

Indentation modulus and hardness of whisker-reinforced heat-cured dental resin composites[☆]

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Abstract

Objectives: Recent studies showed that ceramic whisker reinforcement imparted a two-fold increase in the strength of dental composites. The aim of this study was to investigate the indentation response and measure the elastic modulus, hardness, and brittleness of whisker-reinforced heat-cured resin composites as a function of filler level, heat-cure temperature, and heat-cure duration.

Methods: Silica particles were fused onto silicon nitride whiskers to facilitate silanization and to roughen the whiskers for improved retention in matrix. Whisker filler mass fractions of 0, 20, 40, 60, 70, 74 and 79% were tested. Heat-cure temperature ranged from 100 to 180°C, and duration from 10 min to 24 h. A nano-indentation system enabled the measurement of elastic modulus. Fracture toughness was measured and composite brittleness index was calculated. An inlay/onlay composite and a prosthetic composite were tested as controls.

Results: Whisker filler level and heat-cure duration had significant effects on composite properties, while heat-cure temperature had non-significant effects. The whisker composite with 79% filler level had a modulus in GPa (mean (SD); $n = 6$) of 26.9 (1.0), significantly higher than 15.1 (0.2) of an inlay/onlay control, and 16.1 (0.3) of a prosthetic control (Tukey's multiple comparison test; family confidence coefficient = 0.95). The fracture toughness in MPa·m^{1/2} was 2.22 (0.26) for the whisker composite, higher than 0.95 (0.11) for inlay/onlay control, and (1.13 ± 0.19) for prosthetic control. The brittleness index was (0.49 ± 0.07) for whisker composite, lower than (1.02 ± 0.12) for inlay/onlay control and (0.63 ± 0.13) for prosthetic control.

Significance: Whisker filler level had a profound influence, heat-cure duration had significant effects, while temperature did not have significant effects, on the properties of whisker composite. The whisker composite had significantly higher elastic modulus and fracture toughness, and lower brittleness than the inlay/onlay and prosthetic controls. Published by Elsevier Science Ltd.

Keywords: Ceramic whisker; Heat cure; Resin composite; Filler level; Indentation; Elastic modulus; Brittleness; Fracture toughness

1. Introduction

The influence of fillers, resins, and cure conditions on the properties of dental resin composites has been extensively studied [1–9]. Inlays and onlays of resin composites were developed to overcome problems associated with polymerization shrinkage encountered by direct-filling techniques. The extra-oral polymerization of composite and cementation of the restoration, appear to improve marginal fit with

minimized contraction stress [10]. The composite mechanical properties were also improved by heat-curing or post-cure heat treatment, although such improvements were modest and sometimes not statistically significant [3,4,11–14].

A novel ceramic whisker filler system was recently developed for the reinforcement of dental composites [15]. Silica particles were fused onto ceramic whiskers to facilitate silanization, to minimize whisker entanglement, and to enhance whisker retention in the matrix by providing roughness. Whisker-reinforced composites demonstrated flexural strength nearly twice that of current dental composites [16]. However, that study focused mainly on composite flexural properties. The indentation modulus and hardness of whisker composites as a function of composition and cure method were not investigated.

Indentation tests provide important information on material deformation [17]. Flexural and tensile tests yield results

