

Surface properties of an indirect composite polymerized with five laboratory light polymerization systems

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Abstract: A study was conducted to evaluate the influence of laboratory light polymerization systems on the post-curing properties of a composite. An indirect composite (Sinfony) was polymerized with five polymerization systems (Visio system, Hyper LII, Pearlure Light, Twinkle MIII, and UniXS II) using nine polymerization modes. After light exposure, Knoop hardness number, wear depth, and changes in gloss were determined. The highest hardness number was recorded with the use of the Hyper LII (120 s) and Pearlure Light (120 s) units, whereas the lowest value was obtained with the Visio system and UniXS II (60 s). Six groups demonstrated comparable as well as higher wear resistance to toothbrush abrasion (Hyper LII 60 and 120 s, UniXS II 120 s, Pearlure Light 60 and 120 s, and Twinkle MIII 120 s), and two groups exhibited lower wear resistance (Visio system and UniXS II 60 s). Gloss of the composite was not dependent on the polymerization mode used before wear testing. However, surface gloss was significantly reduced by toothbrush dentifrice abrasion. Within the limitations of the present experiment, it can be concluded that the Sinfony composite can be polymerized sufficiently with high-intensity light polymerization units. (*J Oral Sci* 51, 215-221, 2009)

Keywords: indirect composite; laboratory light polymerization; microhardness; abrasion; gloss.

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Introduction

Indirect composites are used as veneering agents for cast restorations, super-structures of implant-supported prostheses, and denture teeth (1,2). Although the properties of composites have improved substantially, several problems including discoloration, wear, and disappearance of gloss have been reported (3-9). Dentifrices that include hard abrasives tend to induce excessive surface change of composites (10-13), and the roughened surface of restorations promotes plaque accumulation and affects their esthetic appearance. Several heat-polymerized composites exhibited insufficient resistance to wear and roughening of the surface. Both the mechanical and esthetic properties of composites, however, have improved over the last two decades (14-16), and a number of studies have been conducted to improve the properties of indirect composites. Matsumura et al. (17) reported that application of a high-intensity light source considerably improved the properties of a microfilled composite material. Other studies have demonstrated that depth of cure, hardness, wear resistance, water sorption, and solubility are influenced by the type of laboratory polymerization units employed (18-23).

Although the association between the light intensity of polymerization units and post-cure properties of direct composites has been studied by many researchers (24-26), only limited information is available on the relationship between the energy delivered by laboratory polymerizing units and the properties of indirect composites. The purpose of this study was to evaluate the influence of light sources and their energy on Knoop hardness, abrasion resistance, and change in gloss of an indirect composite (Sinfony, 3M ESPE, Seefeld, Germany). The working hypothesis was that Knoop hardness, abrasion resistance, and change in gloss of the composite would be positively influenced by use of high-intensity light sources.

Materials and Methods

Composite material and polymerizing units

An indirect composite (Sinfony, Lot No. 245550, 253243) designed for inlays, onlays, and restoration veneers was selected. The Sinfony composite is composed of aliphatic and cycloaliphatic monomers and inorganic fillers: aluminum glass and SiO₂ with an average particle size of 0.6 μm (27). The E3 shade for the enamel portion was selected for the current experiments. Five laboratory light polymerization systems were assessed. The Visio system, designed for polymerizing the Sinfony composite, consisted of two polymerization units: Visio-Alfa and Visio-Beta Vario (3M ESPE). The light source of the former was a halogen lamp, whereas that of the latter was fluorescent tubes (27,28). The polymerization mode of the Visio-Beta Vario unit was 1-min irradiation under atmospheric pressure followed by 14-min irradiation under reduced pressure. The light sources of the other units were two metal halide lamps for the Hyper LII unit (Toho Dental Products, Saitama, Japan) (17,20), a mercury lamp for the Pearlure Light unit (Tokuyama Dental Corp., Tokyo, Japan), two halogen lamps and a metal halide lamp for the Twinkle MIII unit (Toho Dental Products) (20,21), and two xenon stroboscopic tubes for the UniXS II unit (Heraeus Kulzer GmbH, Wehrheim, Germany) (19,20). Table 1 summarizes the details of the polymerization systems, their power, and energy densities (range: 400-500 nm) determined using a spectroradiometer (USR-40D, Ushio Inc., Tokyo, Japan) equipped with a ND10 filter.

