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原　　著

A Cure Ratio at Deeper Layer in Dental Composite Resin

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ABSTRACT

This study examined curing performance at deeper layer from the exposure surface and determined the cure ratio when cured by visible light. The samples proposed in this study were obtained at each level in VLC composite resins. More cure ratio was achieved at deeper layer when larger irradiation time was exposured. The relation between depth of cure and integral irradiance was clarified using the curing at each deeper layer. The described method here was effective to determine the depth of cure in dental VLC composite resins.

INTRODUCTION

Dental composite resins cured by visible light source have been used in clinical service[1-5]. Visible light-cured (VLC) composite resins contained camphorquinone (CQ) and reducing agent in the resin phase and also including fillers[6]. CQ was activated in the range of visible light wavelength[7], and the spectra wavelength emitted by VL source was between 400 and 650 nm[4,8-10]. The visible light passes through the translucent composite resin and the extent of cure was influenced by the light intensity[11]. The monomer composition of the resin and the concentration of catalyst present within dental composite resins affected the degree of curing (polymerization)[11,12]. To evaluate the extent of cure, sample preparation was carried out using a cylindrical mould (4 mm diameter×5 mm height) and sectioned with a diamond saw, polished on 600 grid SiC paper followed by 1 μm alumina polishing paste[13-15]. Because the top surface exposured by VL source was removed mechanically, the value of surface hardness was not measured and also residual monomer at the layer including top surface or exposured surface was not obtained. The curing performance is needed to improve the resins because of unpolymerized resins at deeper depth of VLC composite resins[4]. Therefore, using monomer mixture of bis-GMA (2, 2-bis [4-(2-hydroxy-3-methacryloyloxy) phenyl] propane) and TEGDMA (triethylene glycol dimethacrylate), the extent of VL curing was examined at each layer[16,17]. Differential scanning calorimetry (DSC) technique also determined that the resin containing aliphatic ternary amines was better than in aromatic tertiary amine[17]. More detailed method to determine a cure ratio at deeper layer is needed using dental composite resin with known photo-initiator and reducing agent.

This study was therefore to use a model for sample preparation to evaluate a cure ratio in dental composite resin and to clarify depth of cure using the proposed method for analysis. The influence of irradiation time to calculate the integral irradiance on the depth of cure was examined according to the calculation method[17].

MATERIALS AND METHODS

The resin material tested was Occlusin (ICI Co, UK), because it is used in our study[18] and also usually popular as the world-wide use. It is a hybrid type composite resin which includes filler content=86.7 (1.21) wt% and DMAEMA=0.86 (0.02) wt% as a mean value (standard deviation)[18]. A VL source tested was Luxor Light (ICI Co, UK) used typically for Occlusin. The standard light source for the VL unit is a tungsten halogen lamp fitted

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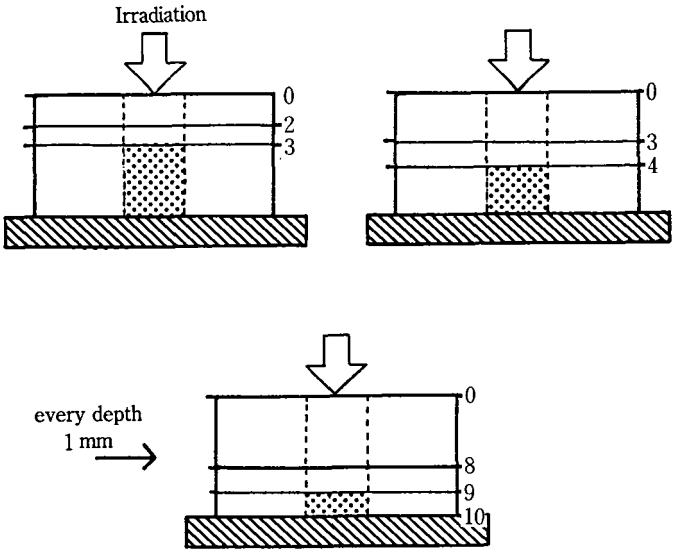

Figure 1 Sample preparation to obtain a cure ratio at each layer, using a teflon mould.

with an integral reflector. Irradiance measurement was carried out using electric power of lamp of 50 W. The value of spectral irradiance ($mW/cm^2 \cdot nm$) in the wavelength range between 400 and 650 nm was normalized to 1 cm^2 area of the fiber-optic tip. The peak of wavelength component is found at 460 nm in this VL source.

At each layer the cylidrical specimen to examine a cure ratio was obtained as indicated in Fig. 1, using a teflon mould (5 mm diameter) placed on a slide glass (thickness =1.5 mm). At each depth from the exposured surface where plastic strip (0.05 mm) was placed, the ratio was measured. At each layer polyethylene sheet (0.01 mm) was set. To evaluate the ratio as a curing performance of the composites, the set composite was immersed into methanol and measured the residual weight using gas-liquid chromatograph (GC; GC 9A, Shimadzu Co, Kyoto). The condition of GC analysis was as follows: Detector, F.I.D. (hydrogen flame), capillary column (CBP-1 W24-300, 25 m×0.53 mm) and carrier gas (25 mL/min He). The other conditions were detailed in[19,20].

A cure ratio in the composite resin at each depth was defined in this study as follows.

$$C_c\ (wt\%)=((C+D)/(A+B))\times 100$$

where A and B mean resin matrix only (wt%) and filler content (wt%) in the set composite resin, and C and D resin matrix and filler (wt%) in drying them after methanol immersion for 7 days at 37°C.

