# Effect of Ultraviolet Light Irradiation on Bonding of Experimental Composite Resin Artificial Teeth

Paola G. LOYAGA-RENDON, Hidekazu TAKAHASHI, Naohiko IWASAKI and Fazal REZA Advanced Biomaterials, Department of Restorative Sciences, Division of Oral Health Sciences, Graduate School, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan Corresponding author, LOYTAGA-RENDON Paola G.; E-mail: loyaga.abm@tmd.ac.jp

Received May 1, 2007/Accepted June 7, 2007

The purpose of the present study was to evaluate how ultraviolet light (UV) irradiation using an ordinary UV sterilizer would affect the bonding of experimental composite resins to an autopolymerizing acrylic resin. To this end, three composite resins and one unfilled resin — of which the compositions were similar to commercial composite resin artificial teeth — were prepared as repair composites. Their shear bond strengths after UV irradiation for one to 60 minutes were significantly greater than those before UV irradiation regardless of composite resins, but that before UV irradiation and after 24 hours' irradiation was mainly adhesive failure. These results thus suggested that a short period of UV irradiation on composite resin teeth would improve the bonding efficacy of composite resin artificial teeth to autopolymerizing resin.

Keywords: UV irradiation, Composite resin artificial teeth, Shear bond strength

### INTRODUCTION

Composite resin artificial denture teeth are frequently used for prosthetic rehabilitation. However, commercial products of composite denture teeth show great variations in hardness, filler shape and content. A common feature of the enamel or occlusal layer of composite resin artificial teeth is its highly crosslinked structure, which exhibited little swelling even after 12 hours of immersion in methyl methacrylate (MMA)<sup>1)</sup>. As for the wear resistance of composite resin artificial teeth, an in vitro study showed that it has been improved compared to conventional denture teeth<sup>2)</sup>. However, it must be highlighted that wear of composite resin denture teeth varies depending on the antagonist and that greater wear is incurred with certain antagonists<sup>3)</sup>. Therefore, wear of composite resin artificial teeth is a potential problem in denture-wearing patients. Eventually, the addition of resin onto the worn occlusal surfaces of artificial denture teeth is needed to maintain or restore the desired occlusal condition for a short or longer period of time. Due to the highly crosslinked nature of the enamel layer, it therefore means the necessity of surface treatment to achieve an appropriate bond. Moreover, the enamel layer of the buccocervical region of denture teeth should be well bonded to the denture base resin; otherwise, the cervical area of the artificial tooth shows discoloration or staining due to insufficient bonding.

To improve the bond strength of polymerized composite resins, several surface treatments have been suggested: create micromechanical retention by physical treatment (air abrasion<sup>4-6</sup>), grinding<sup>7</sup>) or chemical treatment (HF<sup>4</sup>),  $H_2O_2^{(8)}$ , silane coupling agents<sup>5,7,9-11</sup>), swell the surface with an organic sol-

vent (acetone<sup>4,12</sup>), or enhance the adhesive property of tribochemical silica coating using a silane coupling agent<sup>13</sup>. Although the aforementioned treatment methods have led to improved bond strength, technique sensitivity inherent in these methods has posed numerous difficulties to dental practitioners<sup>10,11</sup>, and they are also compounded by other related issues such as specific equipment requirements and elevated costs. Against this backdrop of technical challenges and cost-related issues, a simpler, inexpensive, and effective method for bonding acrylic resin to composite resin seems indeed attractive and desirable for use in both clinical and laboratory settings.

Ultraviolet photons are particularly energetic. They can cause molecular bonds to be broken when absorbed in a molecule, a process referred to as photolytic chain scission<sup>14)</sup>. The use of ultraviolet (UV) irradiation on the surface of polymers such as poly(dimethyl siloxane)<sup>15-17)</sup>, polypropylene<sup>18-20)</sup>, and poly(ethylene terephthalate)<sup>18,21)</sup> have been studied in lithography and microelectronics. In terms of advantages, UV irradiation can improve the adhesive property of coatings as well as wettability and printability of polymers by changing the morphology and chemical properties of the polymer surface. Additionally, UV irradiation has been used to induce photochemical changes resulting in oxidation of the exposed surface, modification of the hydrophobic nature of polymers, and increase in surface energy. Furthermore, when a polymer is exposed to ozone in the presence of UV light, increase in adhesive force at the surface has been reported<sup>20</sup>. In the dental field, use of UV lasers has been reported to enhance the adhesion of composite resins to dental enamel<sup>22)</sup>.