Knoop hardness measurements

The composite was packed into a split polytetrafluoroethylene mold with a cylindrical opening 10 mm in diameter and 2 mm in height, and covered with a glass plate (1.3 mm thickness, Micro Slide Glass, Matsunami Glass, Osaka, Japan). Polymerization was performed under the conditions shown in Table 1 using the five polymerization systems. After polymerization, the top surface of each specimen was wet-ground with a series of silicon carbide papers (#800-2,000, WetorDry Tri-M-ite, 3M Corp., St. Paul, MN, USA) and polished with felt (TexMet 1500, Buehler Ltd., Lake Bluff, IL, USA) and alumina (0.05 μm, Baikalex 0.05CR, Baikowski International Corp., Charlotte, NC, USA). The height of reduced surface was approximately 500 μm. All specimens were stored in distilled water at 37°C for 24 h. Knoop hardness number (KHN) was determined with a universal indenter (Micro Hardness Tester, Akashi Ltd., Yokohama, Japan) by application of a 0.49-N load for 30 s.

Toothbrush dentifrice abrasion

The composite material was packed into a stainless steel mold (25.0 × 18.0 × 2.0 mm) and covered with a glass plate. The specimens were polymerized using the nine modes shown in Table 1 for the five polymerization systems. After polishing in the same manner as described in the previous section, all specimens were stored in distilled water at 37°C for 14 days. Each specimen was fixed on a specimen holder attached to a toothbrush

Table 1 Light polymerizing units assessed

| Polymerizing unit | Light source | Exposure time | Power density (μW cm ⁻²) | Energy density (mJ cm ⁻²) | Distance (light-specimen) |
|-----------------------------|--|---------------|--------------------------------------|---------------------------------------|---------------------------|
| Visio-Alfa 9010546 | Halogen lamp * 100W×1, 400-550 nm | 15 s | 8,600 | 129 | 10 mm |
| Visio-Beta Vario 9110125 | Fluorescent tubes * 250W×2, 400-550 nm | 15 min | 300 | 270 | 40 mm |
| Hyper LII 160908 | Metal halide lamp ** 150W×2, 250-600 nm | 60 s 120 s | 5,400 | 324 648 | 80 mm |
| Pearlure Light L06G088 | Mercury lamp 150W×1, 380-550 nm | 60 s 120 s | 10,100 | 606 1,212 | 70 mm |
| Twinkle MIII 15030305 | Halogen lamp 150W×2, 400-600 nm Metal halide lamp ** 150W×1, 250-600 nm | 60 s 120 s | 4,800 | 288 576 | 80 mm |
| UniXS II 22764 | Xenon stroboscopic tube ** 100W×2, 350-520 nm | 60 s 120 s | 1,000 | 60 120 | 45 mm |

* Gohring et al. (28), ** Tanoue et al. (20)

abrasion testing machine (K236, Tokyo-Giken Co., Ltd, Tokyo, Japan) (13,18). A dentifrice containing abrasives of conventional silica (16,29) (Crest Tartar Protection Regular Paste, RDA-Value 136, Procter & Gamble Co., Cincinnati, OH, USA) was used as an abrasive slurry with a paste-to-water ratio of 1:1. The vessel of the machine was loaded with 150 g of slurry. A toothbrush (Bee King, Bee Brand Medico Dental Co., Ltd., Osaka, Japan) with nylon filaments (Tynex, Dupont Japan, Tokyo, Japan) was fixed in the toothbrush holder of the machine, and moved back and forth on the specimen at 140 strokes per minute. A consistent load of 3.4 N was applied to the top of the toothbrush holder with a steel weight (13,18). The specimens were abraded with a total of 20,000 reciprocal strokes. The toothbrush and slurry were replaced for the testing of each specimen. Six specimens were tested for each polymerization mode. After the testing, specimens were removed from the machine, ultrasonically cleaned in distilled water for 10 min, and gently air-dried.