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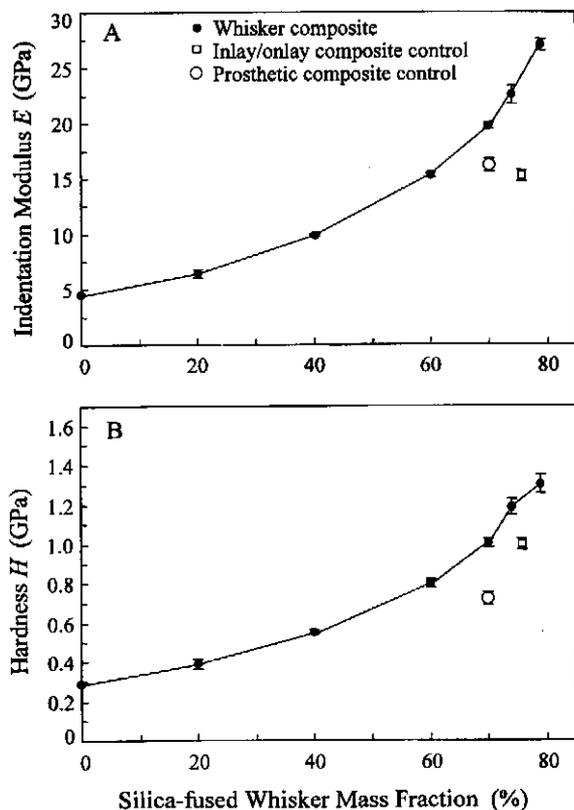


Fig. 1. (A) Indentation elastic modulus; and (B) hardness of whisker composite as a function of filler level, together with the inlay/onlay composite at a filler level of 76% and the prosthetic composite at a filler level of 70%. Each datum is mean with the error bar showing one standard deviation (SD) of six measurements ($n = 6$).

whether a change in hardness or modulus of whisker composite was related to that of the matrix, specimens of the unfilled resin were also cured at each of the above temperatures.

To examine the effect of heat-cure time, the whisker specimens were cured at 120°C at four different times: 10 min, 30 min, 3 h, and 24 h. Six specimens were cured at each cure time. Six specimens of the unfilled resin were also cured at each of the above times in the same oven.

2.4. Fracture toughness specimens

Toughness was measured for whisker composite at 70% filler level cured at 140°C for 30 min, and the inlay/onlay and prosthetic controls. Six specimens of each material were cured in the $2 \times 2 \times 20$ mm³ molds. A notch of a depth of 600 μ m was machined into each specimen by using a diamond blade (Brasseler, Lemgo, Germany) of a thickness of 150 μ m.

2.5. Testing

All the specimens were immersed in distilled water at 37°C for 24 h prior to being tested. A nano-indentation system (Nano Instruments, Knoxville, TN) with a diamond

Berkovich indenter, which is a three-sided pyramid with the same depth-to-projected area ratio as the Vickers indenter [25], was used to produce indentations. The indentation loads and the corresponding displacements were recorded continuously throughout a loading-unloading cycle, enabling measurement of the elastic modulus of the indented specimen. The calculation of hardness and elastic modulus was made according to a method described previously [26]. The method involves the extrapolation of a tangent to the top of the unloading curve to determine the depth (a combination of elastic and plastic displacement) over which the indenter tip is in contact with the specimen at the maximum load, P_{\max} . This depth, and the knowledge of the indenter geometry, gives the contact area, A ; hardness then follows directly from [26,28]:

$$H = P_{\max}/A \quad (2)$$

The slope of the unloading curve also provides a measure of the contact stiffness, which can be used with the contact area to determine the elastic modulus. The modulus obtained, sometimes referred to as the indentation modulus, E , is related to the Young's modulus E_Y by [26,28]

$$E = E_Y/(1 - \nu^2) \quad (3)$$

where ν is the Poisson's ratio. The Young's modulus E_Y can be obtained via Eq. (3) for materials with known ν . For materials with ν of approximately 0.25 [28], $E_Y = 0.94E$. The present study measured indentation modulus E , without trying to calculate E_Y .

Twenty-four indentations were made for each material with four indentations in each of the six specimens. This process produced a total of 552 indentations in 23 materials. A P_{\max} of 1 N was used to yield an indentation contact area of about 600–3000 μ m², depending on the hardness of material. This method ensures that the measured hardness and modulus approximate those of the composite bulk rather than the resin phase or filler particles.