To date, the effects of UV irradiation have been

observed as chemical and structural changes on the polymers. By means of Fourier transform infrared spectroscopy (FTIR), photolytic chain scission, formation of different functional groups, and degradation of polymer backbone have been observed<sup>15,19</sup>. By means of X-ray photoelectron spectroscopy (XPS), changes in chemical bonding energy have been detected<sup>16,18,19,21</sup>. Then, by means of contact angle measurement, changes in surface hydrophobicity have been monitored<sup>15,16,18,19</sup>.

In a dental office, a UV sterilizer is an effective but inexpensive and speedy means of disinfection<sup>23-25)</sup>. It can also double as a potential source of UV irradiation for composite resin bonding treatment. The purpose of the present study, therefore, was to evaluate the effectiveness of UV irradiation — using an ordinary UV sterilizer — in promoting the bonding between experimental composite resin artificial teeth and acrylic resin.

#### MATERIALS AND METHODS

#### Specimen preparation

Materials used in the present study are listed in Table 1. An experimental heat-polymerizing resin was prepared based on a formulation of composite resin denture teeth<sup>3</sup>). The ratio of urethane dimethacrylate, triethylene glycol dimethacrylate, and neopenthylglycol dimethacrylate was 75/10/15 by mass%, and 1.5 mass% of benzoyl peroxide was also added.

Three types of fillers were used to prepare the composite resins: silanized microfine silica with average particle size of 0.06  $\mu$ m (MF); hybrid type of

silanized silica with average particle size of 6.6  $\mu$ m (HY); crushed prepolymerized microfine silica with trimethylolpropane trimethacrylate (TMPT) used for a commercial brand of composite artificial denture teeth, at an inorganic content of 47 mass% and with particle size ranging from 50  $\mu$ m to submicrons<sup>1)</sup> (PP). Amount of inorganic filler content of composite resins was 20 mass%.

Disk-shaped specimens, 4 mm in diameter and 2 mm in thickness, of the experimental unfilled resin and three composite resins were heat-polymerized. Vickers hardness values (HV0.3) of the unfilled resin and composite resins ranged from 21.5 to 27.3 kgf/ mm, which were similar to commercial composite resin artificial teeth<sup>2</sup>). Seventy-five specimens of each resin were prepared. Sixty specimens for the shear bond test were embedded in an acrylic resin tube (20 mm in diameter and 10 mm in height) with an autopolymerizing acrylic resin (Palapress Vario, Heraeus Kulzer, Hanau, Germany). The surfaces of all specimens were polished with 600-grit SiC paper using a polishing machine (Dialap ML150P, Maruto, Tokyo, Japan), then ultrasonically cleaned for 15 minutes and stored in a desiccator for at least 24 hours.

#### UV irradiation

UV irradiation was performed in an ultraviolet sterilizing oven (DM5, Daishin, Osaka, Japan) at a distance of 6 cm from the UV lamp in air at an ambient temperature. The UV lamp (GL-10, NEC Lighting, Tokyo, Japan) emitted UV light with mostly a wavelength of 253.7 nm at an intensity of 10 W. Exposure times of UV irradiation were 0 minutes (control), 1 minute, 10 minutes, 1 hour, and 24 hours.

Material	Brand	Lot No.	Manufacturer		
Matrix					
Urethane dimethacrylate	Art resin SH-500	N/A	Nagami Chemical Industrial, Japan		
Neopenthylglycol dimethacrylate	Purity 95.7%	$09624\mathrm{CU}$	Aldrich, Germany		
Triethylene glycol dimethacrylate	NK Ester-3G	0919R	Shin-Nakamura Chemical, Japan		
Benzoyl peroxide	1st grade of SAJ	A8345	Sigma Aldrich, Japan		
Fillers					
Silanized silica nanofiller	RM50	N/A	Nippon Aerosil, Japan		
Silanized silica hybrid filler	CMC12-S	2738-A1	Tatsumori, Japan		
Silanized silica prepolymerized filler	TMPT filler	171222-1	Nissin, Japan		
Adhesive					
Autopolymerizing acrylic resin	Unifast III	0.606066	GC, Japan		
		0.606022			
		N/A: Not a	vailable		

Table 1 Materials used in the present study

## Scanning electron microscope (SEM) observation

Carbon-coated, polished surfaces of three specimens of each unfilled resin and composite resin were observed before and after 24-hour UV irradiation with a scanning electron microscope (S-4500, Hitachi High-Technologies Corp., Tokyo, Japan).

