation was found between wear and elastic modulus, with  $R = 0.97$  (Fig. 4B).

A linear correlation between wear and flexural strength was found previously (Peutzfeldt and Asmussen, 1992). A similar relationship was found for whisker composites (Fig. 4C), with strength from an earlier study (Xu, 2000).

Wear involves microfracture; hence, it is also expected to

depend on fracture toughness (arrows in Figs. 3A, 3D). However, besides correlating fracture toughness with surface chipping and bulk fracture, little evidence was available on the relationship between fracture toughness and wear of dental composites (Tyas, 1990). Using the fracture toughness and wear depth data measured here, we obtained a correlation with  $R = 0.95$  (Fig. 4D).

When the inlay/onlay and prosthetic controls were included in the fitting, the correlation coefficients between wear depth and hardness, elastic modulus, flexural strength, and fracture toughness fell to 0.86, 0.87, 0.74, and 0.78, respectively. This indicates that the resin composition and degree of cure may also have influenced wear. Further study should examine whether such relationships are unique to a single class of composites or can be generalized across classes of composites. The measured wear and mechanical properties depend on the measurement methods. For example, flexural strength may depend on the bending span and the loading rate. Therefore, while fundamental relationships may exist, one should not expect to produce relationships similar to those illustrated in Fig. 4 by simply taking data in the literature of different materials measured in different laboratories.

In conclusion, novel nano-silica-fused whisker composites were developed with *in vitro* wear resistance higher than that of conventional glass-particle-filled composites and similar to that of dental amalgam. The wear surfaces of whisker composites were smooth and free of cracks. Linear correlations were established between composite wear and hardness, elastic modulus, flexural strength, and fracture toughness. Nano-silica-fused whisker composites with superior strength, fracture toughness, and wear resistance are relatively opaque and may be useful in large stress-bearing posterior restorations involving cusps and indirect applications. Further studies should match the refractive index of whiskers to that of the resin to improve the esthetics for anterior applications.

## ACKNOWLEDGMENTS

The authors thank Dr. Frederick C. Eichmiller for discussions, and Dr. Joseph M. Antonucci for providing the resin monomer. This study was supported by NIDCR grant R29 DE12476 (Xu), by NIST, and by the ADAF.

## DISCLAIMER

Certain commercial materials and equipment are identified to specify the experimental procedure. This does not imply recommendation or endorsement by NIST or ADAF or that the material or equipment identified is necessarily the best available for the purpose. One standard deviation was given in this paper for comparative purposes as the estimated standard uncertainty of the measurements. These values should not be compared with data obtained in other laboratories under different conditions.