Fracture toughness was measured by using a single-edge notched beam method [30]. A three-point flexural fixture with a span of 10 mm was used to fracture the specimens at a crosshead speed of 1 mm/min on a computer-controlled universal testing machine (model 5500R, Instron Corp., Canton, MA). The notched specimens were fractured with the notch on the tensile side and the loading pin aligned with the notch.

One-way ANOVA was performed to detect significant ($\alpha = 0.05$) effects of parameters. Tukey's Multiple Comparison procedures were used to group and rank the measured values at a family confidence coefficient of 0.95.

3. Results

The elastic modulus and hardness of the whisker composite increased non-linearly with filler level from 0 to 79%

on deformation and fracture of the bulk specimens. Indentation, on the other hand, offers information that may be more relevant to applications that involve localized, non-uniform deformation or point-contacts, such as occlusal contacts with surface asperities or third bodies during chewing and wear. The indentation method is especially useful when specimen dimensions are limited, such as in the case of tooth enamel or restoration in a tooth preparation. Indentation has been used to characterize mechanical properties of restorative materials [18–23], and to quantify the work-hardening inside a small fatigued zone [24].

The indentation hardness, H , can be used with fracture toughness, T , to yield the brittleness of materials, B , defined as [17]:

$$B = H/T \quad (1)$$

In restorative materials with the same fracture toughness, the material with a smaller hardness possesses a lower brittleness because it more readily yields under contact load, increasing the contact area to produce a smaller contact stress. For materials with the same hardness, the tougher material possesses a lower brittleness. With the unit of hardness being GPa, and that of fracture toughness being $\text{MPa}\cdot\text{m}^{1/2}$, the unit of brittleness is $\text{GPa}/(\text{MPa}\cdot\text{m}^{1/2})$. For simplicity, a brittleness index can be used with the unit of brittleness being omitted. As examples, the brittleness index for a silicate glass is approximately 8 (highly brittle), and for steel it is about 0.1 (low brittleness) [17].

In addition to hardness, instrumented indentation techniques can continuously monitor loading–unloading during an indentation cycle. This process provides information on the energy absorbed by the material during indentation [25] and the elastic modulus [26–28].

The aim of the present study, therefore, was to investigate the hardness, modulus, fracture toughness and brittleness of whisker-reinforced heat-cured composites versus filler level, heat-cure temperature, and heat-cure time. A current inlay/onlay composite and a prosthetic composite were tested as controls. It was hypothesized that whisker-reinforced composite would possess significantly higher elastic modulus and fracture toughness than the control composites. It was further hypothesized that whisker filler level, heat-cure temperature and duration of exposure would significantly influence the properties of whisker composite.

2. Materials and methods

2.1. Filler powder and resin preparation

Silicon nitride ($\beta\text{-Si}_3\text{N}_4$) whiskers (UBE Industries, New York, NY) with a mean diameter of $0.4\ \mu\text{m}$ and length of $5\ \mu\text{m}$ were mixed with fumed silica of $0.04\ \mu\text{m}$ particle size (Aerosil OX50, Degussa Corp., Ridgefield, NJ). A whisker:silica mass ratio of 2:1 was used and the mixture was dispersed by stirring in ethyl alcohol under moderate

vacuum until dry [16]. To fuse silica onto the whiskers, the dried mixture was heated in air for 30 min at 800°C [16]. The heated powder was silanized by mixing it with mass fractions of 2% *n*-propylamine (Aldrich, Milwaukee, WI) and 4% 3-methacryloxypropyltrimethoxysilane (MPTMS) (Aldrich, Milwaukee, WI) in cyclohexane by means of a rotary evaporator in a 90°C water bath until dry.