## Shear bond strength test

Sixty specimens of each experimental unfilled resin and composite resin were embedded in an acrylic resin tube (20 mm in diameter and 10 mm in height) with an autopolymerizing acrylic resin (Palapress Vario, Heraeus Kulzer, Hanau, Germany). Surfaces of the specimens were polished with 600-grit SiC paper, then ultrasonically cleaned for 15 minutes and stored in a desiccator for at least 24 hours.

Specimens were divided into five groups (n = 12) to be subjected to the different exposure times of UV irradiation. Immediately after irradiation, another autopolymerizing acrylic resin (Unifast III, GC Corp., Tokyo, Japan) was built up on the specimen surface using a Teflon tube, 4 mm in diameter and 3 mm in height. A bonding area of 2 mm diameter was defined using a double-sided adhesive tape placed on the surface before build-up. Two acrylic resin tabs were built up on each specimen. The resin build-up was finished within 60 minutes after irradiation. Specimens were then stored in distilled water at 37°C for 24 hours in an incubator before the shear bond test.

Each specimen was inserted into a test jig, which ensured that the force applied was parallel to the bonding interface. Each specimen was subjected to a shear bond test using a micro material testing machine (MMT-250NB-10, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.5 mm/minute. Maximum load at failure was recorded. Shear bond strength (SBS) was determined as the maximum load divided by the cross-sectional bonding area. The average of two bond strength measurements was used as the representative value of each specimen.

Debonded surfaces of the acrylic resin build-up and the specimens were observed using a lowmagnification microscope ( $\times 20$ ; SZ, Olympus, Tokyo, Japan) to determine the failure mode. Failure modes of the specimens were classified into three categories as follows:

- Adhesive failure (AF): no crack of adherend (experimental unfilled resin or composite resins) or adhesive (autopolymerizing acrylic resin) was observed on the adherend surface.
- Mixed and cohesive failures of the adherend (CA): crack or fracture of the adherend was observed without any crack or fracture of the adhesive.
- Cohesive failures of the adherend and adhesive (CB): crack or fracture was observed on both the

adherend and adhesive.

# FTIR

Attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectra (sum of 64 scans) were recorded using a FTIR spectrometer (FTIR-8300, Shimadzu) equipped with an ATR accessory (DuraSampIIR<sup>™</sup>, ASI Technologies, Danbury, CT, USA). Spectral range covered was 4,000-600cm<sup>-1</sup> with a resolution of 4  $cm^{-1}$ . For each experimental unfilled resin and composite resin, three specimens were recorded before (as control) and after UV irradiation at 1 minute, 10 minutes, 1 hour, and 24 hours. For each specimen, three measurements were recorded within three minutes after the start of measurement. Spectra obtained were analyzed using a software (IR-Mentor Pro, Bio-Rad Japan, Tokyo, Japan).

#### Contact angle measurement

The contact angle of distilled water on specimens at 1 minute after placement was measured using a contact angle goniometer (CA-DT, Kyowa Kaimen Kagaku, Tokyo, Japan) with a sessile drop technique.

Two drops were placed on the surface of six specimens of each unfilled resin and composite resin, and measurement was finished within five minutes after the start of measurement. Measurements were done at 1 minute, 10 minutes, 60 minutes, and 24 hours after UV irradiation.

#### Statistical analysis

SBS and contact angle data were analyzed by twoway ANOVA and Tukey's multiple comparison test. Failure modes of the fractured surfaces were analyzed with nominal logistic analysis. A statistical software (JMP In 5.1, SAS, Cary, NC, USA) was used for these calculations ( $\alpha = 0.05$ ).

#### RESULTS

All UV-irradiated specimens, except for 24-hour irradiation, did not show obvious color changes. As for specimens after 24 hours of UV irradiation, they showed a detectable yellowish color change according to visual examination by the naked eye.

#### SEM observation

Figure 1 shows the SEM images of the unfilled resin and composite resins before and after 24-hour UV irradiation. Scratches at random directions were observed in all images, and the shapes of the prepolymerized filler could be recognized. However, there were no distinct differences between the images before and after irradiation.