## REFERENCES

- Baran G, Sadeghipour K, Jayaraman S, Silage D, Paul D, Boberick K (1998). Crack propagation directions in unfilled resins. *J Dent Res* 77:1864-1873.
- Bayne SC, Taylor DF, Heymann HO (1992). Protection hypothesis for composite wear. *Dent Mater* 8:305-309.
- Christensen GJ (1999). Porcelain-fused-to-metal vs. nonmetal crowns. *J Am Dent Assoc* 130:409-411.
- Condon JR, Ferracane JL (1997). *In vitro* wear of composite with varied cure, filler level, and filler treatment. *J Dent Res* 76:1405-1411.
- de Gee AJ, Van Duinen RNB, Werner A, Davidson CL (1996). Early and long-term wear of conventional and resin-modified glass ionomers. *J Dent Res* 75:1613-1619.
- DeLong R, Sakaguchi RL, Douglas WH (1985). The wear of dental amalgam in an artificial mouth: a clinical correlation. *Dent Mater* 1:238-242.
- Donly KJ, Jensen ME, Triolo P, Chan D (1999). Clinical comparison of resin composite inlay and onlay posterior restorations and cast-gold restorations at 7 years. *Quintessence Int* 30:163-169.
- Ferracane JL, Berge HX, Condon JR (1998). In vitro aging of dental composites in water—effect of degree of conversion, filler volume, and filler/matrix coupling. *J Biomed Mater Res* 42:465-472.
- Krause WR, Park SH, Straup RA (1989). Mechanical properties of Bis-GMA resin short glass fiber composites. *J Biomed Mater Res* 23:1195-1211.
- Kübler J (1999). The Versailles project on advanced materials and standards (VAMAS) and the European Structural Integrity Society (ESIS). Fracture toughness of ceramics using the SEVNB method; round robin. Swiss Federal Laboratories for Materials Testing and Research.
- Leinfelder KF, Suzuki S (1999). In vitro wear device for determining posterior composite wear. *J Am Dent Assoc* 130:1347-1353.
- Lim BS, Ferracane JL, Condon JR, Adey JD (2002). Effect of filler fraction and filler surface treatment on wear of microfilled composites. *Dent Mater* 18:1-11.
- Loza-Herrero MA, Rueggeberg FA, Caughman WF, Schuster GS, Lefebvre CA, Gardner FM (1998). Effect of heating delay on conversion and strength of a post-cured resin composite. *J Dent Res* 77:426-431.
- Manhart J, Kunzelmann KH, Chen HY, Hickel R (2000). Mechanical properties and wear behavior of light-cured packable composite resins. *Dent Mater* 16:33-40.
- McKinney JE, Antonucci JM, Rupp NW (1987). Wear and micro-hardness of glass-ionomer cements. *J Dent Res* 66:1134-1139.
- Pallav P, de Gee AJ, Davidson CL, Erickson RL, Glasspoole EA (1989). The influence of admixing microfiller to small-particle composite resin on wear, tensile strength, hardness, and surface roughness. *J Dent Res* 68:480-490.
- Peutzfeldt A, Asmussen E (1992). Modulus of resilience as predictor for clinical wear of restorative resins. *Dent Mater* 8:146-148.
- Quinn JB, Sundar V, Lloyd IK (2003). Influence of microstructure and chemistry on the fracture toughness of dental ceramics. *Dent Mater* 19:603-611.
- Ruddell DE, Maloney MM, Thompson JY (2002). Effect of novel filler particles on the mechanical and wear properties of dental composites. *Dent Mater* 18:72-80.
- Sakaguchi RL, Douglas WH, DeLong R, Pintado MR (1986). The wear of a posterior composite in an artificial mouth: a clinical correlation. *Dent Mater* 2:235-240.
- Scherrer SS, Denry IL, Wiskott HWA (1998). Comparison of three fracture toughness testing techniques using a dental glass and a dental ceramic. *Dent Mater* 14:246-255.
- Suzuki S, Suzuki SH, Cox CF (1996). Evaluating the antagonistic wear of restorative materials. *J Am Dent Assoc* 127:74-80.
- Tyas MJ (1990). Correlation between fracture properties and clinical performance of composite resins in Class IV cavities. *Austr Dent J* 35:46-49.
- Wassell RW, McCabe JF, Walls AWG (1994). A two-body frictional wear test. *J Dent Res* 73:1546-1553.
- Watts DC, Hindi AA (1999). Intrinsic "soft-start" polymerization shrinkage-kinetics in an acrylate-based resin composite. *Dent Mater* 15:39-45.
- Wendt SL (1987). The effect of heat as a secondary cure upon the physical properties of three composite resins: I. Diametral tensile strength, compressive strength and marginal dimensional stability. *Quintessence Int* 18:265-271.
- Xu HHK (1999). Dental composite resins containing silica-fused ceramic single-crystalline whiskers with various filler levels. *J Dent Res* 78:1304-1311.
- Xu HHK (2000). Whisker-reinforced heat-cured dental resin composites: effects of filler level and heat-cure temperature and time. *J Dent Res* 79:1392-1397.
- Xu HHK, Eichmiller FC, Giuseppetti AA, Ives LK, Parry EE, Schumacher GE (1999). Three-body wear of a hand-consolidated silver alternative to amalgam. *J Dent Res* 78:1560-1567.
- Xu HHK, Smith DT, Schumacher GE, Eichmiller FC, Antonucci JM (2000). Indentation modulus and hardness of whisker-reinforced heat-cured dental resin composites. *Dent Mater* 16:248-254.
- Xu HHK, Quinn JB, Giuseppetti AA, Eichmiller FC, Parry EE, Schumacher GE (2004). Three-body wear of dental resin composites reinforced with silica-fused whiskers. *Dent Mater* 20:220-227.