The whiskers were mixed by hand spatulation with a resin monomer consisting of mass fractions of 48.965% of an oligomeric urethane derivative of Bis-GMA (NCO/Bis-GMA, Caulk/Dentsply, Milford, DE), 48.965% triethylene glycol dimethacrylate (TEGDMA) (Esstech, Essington, PA), 0.070% 4-methoxyphenol (MEHQ) (Aldrich, Milwaukee, WI), and 2.000% benzoyl peroxide (BPO) (Aldrich, Milwaukee, WI). The resulting paste was placed into steel molds of $2 \times 2 \times 20\ \text{mm}^3$ dimensions and heat cured as described later.

2.2. Effect of whisker filler level

Seven whisker/(whisker + resin monomer) mass fractions were used: 0, 20, 40, 60, 70, 74 and 79%. They corresponded to volume fractions of approximately 0, 13, 26, 39, 45.5, 48.1 and 51.4%, respectively. At each filler level, six specimens were cured in an oven (model 48, Fisher Scientific, Pittsburg, PA) at 120°C for 30 min. The temperature was measured by an analog thermometer (Kessler, Westbury, NY) installed inside the oven. All the whisker specimens were cured in this oven at atmospheric pressure.

Control specimens were also fabricated. Following the manufacturer's instructions, the paste of an inlay/onlay composite (Concept™, Ivoclar North America, Amherst, NY) was placed into the same molds and cured in a proprietary curing unit (Concept Heat Integrated Processor, Ivoclar) at 120°C for 10 min under a pressure of 85 psi (0.6 MPa). Concept consists of a mass fraction of approximately 76% of silicate fillers in a urethanedimethacrylate resin (Technical Data Sheet, Ivoclar). The paste of a prosthetic composite (Artglass™, Heraeus Kulzer GmbH, Wehrheim, Germany) was placed into molds and cured in a photo-curing unit (Dentacolor XS, Heraeus Kulzer GmbH, Wehrheim, Germany) for 90 s on each side of the specimen. According to the manufacturer, Artglass contains a mass fraction of 70% of barium glass in a resin with tetra- and hexa-functional groups in addition to conventional bi-functional methacrylates.

2.3. Effect of heat-cure temperature and time

The following heat-cure temperatures were used: 100, 120, 140, 160 and 180°C . Six specimens were cured at each temperature. The filler mass fraction was 70% and the cure time was 30 min. Samples were not cured at temperatures of 200°C , or higher, due to a slight discoloration of the specimens at this temperature. Temperatures of 80°C or lower were not tested because most indirect curing techniques call for approximately 120°C [3,29]. To examine

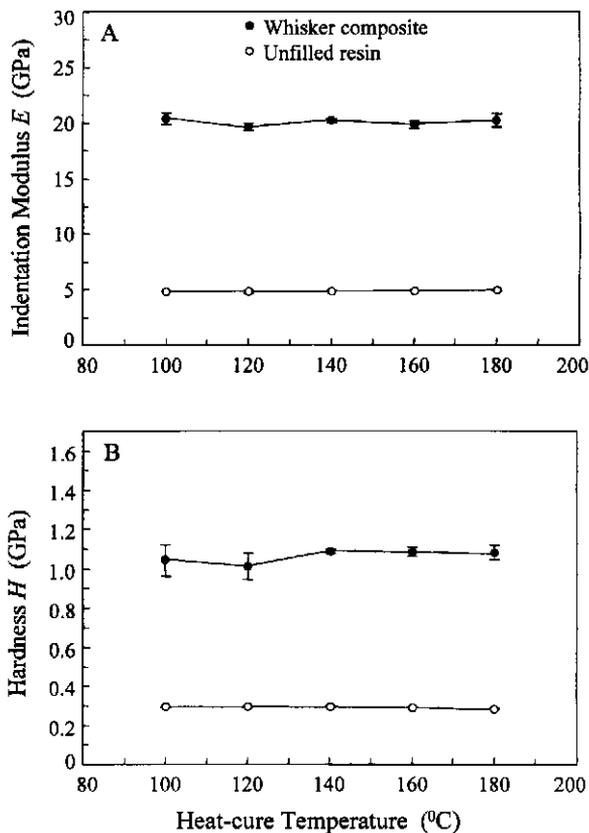


Fig. 2. Effect of heat-cure temperature on: (A) modulus; and (B) hardness of whisker composite at a filler level of 70% and unfilled resin at a cure time of 30 min. Each datum is mean with the error bar showing one SD, $n = 6$. Changing the heat-cure temperature from 100 to 180°C did not have a significant effect on the modulus or hardness.