60 µm

Fig. 1 SEM images of specimen surfaces before and after 24-hour UV irradiation (original magnification ×500). MF-C: microfine silica composite resin before irradiation, MF-24h: microfine silica composite resin after irradiation, HY-C: hybrid type silica composite resin before irradiation, HY-24h: hybrid type silica composite resin after irradiation, PP-C: prepolymerized microfine silica composite resin before irradiation, PP-24h: prepolymerized microfine silica composite resin after irradiation, UF-C: unfilled resin before irradiation, UF-24h: unfilled resin after irradiation.

	Irradiation Time								
Type of resin	Control	1 min	10 min	1 hr	24 hrs				
MF	$4.8 \pm 1.6$	$22.7 \pm 4.7$	$21.5 \pm 5.8$	$10.3 \pm 3.1$	$6.7 \pm 0.9$				
HY	$6.9 \pm 2.5$	$19.1 \pm 9.3$	$26.3 \pm 3.5$	$13.3 \pm 5.3$	$6.1 \pm 1.1$				
PP	$4.4 \pm 1.3$	$22.3 \pm 7.4$	$25.9 \pm 5.8$	$11.0 \pm 3.1$	$6.3 \pm 1.6$				
UF	$5.4 \pm 2.4$	$20.2 \pm 7.5$	$22.2 \pm 10.2$	$13.2 \pm 4.2$	$6.2 \pm 2.8$				
					$M_{opp} + SD$				

Table 2 Shear bond strengths (MPa) after UV irradiation

Mean  $\pm$  S.D.

MF: microfine silica composite resin; HY: hybrid type silica composite resin; PP: prepolymerized microfine silica composite resin; UF: unfilled resin

Source	$\mathbf{DF}$	Sum of squares	F ratio	Probability
Type of resin	3	49.5	0.8	0.4774
UV irradiation time	4	13692.1	172.7	< 0.0001
Type of resin $\times$				
UV irradiation time	12	403.2	1.7	0.0691
Error	220	4360.1		

Table 3 Two-way ANOVA analysis of shear bond strength results

Table 4	Percentages of failure modes of fractured surfaces after shear bond test	
	UV irradiation time	

	U v irradiation time														
	Control			1 min			10 min			1 hr			24 hr		
	AF	CA	CB	AF	CA	CB	AF	CA	CB	AF	CA	CB	AF	CA	CB
MF	100.0	0.0	0.0	0.0	83.3	16.7	0.0	95.8	4.2	58.3	41.7	0.0	83.	3 16.7	7 0.0
HY	100.0	0.0	0.0	33.3	50.0	16.7	0.0	83.3	16.7	9.1	81.8	9.1	83.	3 16.7	7 0.0
PP	87.5	12.5	0.0	0.0	69.6	30.4	0.0	58.3	41.7	25.0	75.0	0.0	66.	7 33.3	3 0.0
UF	100.0	0.0	0.0	8.3	79.2	12.5	0.0	52.2	47.8	8.3	91.7	0.0	87.	5 12.8	5 0.0
	96.9	3.1	0.0	10.4	70.5	19.1	0.0	72.4	27.6	25.2	72.5	2.3	80.	2 19.8	3 0.0

AF: Adhesive failure; CA: Mixed and cohesive failures of the adherend; CB: Cohesive failures of the adherend and adhesive.



Fig. 2 Effects of UV irradiation time on shear bond strength. Bars with the same letter were not statistically different. 95% CI: confidence interval of 95%.

#### Shear bond strength

Table 2 summarizes the SBS results. SBS values of the control specimens ranged from 4.4 to 6.9 MPa. After 1-minute UV irradiation, SBS increased to 19.1-22.7 MPa. However, SBS after 24-hour UV irradiation ranged from 6.1 to 6.7 MPa. Two-way ANOVA revealed that UV irradiation time was statistically significant (p<0.0001) (Table 3), but not resin type nor their interaction. Figure 2 then summarizes the effects of UV irradiation on shear bond strength. UV irradiation of 1 minute, 10 minutes, and 1 hour showed significantly greater SBS values compared to the control specimens and those after 24-hour irradiation according to Tukey's multiple comparison test. No significant differences were found between the control and 24-hour irradiation specimens.