The vertical loss of each specimen in μm was determined with a confocal scanning laser microscope (1LM21W, Lasertec Corp., Yokohama, Japan) equipped with a He-Ne (Wavelength: 633 nm) laser light source. The resolution performance of the microscope was approximately 0.03 μm . The distance from the original specimen surface to the deepest abraded point was defined as the wear depth. Diagrams of the wear depth determination are shown in Fig. 1.

Gloss

The surface gloss of the specimens before and after abrasion testing was measured with a gloss meter (GM-26D, Murakami Color Research Laboratory Co., Ltd., Tokyo, Japan). Gloss was expressed in gloss units (GU). The gloss meter was calibrated before each recording

session using a standard black board with a reference value of 92.1 at 60° incidence angle (5). The specimens were rinsed with distilled water and air-dried, then placed on the specimen stage at the top stage of the device. The specimen was covered with a black plate to prevent reflection from the backside of the specimen and external light. The average of three measurements was recorded per surface.

Scanning electron microscopic observation

The specimens polymerized with the Visio system and the specimens polymerized with the Hyper LII unit for 120 s were observed with a scanning electron microscope (ERA-8800FE, Elionix Inc., Tokyo, Japan). Typical specimens were mounted on stubs, adequately dried in a vacuum desiccator for 24 h, and vacuum-coated with osmium (HPC-1S, Vacuum Device Inc., Mito, Japan) for 30 s. The surfaces before and after abrasion testing were observed with the microscope operated at 15 kV.

Statistical analysis

Statistical analyses were performed with the SPSS software package version 15.0 (SPSS Inc, Chicago, IL, USA). For the three tests, mean values and standard deviations of six specimens were calculated for nine groups. Data distribution was evaluated by the Kolmogorov-Smirnov test. The results of KHN, wear depth, and gloss were primarily analyzed by the Levene test for evaluation of equality of variance. When the results of the Levene test showed homoscedasticity for all categories, one-way analysis of variance (ANOVA) and the Tukey HSD test were further performed with the value of statistical significance set at $P = 0.05$.

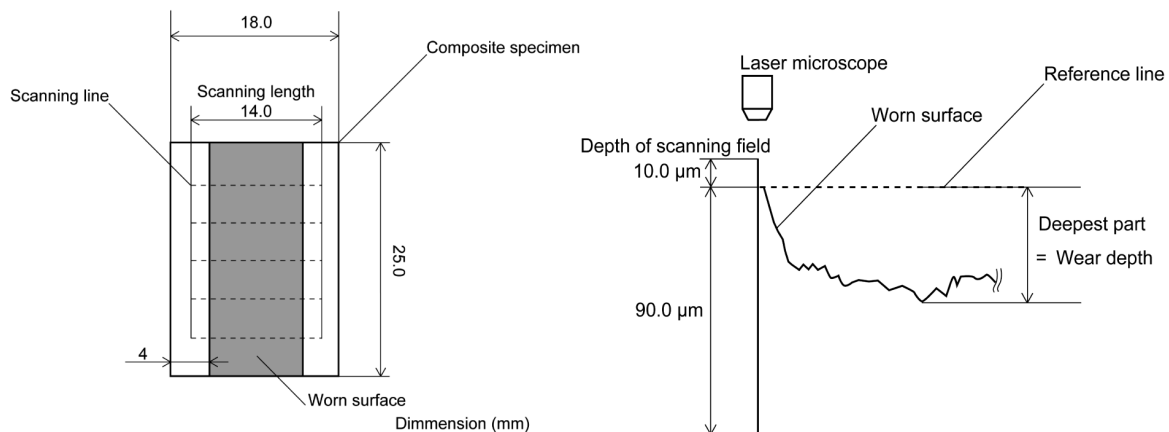


Fig. 1 Specimen design and location of the laser microscope scanning for evaluation of wear depth of the composite specimens.