(Fig. 1). The modulus (mean (SD); $n = 6$) at a whisker filler level of 0% (the unfilled resin) was 4.63 (0.05) GPa; it increased to 26.9 (1.0) GPa at a filler level of 79%, which was significantly higher than 15.1 (0.2) GPa for the inlay/onlay control and 16.1 (0.3) GPa for the prosthetic control (Tukey's Multiple Comparison method; family confidence coefficient = 0.95). The hardness was 0.29 (0.01) GPa at 0% filler level; it increased to 1.30 (0.05) GPa at filler level of 79%, which was significantly higher than 0.97 (0.03) GPa for the inlay/onlay composite and 0.71 (0.02) GPa for the prosthetic composite.

Fig. 2 shows that changing the heat-cure temperature from 100°C through 180°C did not have a significant effect on the modulus and hardness of the whisker composite at a filler level of 70% or the unfilled resin. On the other hand, heat-cure time had a significant effect on modulus and hardness (Fig. 3). The modulus at a cure time of 24 h was 20.3 (0.7) GPa, significantly higher than 17.8 (0.8) GPa at a cure time of 10 min. Sample hardness was 1.08 (0.04) GPa at a cure time of 24 h, significantly higher than 0.85 (0.03) GPa at a cure time of 10 min. For the unfilled resin, however, cure time did not have a significant effect on modulus or hardness, showing that the increase in the modulus or

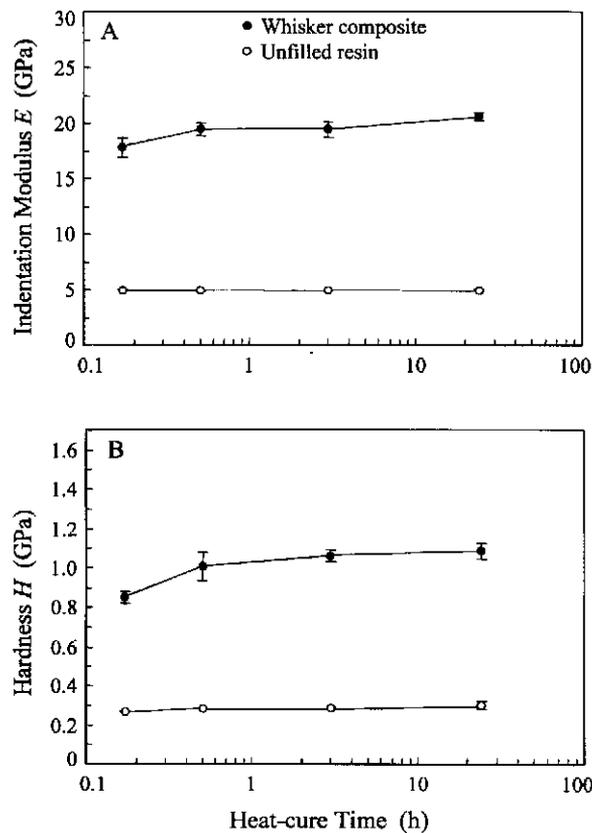


Fig. 3. Effect of heat-cure time on: (A) modulus; and (B) hardness of whisker composite at a 70% filler level and unfilled resin cured at 120°C. Each datum is mean with the error bar showing one SD, $n = 6$. Heat-cure time had a significant effect on the modulus and hardness of whisker composites, but not the unfilled resin.

hardness of the whisker composite was not a result of a stiffer or harder matrix.