Table 4 shows the distribution of the failure modes according to UV irradiation time and resin type. For the control specimens, the predominant

Table 5 Nominal logistic analysis of failure mode results

Source	DF	Wald chi square	Probability
Type of resin	6	10.46	0.107
UV irradiation time	4	59.38	0.000
Type of resin			
×	8	9.98	0.1253
UV irradiation time			

failure mode was adhesive failure (AF) (96.9%). After UV irradiation for 1 minute and 10 minutes, mixed and cohesive failures of the adherend (CA) was markedly observed - except for the HY composite resin after 1-minute UV irradiation which showed 50% of CA and 33.3% of AF. After 10-minute UV irradiation, no AF was observed; predominant failure mode was CA (72.42%). The ratio of CA after 1-hour UV irradiation was similar to that after 10 minutes; but the ratio of AF increased, while cohesive failures of the adherend and adhesive (CB) decreased. After 24-hour irradiation, the failure mode was mainly AF. Nominal logistic analysis of the failure mode results revealed a significant effect exerted by UV irradiation time. Conversely, the effects of resin type and the interaction between UV irradiation time and resin type were not significant (Table 5).

#### FTIR

Figure 3 shows the FTIR spectra of the four experimental resins after UV irradiation. Obvious differences were observed within the spectral range of 1,300 to 800 cm<sup>-1</sup>. At 1 hour after UV irradiation, no apparent changes were observed in the FTIR spectra. However, at 24 hours after irradiation, changes in peak intensity were observed. Peak intensities around 810 and 740 cm<sup>-1</sup> decreased, two peaks at 1,388 and 1,360 cm<sup>-1</sup> became one peak, and a new peak appeared at 1,320 cm<sup>-1</sup>. These findings were observed regardless of resin type. The peaks



Fig. 3 Changes of FTIR spectrum after UV irradiation. Lines from the bottom to top are the control, 1-minute, 10-minute, 1-hour, and 24-hour irradiation. After 24 hours' irradiation, peak intensities at **a** (740 cm<sup>-1</sup>) and **b** (810 cm<sup>-1</sup>) decreased, two peaks became one peak at **d** (1,360 cm<sup>-1</sup>), and a new peak appeared at **c** (1,320 cm<sup>-1</sup>).



Fig. 4 Effects of UV irradiation time on contact angle ( $^{\circ}$ ).

Table 6 Two-way ANOVA analysis of contact angle results

Source	$\mathbf{DF}$	Sum of squares	F ratio	Probability	
Type of resin	3	364.8	22.9	< 0.0001	
UV irradiation time	4	58.1	2.7	0.0328	
Type of resin					
×					
UV irradiation time	12	23.9	0.4	0.9693	
Error	100	529.8			

of 1,388, 1,360, and 1,320 cm<sup>-1</sup> could be assigned as CH<sub>3</sub>, CH<sub>3</sub>, and C-O respectively, but those of 810 and 740 cm<sup>-1</sup> could not be assigned.

## Contact angle

Figure 4 summarizes the contact angle results. Contact angles of the control specimens ranged from 81.5 to 90.2°. Two-way ANOVA revealed that the effects of UV irradiation time and resin type were statistically significant, but no significant interaction was observed (Table 6). The contact angle showed a tendency to increase with longer UV irradiation time, although Tukey's analysis indicated no significant differences among the groups. Among the resin types, PP composite resin showed a significantly smaller value (83.5°) than the other resins.

#### DISCUSSION

With a view to improving polymer surface characteristics for different applications (such as lithography and microelectronics), UV irradiation has been intensively studied in polymer science. Consequently, changes in morphological and chemical properties of the polymer surface have been reported. Amongst which, UV irradiation reportedly induces surface radicals that can recombine to form various types of networks and terminal groups<sup>15)</sup>. In other words, oxygen generated during UV irradiation has a fundamental influence on the surface modification of polymers.

Compared to UV irradiation without ozone<sup>15</sup>, UV irradiation with ozone has shown a significant effect on surface oxidation<sup>17</sup> with greater contact angle modification. With a UV lamp, the typical wave-

length range is 184.9-253.7 nm. The UV light of 184.9 nm creates ozone from oxygen in air, and this ozone is converted into atomic oxygen by UV light of 253.7nm<sup>16,17,19</sup>. As a result, selection of the UV source is an influential factor in surface modification. In the present study, the device used was an ordinary UV sterilizer in the dental office. The UV lamp emitted UV light with mostly a wavelength of 253.7 nm and with a small amount of 184.9 nm. Consequently, little ozone was created such that the effect of ozone was negligible in this study.