Results

The Kolmogorov-Smirnov test run on the results of KHN, wear depth, and worn surface gloss revealed a normal distribution for each of the categories. Also, the Levene test run on the experimental results showed P-values greater than 0.05 for all tests, i.e., 0.367 for KHN, 0.641 for wear depth, and 0.713 for gloss. The results of the three tests were therefore analyzed by one-way ANOVA, and this revealed that the KHN ($P < 0.01$), wear depth ($P < 0.01$), and worn surface gloss ($P < 0.01$) were influenced by the polymerization mode. The results were finally analyzed with the post-hoc Tukey HSD test, and the mean values were categorized alphabetically.

The results of the hardness testing are presented in Table 2. KHN varied from 17.8 to 30.0, and was divided into five categories (a, b, c, d, and e) according to the post-hoc Tukey HSD test. Two groups polymerized either with the Hyper LII unit for 120 s (KHN 30.0) or with the Pearlcare Light unit for 120 s (KHN 28.6) showed the highest hardness values (category a). The hardness value of the specimens polymerized with the Visio system (KHN 19.5) was categorized into the lowest group (category e). For all polymerization units, the hardness value after 120 s light exposure was higher than that after 60 s exposure.

The results of toothbrush dentifrice abrasion testing (i.e., wear depth values) are shown in Table 3. Wear depth varied from a low of 26.2–32.7 μm (category f) to a high of 38.1–41.2 μm (category i). Tukey HSD test divided the results into four categories (f–i). Six groups (category f) demonstrated the lowest wear depth. For the Pearlcare Light and Hyper LII units, no significant differences in wear values were found between the 60 s and 120 s exposure periods. The wear depth value of the specimens polymerized with the Visio system (38.1 μm) was categorized into the most worn group (category i), although no significant difference was found among the three groups (category h) and between the two groups (category i).

Table 4 summarizes the gloss units determined before and after the toothbrush dentifrice abrasion test. The surface gloss of specimens before the abrasion test ranged from 82.2 to 85.9, and they were not significantly different from each other. The gloss of the worn surface varied from 28.0 to 46.3. Two categories (j and k) were generated after the wear testing. Two groups polymerized with the UniXS II unit for 60 s (GU 29.3, category k) and the Visio system (GU 28.0, category k) showed significantly lower gloss values than the other seven groups (GU 38.9–46.3, category j). Wilcoxon signed-rank tests run on the pre- and

Table 2 Knoop hardness test results

| Polymerization mode | Median | Mean | SD | Category |
|-----------------------|--------|------|-----|----------|
| Hyper LII 120 s | 30.0 | 30.0 | 1.6 | a |
| Pearlcare Light 120 s | 28.2 | 28.6 | 1.3 | a b |
| Twinkle MIII 120 s | 26.7 | 26.7 | 1.0 | b |
| Hyper LII 60 s | 24.8 | 24.6 | 1.2 | c |
| Pearlcare Light 60 s | 24.3 | 24.0 | 1.1 | c |
| Twinkle MIII 60 s | 23.9 | 23.7 | 0.7 | c |
| UniXS II 120 s | 20.8 | 20.8 | 0.7 | d |
| Visio system | 19.6 | 19.5 | 0.8 | d e |
| UniXS II 60 s | 18.0 | 17.8 | 0.7 | e |

SD, standard deviation; Category, Values with identical letters are not statistically different at $P = 0.05$ (Tukey HSD test).

Table 3 Wear test results after toothbrush dentifrice abrasion (μm)

| Polymerization mode | Median | Mean | SD | Category |
|-----------------------|--------|------|-----|----------|
| Hyper LII 120 s | 25.9 | 26.2 | 2.5 | f |
| UniXS II 120 s | 26.0 | 26.5 | 3.0 | f |
| Pearlcare Light 120 s | 28.6 | 28.3 | 2.6 | f g |
| Twinkle MIII 120 s | 28.4 | 28.6 | 3.9 | f g |
| Pearlcare Light 60 s | 28.8 | 30.3 | 3.4 | f g |
| Hyper LII 60 s | 33.0 | 32.7 | 4.0 | f g h |
| Twinkle MIII 60 s | 32.3 | 33.0 | 3.3 | g h |
| Visio system | 37.5 | 38.1 | 3.1 | h i |
| UniXS II 60 s | 42.0 | 41.2 | 4.9 | i |

SD, standard deviation; Category, Values with identical letters are not statistically different at $P = 0.05$ (Tukey HSD test).