Fig. 4 plots fracture toughness and brittleness of the inlay/onlay composite, the prosthetic composite, and the whisker composite at a filler level of 70% cured at 140°C for 30 min. The fracture toughness was 2.22 (0.26) $\text{MPa}\cdot\text{m}^{1/2}$ for the whisker composite, significantly higher than 0.95 (0.11) $\text{MPa}\cdot\text{m}^{1/2}$ for the inlay/onlay composite, and 1.13 (0.19) $\text{MPa}\cdot\text{m}^{1/2}$ for the prosthetic composite. The brittleness index was calculated using Eq. (1) to be 0.49 (0.07) for the whisker composite, 0.63 (0.13) for the prosthetic composite, and 1.02 (0.12) for the inlay/onlay composite.

4. Discussion

Heat-cured resin composites reinforced with silica-fused ceramic whiskers possessed elastic moduli, hardness, and fracture toughness values significantly greater than those of the control inlay/onlay resin composite and prosthetic composite. In the research and development of tooth-like restorative materials for stress-bearing occlusal applications, it should be noted that the elastic modulus of tooth

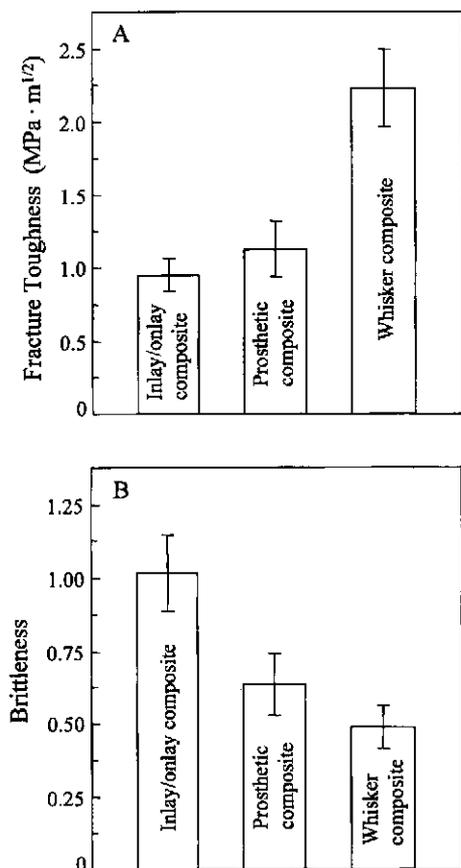


Fig. 4. (A) Fracture toughness; and (B) brittleness (Eq. (1)) of composites. Each datum is mean with the error bar showing one SD, $n = 6$.

enamel is approximately 85 GPa, and the hardness of tooth enamel is about 3.7 GPa [28]. The modulus value and hardness value of the whisker composite at a filler level of 79% (Fig. 1) are still lower than those of tooth enamel, and are nearly equal to a modulus of 27.3 GPa [31] and a hardness of 1.4 GPa of a dental amalgam [24]. As the filler level was reduced, the modulus and hardness of the whisker composite decreased. Previous studies have investigated the effects of filler level on the composite properties [7,32–36]. The composite wear depth was found to decrease with increasing filler level [37]. The diametral tensile strength and toughness of composite first increased, then decreased with increasing filler level [35]. The fatigue resistance of composite first increased, reaching a maximum, then decreased with increasing filler level [36]. These studies suggest that strength and fatigue resistance of conventional composites usually reach a maximum at an intermediate filler level. Hardness and modulus, on the other hand, appeared to increase in a monotonic manner with increasing filler level, as manifested in the results of the present study with filler level ranging from 0 to 79%. This result is consistent with a previous study showing that composite modulus increased monotonically with increasing filler level [34].