In the present study, compositions of the experimental unfilled resin and composite resins were identical to that of a commercial brand of artificial teeth<sup>3</sup>. In the enamel layer of composite resin artificial teeth, filler size was reported to range from 80  $\mu$ m to submicrons and inorganic filler content from 5.9 to 42.9 mass%<sup>1</sup>. Therefore, to the end of evaluating the effects of filler on shear bond strength (SBS) and contact angle, three types of silica fillers and a filler content of 20 mass% were selected. In addition, the effects of unfilled resin were also examined.

Results of the present study are summarized as follows: SBS increased after 1 minute to 1 hour of UV irradiation but decreased after 24 hours of UV irradiation; FTIR spectra did not change except after 24 hours of UV irradiation; and the contact angle showed a tendency to increase with longer UV irradiation time.

An SBS greater than 18 MPa has been reported to show acceptable clinical results<sup>4</sup>). For the control specimens before UV irradiation, their SBS values of 4.4 to 6.9 MPa were less than this suggested value, thus accounting for predominantly adhesive failure mode. These results thus indicated the necessity of surface treatment for the improvement of bonding strength.

At 1 and 10 minutes after UV irradiation, the SBS values were significantly increased and were greater than 18 MPa. Moreover, failure mode was mainly cohesive failure. These findings implied the likely changes in the UV-irradiated surface structure of the adherend, although no apparent changes could be detected in the FTIR spectra nor significant changes in the contact angle measurements. At this juncture, it must be mentioned that the typical probing depth of FTIR ranges from a few hundred nanometers to several micrometers — which is too deep for detecting chemical changes in the near-surface region<sup>15,18</sup>). A sensitive device that analyzes the shallower region of a surface, such as XPS<sup>16,18,19)</sup> or an atomic force microscope<sup>20)</sup>, would better detect the changes at the UV-irradiated surface.

UV irradiation, especially with ozone, has been reported to decrease the contact angle of distilled water on the irradiated surface<sup>15,16,18,19</sup>. UV irradiation causes surface radicals or low molecular weight oxidized materials (LMWOM) to be formed<sup>15,16,18,20</sup>, which might thus be responsible for changes in the contact angle. The LMWOM is more hydrophilic when the surface is UV-irradiated with ozone because a large number of hydrophilic species are formed<sup>15</sup>; conversely, UV irradiation without ozone creates mainly hydrophobic species<sup>15</sup>. Apart from the effect of UV/ozone on contact angle, the resultant UV effect is also polymer-dependent<sup>18</sup>. Therefore, discrepancies in contact angle values after UV irradiation between the present study and those of previous studies might be due to the intertwining effects of two factors: presence of ozone and the different polymers examined.

At this juncture, it must be clarified that the contact angle of distilled water suggests only the possibility of chemical changes in the surface<sup>15,16,18,19</sup>. It certainly does not predict the bond strength of the resin<sup>11</sup>. This is because the surface energy components of MMA and distilled water are completely different.

In the present study, SBS decreased after 1 and 24 hours of UV irradiation with corresponding increase in adhesive failure. On the contrary, the contact angle increased after 24 hours of UV irradiation, thereby suggesting that the surface had undergone some chemical changes after UV irradiation. Indeed, the FTIR spectra suggested a decrease of CH<sub>3</sub> and an increase of C-O, indicating therefore a degradation of the polymer chain<sup>15)</sup> and the existence of LMWO<sup>19)</sup>. In the same vein, the yellowish appearance of the specimens after 24-hour UV irradiation complemented the suggestion of chemical changes at the specimen surface<sup>21)</sup>. Taken together, these results strongly implied a decomposition or degradation of the resin surface. As a result, mechanical properties of the resin surface should presumably decrease - which meant that SBS should decrease and adhesive failure should increase.

Four types of experimental resins, one unfilled resin and three composite resins, were examined in the present study. The effect of filler/resin type was not significant in this study, except on contact angle measurement. PP composite resin showed the smallest contact angle among the four resins. PP filler was prepolymerized microfine silica filler with TMPT. Hence, it was possible that the presence of TMPT was responsible for the small contact angle values of PP composite resin.