Table 4 Gloss results before and after toothbrush dentifrice abrasion

| Polymerization mode | Before abrasion testing | | | After abrasion testing | | | Category | P-value |
|-----------------------|-------------------------|------|-----|------------------------|------|-----|----------|---------|
| | Median | Mean | SD | Median | Mean | SD | | |
| Twinkle MIII 120 s | 84.6 | 83.1 | 3.3 | 46.7 | 46.3 | 3.9 | j | 0.028* |
| Hyper LII 120 s | 82.8 | 82.9 | 0.6 | 44.4 | 43.9 | 2.5 | j | 0.028* |
| Pearlcure Light 120 s | 84.4 | 84.5 | 2.0 | 44.4 | 43.5 | 4.1 | j | 0.028* |
| Twinkle MIII 60 s | 86.0 | 85.9 | 1.2 | 41.9 | 42.3 | 3.2 | j | 0.028* |
| UniXS II 120 s | 82.0 | 82.2 | 0.7 | 41.6 | 41.7 | 4.4 | j | 0.028* |
| Pearlcure Light 60 s | 85.0 | 84.4 | 2.5 | 40.2 | 40.4 | 3.6 | j | 0.028* |
| Hyper LII 60 s | 83.5 | 83.5 | 0.4 | 39.3 | 38.9 | 6.7 | j | 0.028* |
| UniXS II 60 s | 83.6 | 83.6 | 0.6 | 31.5 | 29.3 | 5.5 | k | 0.028* |
| Visio system | 86.6 | 85.3 | 3.3 | 28.7 | 28.0 | 4.9 | k | 0.028* |

SD, standard deviation; Values with identical letters are not statistically different at $P = 0.05$ (Tukey HSD test). *Significant difference between the pre- and post-abrasion testing was found (Wilcoxon signed-ranks test; $P < 0.05$).

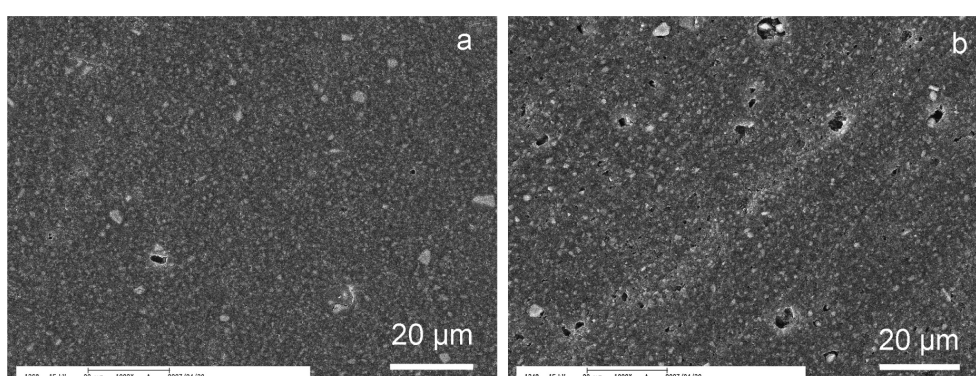


Fig. 2 Scanning electron micrographs ($\times 1,000$, bar = 20 μm) of the Sinfony surface polymerized with the Visio-Alfa and Beta Vario units. (a) before abrasion testing; (b) after abrasion testing.