The heat-cure temperature ranging from 100 to 180°C did not have a significant effect on the modulus and hardness of

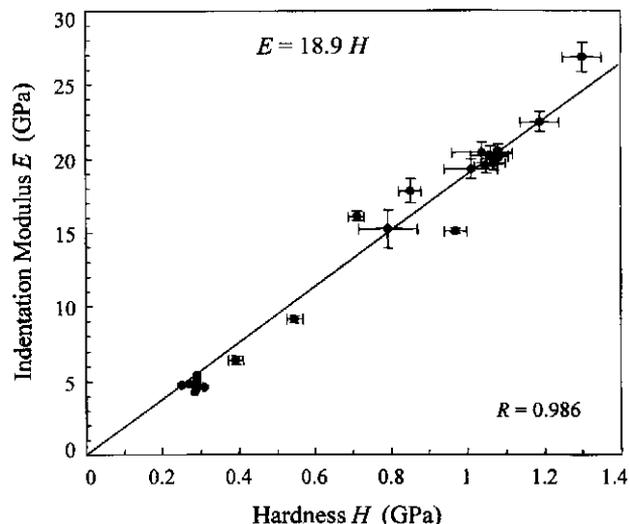


Fig. 5. Elastic modulus versus hardness for whisker composites at various filler levels, the unfilled resin, and the two control composites. Each datum is mean with horizontal error bar showing one SD with $n = 6$ in hardness and vertical error bar showing one SD with $n = 6$ in modulus. Linear regression through the origin provided a high correlation coefficient ($R = 0.986$). The elastic modulus is linearly related to the hardness by a constant $E = 18.9H$.

the whisker composites. However, increasing the heat-cure time from 10 min to 24 h significantly increased the elastic modulus and hardness of the whisker composite, but not the unfilled resin (Fig. 3). These results suggest that the hardness increase in the whisker composite in Fig. 3 was not a result of a harder matrix due to prolonged cure. The matrix hardness did not increase; the whisker hardness did not increase (silicon nitride is stable at temperatures even higher than 1000°C); but the whisker–resin composite hardness did increase. Finding the contributor(s) to this increase will require further study. One possible explanation is that the silane interface between the whiskers and the resin was influenced by varying the heat-cure time, and had contributed to the composite hardness and modulus.

The ratio of hardness/fracture toughness (Fig. 4) serves as a useful parameter in comparing the brittleness of material [17]. The brittleness index of the inlay/onlay composite (1.02) and the prosthetic composite (0.63) are higher than that of the whisker composite (0.49). For microstructural design of new restorative materials with reduced brittleness, efforts should be focused on increasing the toughness more than increasing the hardness. This is because increasing the hardness more than increasing the toughness only yields more brittle material.

The elastic modulus/hardness ratio is useful in describing the deformation of materials [17,38,39]. The modulus is the material's resistance to elastic deformation; the hardness is the material's resistance to local plastic deformation. To evaluate the modulus/hardness ratios of composites in this study, Fig. 5 plots modulus versus hardness for whisker composites at various filler levels and the unfilled resin.

together with the control composites. With linear regression through the origin and a correlation coefficient R of 0.986, the modulus is related to hardness by

$$E = 18.9 H \quad (4)$$

Therefore, the modulus/hardness ratio is approximately a constant for all the materials tested in the present study. While this result suggests that the nature of atomic bonding and the fundamental deformation mechanisms for these materials are similar, the practical usefulness is that the modulus can be estimated once the hardness is known, or vice versa. Further study should explore whether Eq. (4) holds true for other dental materials, such as direct-filling resin composites and glass ionomer-type materials. It is possible that Eq. (4) is only applicable when the polymer network has developed sufficient cure. Therefore, it will be interesting to examine whether Eq. (4) holds true for resin composites with different degrees of polymerization conversion. Furthermore, since the measured hardness of a material may depend on indenter geometry and indentation load [24], one probably should not expect to produce a relation similar to Eq. (4) by simply taking the hardness data in the literature of different materials measured in various laboratories.

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