In previous studies, recovery of polymers from UV irradiation effects has been discussed<sup>16,18)</sup>. With poly(dimethylsiloxane), UV irradiation effects have been evaluated with contact angle and scanning force microscopy measurements<sup>16)</sup>. Results suggested that the effects remained longer when UV irradiation time was longer. For example, the effect of 10-minute UV irradiation remained for 0.1 hour after irradiation,

while that of 30-minute irradiation remained for more than 50 days. Moreover, the effects of UV irradiation were attenuated after water washing probably because the LMWOM was removed by water<sup>18)</sup>. In the present study, properties of UV-irradiated surfaces were measured within 30 minutes after irradiation. Therefore, recovery from UV irradiation probably occurred in the present study. Nonetheless, the effects of UV irradiation were successfully detected and monitored by means of SBS testing, FTIR spectroscopy, and contact angle measurement.

According to the obtained findings, a relatively short period of UV irradiation — such as 1 minute to 10 minutes — was effective in improve the bonding efficacy of composite resin denture teeth.

Compared to surface treatments such as air abrasion and silanization, UV irradiation is one means which is less expensive and more easily applicable for bonding improvement. This is because a UV sterilizer is commonly used for disinfection of instruments in a dental office. Nonetheless, it must be pointed out that several factors of UV irradiation influence the bonding efficacy of composite resin artificial denture teeth — such as the distance between UV lamp and specimen surface, angle of specimen surface to the UV lamp, power intensity of the UV lamp, type of UV lamp, and absence or presence of Moreover, the effect of UV irradiation is ozone. expected only on the surface exposed to UV irradiation. Therefore, for restorations with complicated geometries, a special UV irradiation setup should be designed.

To date, the bonding durability after UV irradiation is yet to be clearly confirmed. Coupled with the many UV irradiation factors affecting bonding efficacy as well as the need to address restorations with complicated geometries, future research should indeed be carried out to tackle these problems.

# CONCLUSIONS

Within the limitations of the present study, the following conclusions were drawn based on the obtained results:

- 1. UV irradiation for 1 and 10 minutes significantly enhanced the shear bond strength regardless of resin type. Correspondingly, failure mode was mainly mixed and cohesive failures of the adherend.
- 2. UV irradiation for 24 hours did not significantly improve the shear bond strength regardless of resin type. Correspondingly, failure mode was mainly adhesive failure.

# ACKNOWLEDGEMENTS

The authors wish to express their sincere thanks and

gratitude to Associate Professor Hideharu Hirose from the Nihon University for FTIR measurement and Assistant Professor Akihiro Fujishima from the Showa University for contact angle measurement.

#### REFERENCES

- Loyaga-Rendon PG, Takahashi H, Hayakawa I, Iwasaki N. Compositional characteristics and hardness of acrylic and composite resin artificial teeth. J Prosthet Dent 2007; 98: 141-149.
- Suzuki S. *In vitro* wear of nano-composite denture teeth. J Prosthodont 2004; 13: 238-243.
- Abe Y, Sato Y, Akagawa Y, Ohkawa S. An *in vitro* study of high-strength resin posterior denture tooth wear. Int J Prosthodont 1997; 10: 28-34.
- 4) Lucena-Martin C, Gonzalez-Lopez S, Navajas-Rodriguez de Mondelo JM. The effect of various surface treatments and bonding agents on the repaired strength of heat-treated composites. J Prosthet Dent 2001; 86: 481-488.
- Tezvergil A, Lassila LVJ, Yli-Urpo Antti, Vallittu PK. Repair bond strength of restorative resin composite applied to fiber-reinforced composite substrate. Acta Odontol Scand 2004; 62: 51-60.
- 6) Papacchini F, Dall'Oca S, Chieffi N, Goracci C, Sadek FT, Suh BI, Tay FR, Ferrari M. Compositeto-composite microtensile bond strength in the repair of a microfilled hybrid resin: effect of surface treatment and oxygen inhibition. J Adhes Dent 2007; 9: 25-31.
- Brosh T, Pilo R, Bichacho N, Blustein R. Effect of combinations of surface treatments and bonding agents on the bond strength of repaired composites. J Prosthet Dent 1997; 77: 122-126.
- 8) Vano M, Goracci C, Monticelli F, Tognini F, Gabriele M, Tay FR, Ferrari M. The adhesion between fiber posts and composite resin cores: the evaluation of microtensile bond strength following various surface chemical treatments to posts. Inter Endod J 2006; 39: 31-39.
- Aksornmuang J, Foxton RM, Nakajima M, Tagami J. Microtensile bond strength of a dual-cure resin core material to glass and quartz fiber posts. J Dent 2004; 32: 443-450.
- Matinlinna JP, Lassila LVJ, Ozcan M, Yli-Urpo A, Vallittu PK. An introduction to silanes and their clinical applications in dentistry. Int J Prosthodont 2004; 17: 155-164.
- Monticelli F, Toledano M, Osorio R, Ferrari M. Effect of temperature on the silane coupling agents when bonding core resin to quartz fiber posts. Dent Mater 2006; 22: 1024-1028.
- 12) Shiau JY, Rasmussen ST, Phelps AE, Enlow DH, Wolf GR. Analysis of the shear bond strength of pretreated aged composites used in some indirect bonding techniques. J Dent Res 1993; 72: 1291-1297.
- 13) Ozcan M, Barbosa SH, Melo RM, Galhano GA, Bottino MA. Effect of surface conditioning methods on the microtensile bond strength of resin composite to composite after aging conditions. Dent Mater 2007; 23: 1276-1282.
- 14) Buchman A, Dodiuk H, Rotel M, Zahavi J. Laser-