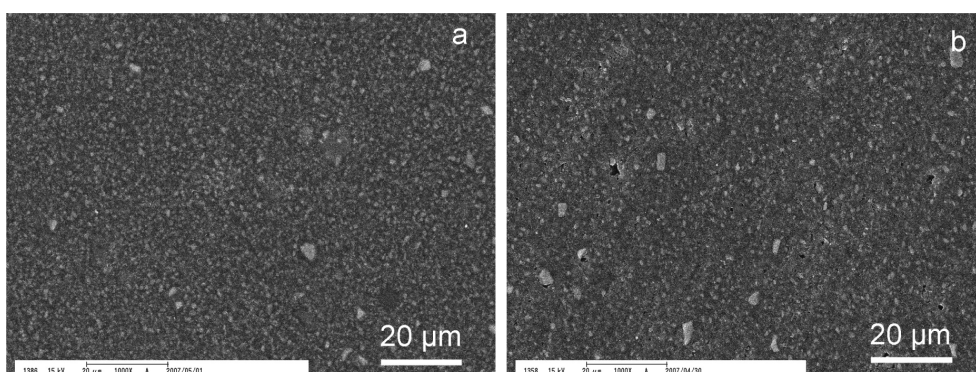


Fig. 3 Scanning electron micrographs ($\times 1,000$, bar = 20 μm) of the Sinfony surface polymerized with the Hyper LII unit for 120 s. (a) before abrasion testing; (b) after abrasion testing.

post-wear gloss values revealed that gloss was negatively affected by application of reciprocal brushing with dentifrice ($P = 0.028$) for all groups.

Figures 2 and 3 show electron micrographs before and after toothbrush abrasion testing. The abraded specimen polymerized with the Visio system exhibited multiple voids or detachment of macrofiller particles (Fig. 2b) as

compared with the post-abrasion specimen polymerized with the Hyper LII unit (Fig. 3b).

Discussion

Knoop hardness testing is useful for evaluating the properties of composites in relation to wear resistance (12,15,16). The present results demonstrated that Knoop

hardness was influenced considerably by both the type of polymerization unit and light exposure period. As shown in Table 2, hardness values after 120 s of light exposure were significantly greater than those after 60 s of exposure for the four polymerization units. These results indicate that the hardness of the Sinfony composite can be increased by extension of the light exposure period (i.e., energy density) of the four units. Also, the rank order of the polymerization units provided from Knoop hardness testing was almost comparable with the rank order of the polymerization units provided from the power density of each unit. Although the power density of a unit is affected by the distance between the light source and the specimen, as well as the set-up position of the light source, the present results suggest a positive relationship between the power or energy density of the light source and the hardness number of the composite material (30). The present results agree with those of previous studies that have examined the relationship between the hardness of direct composites and the energy of hand-held curing units (24-26).

Wear resistance is an important characteristic of indirect composite materials. This study demonstrated improvement of the wear resistance of the Sinfony composite by application of four different light polymerization units. These units have a stronger irradiation power than the Visio system. The results suggest that the wear resistance of the Sinfony composite can be improved by application of a high-energy light polymerization unit with a shorter light exposure period. However, evaluation of wear resistance showed that six polymerization modes recorded comparable wear depth values. The author speculates that the indentation generated by a hardness tester represents a substantial mechanical property of the material surface rather than the depth of the surface abraded by a toothbrush and dentifrice. As shown in the post-abrasion micrographs, wear was related to surface roughness or loss of filler particles of the composite. The results in Tables 2 and 3 show the characteristics of the polymerization units: greatest hardness and wear resistance with the Hyper LII and Pearlure Light units (120 s) and low hardness and wear resistance with the Visio system (15 min). The association between hardness and wear resistance of a composite under different conditions, shown in this study, agrees in part with other reports that have discussed the relationship between the hardness and wear resistance of various composites (16,31).

The gloss of the Sinfony composite was determined before and after the toothbrush abrasion test. No significant differences in the gloss of the composite among polymerization modes was evident before the abrasion test. This was probably due to compatibility between the polishing

method and composition of the composite. The gloss of the composite was significantly reduced for all polymerization modes by toothbrush dentifrice abrasion. These results suggest that reduction of the gloss cannot be avoided for the Sinfony composite when using the Crest Tartar Protection dentifrice. The use of dentifrices with strong abrasive is not recommended in patients with restorations and dentures made of the Sinfony composite. The two polymerization modes including the Visio system produced lower gloss values after abrasion as compared with the other seven modes. It is reasonable to consider that the composite material should be polymerized with sufficient energy to maintain the original surface characteristics.

The working hypothesis proposed in the Introduction section was accepted through the three types of property test. Clinicians and dental laboratory technicians should be aware that properties of indirect composite materials are affected considerably by the characteristics of light polymerization units.

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