induced adhesion enhancement of polymer composites and metal alloys. In: Polymer surface modification: relevance to adhesion, Mittal KL (ed.), VSP, Utrecht, 1996, pp.199-212.

- 15) Efimenko K, Wallace WE, Genzer J. Surface modification of Sylgard-184 poly(dimethyl siloxane) networks by ultraviolet and ultraviolet/ozone treatment. J Colloid Interface Sci 2002; 254: 306-315.
- 16) Hillborg H, Tomczak N, Olah A, Schönherr H, Vancso GJ. Nanoscale hydrophobic recovery: a chemical force microscopy study of UV/ozone-treated cross-linked poly(dimethylsiloxane). Langmuir 2004; 20: 785-794.
- 17) Ye HK, Gu ZY, Gracias D. Kinetics of ultraviolet plasma surface modification of poly(dimethylsiloxane) probed by sum frequency vibrational spectroscopy. Langmuir 2006; 22: 1863-1868.
- 18) Hill JM, Karbashewski E, Lin A, Strobel M, Walzak MJ. Effects of aging and washing on UV and ozone-treated poly(ethylene terephthalate) and polypro-pylene. In: Polymer surface modification: relevance to adhesion, Mittal KL (ed.), VSP, Utrecht, 1996, pp.273-289.
- Macmanus LF, Walzak MJ, McIntyre NS. Study of ultraviolet light and ozone surface modification of polypropylene. J Polym Chem 1999; 37: 2489-2501.

- 20) Nie HY, Walzak MJ, McIntyre NS. Atomic force microscopy study of UV/ozone treated polypropylene films. In: Polymer surface modification: relevance to adhesion, Volume 2, Mittal KL (ed.), VSP, Utrecht, 2000, pp.377-392.
- 21) Zhang JY, Esron H, Kogelschatz U, Emig G. Modification of polymers with UV excimer radiation from lasers and lamps. In: Polymer surface modification: relevance to adhesion, Mittal KL (ed.), VSP, Utrecht, 1996, pp.153-184.
- 22) Wheeler CR, Fried D, Featherstone JDB, Watanabe LG. Irradiation of dental enamel with Q-switched I=355-nm laser pulses: surface morphology, fluoride adsorption, adhesion to composite resin. Lasers Surg Med 2003; 32: 310-317.
- 23) Chang JCH, Ossof SF, Lobe DC, Dorfman MH, Dumais CM, Qualls RG, Johnson JD. UV inactivation of pathogenic and indicator microorganisms. Appl Environ Microbiol 1985; 49: 1361-1365.
- 24) Ishida H, Nahara Y, Tamamoto, Hamada T. The fungicidal effect of ultraviolet light on impression materials. J Prosthet Dent 1991; 65: 532-535.
- 25) Devine DA, Keech AP, Wood DJ, Killington RA, Boyes H, Doubleday B, Marsh PD. Ultraviolet disinfection with a novel microwave-powered device. J Applied Microbiol 2001; 91: 786-794.