A cure ratio in resin matrix (C_r) and the weight after firing 900°C (W_f) at each depth were defined as follows.

$$C_r\ (wt\%)=(C/A)\times 100$$
$$W_f\ (wt\%)=(D/(C+D))\times 100$$

RESULTS AND DISCUSSION

Figs. 2 and 3 show the cure ratio at each depth, to represent depth of cure in the composite resin and the resin matrix, indicating that the ratio decreased with deeper depths. According to this result, the magnitude of cure ratio in the composite (Fig. 2) and resin matrix (Fig. 3) were different every the layer from the top surface, and also depended on the irradiation time. The cured layer with 90 wt% (cure ratio) was 0–5 (irradiation time=20 sec), 0–7 (40 sec) and 0–8 (60 sec) mm in the composite resin, and 0–2 (20 sec), 0–3 (40 sec) and 0–5 mm (60 sec) in the resin matrix. The cured layer in this composite resin by Luxor VL source was deeper as compared with the other VL source[17]. W_f value at each depth was not significantly different when irradiation time was 60 sec (for example, between 0–2 and 2–3 mm, or 0–3 and 3–4 mm, etc), except between 0–8 and 8–9 mm. At 20 sec irradiation the unpolymerized resin was found at the layer with more than 8 mm from the top surface.

Fig. 4 shows depth of cure in the composite resin with respect to logarithm of integral irradiance at irradiation times 20, 40 and 60 sec when cured by VL source. The

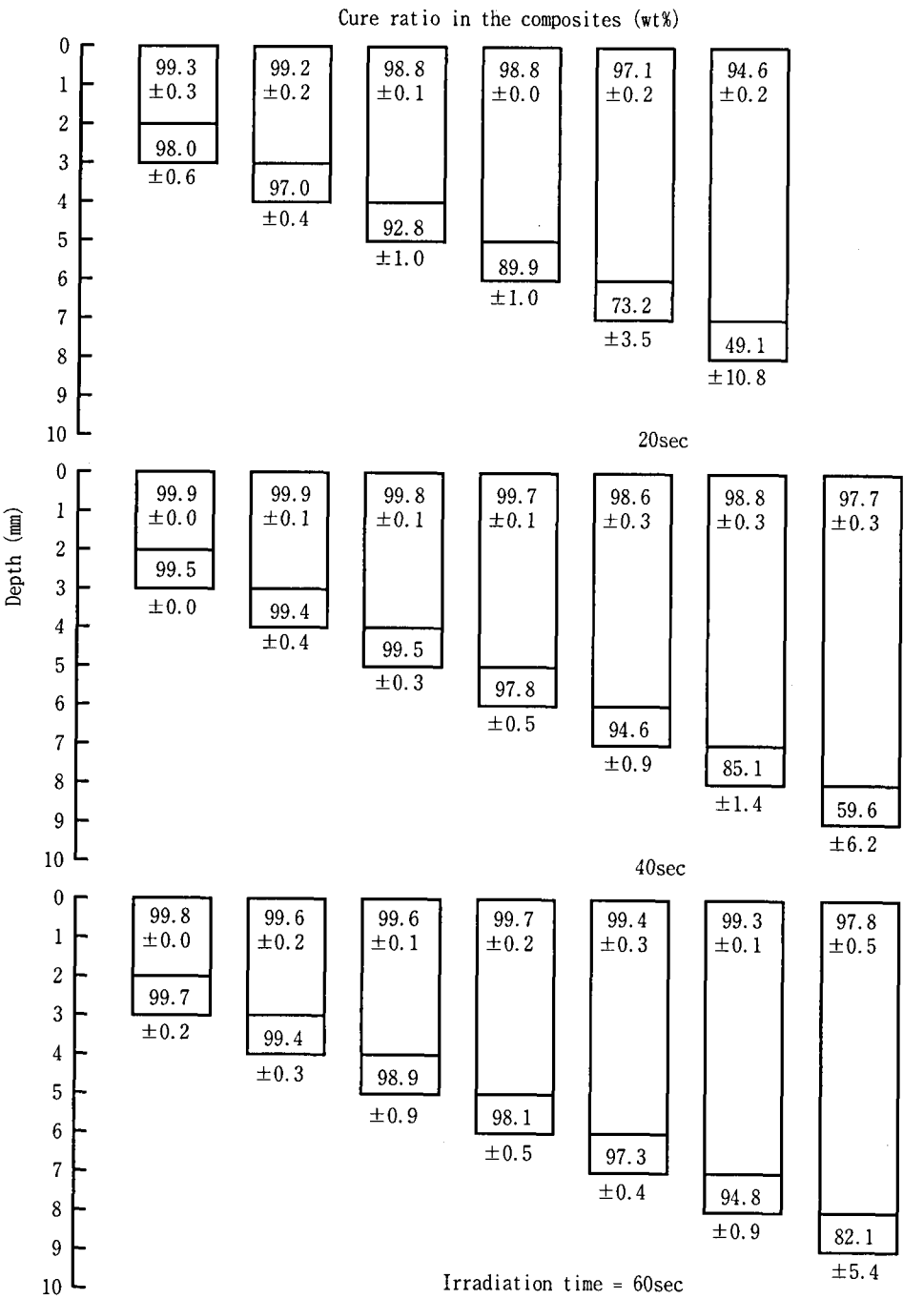

Figure 2 Cure ratio at each depth from the top surface in the composite resin.

depth of cure (cured layer) was defined as the depth of the layer where the cure ratio was more than 90 wt%[12-15]. The positive correlation between depth of cure and logarithm of integral irradiance was found with r=0.999.

This model exhibited that one commercial composite resin was evaluated by new method to obtain a cure ratio at deeper layer. Because VLC resin was cured by VL source, the characteristics of VL activating units including filter, fibre-optic, tip diameter, a kind of fibres (glass fibre and plastic fibre), light tube and light source was discussed

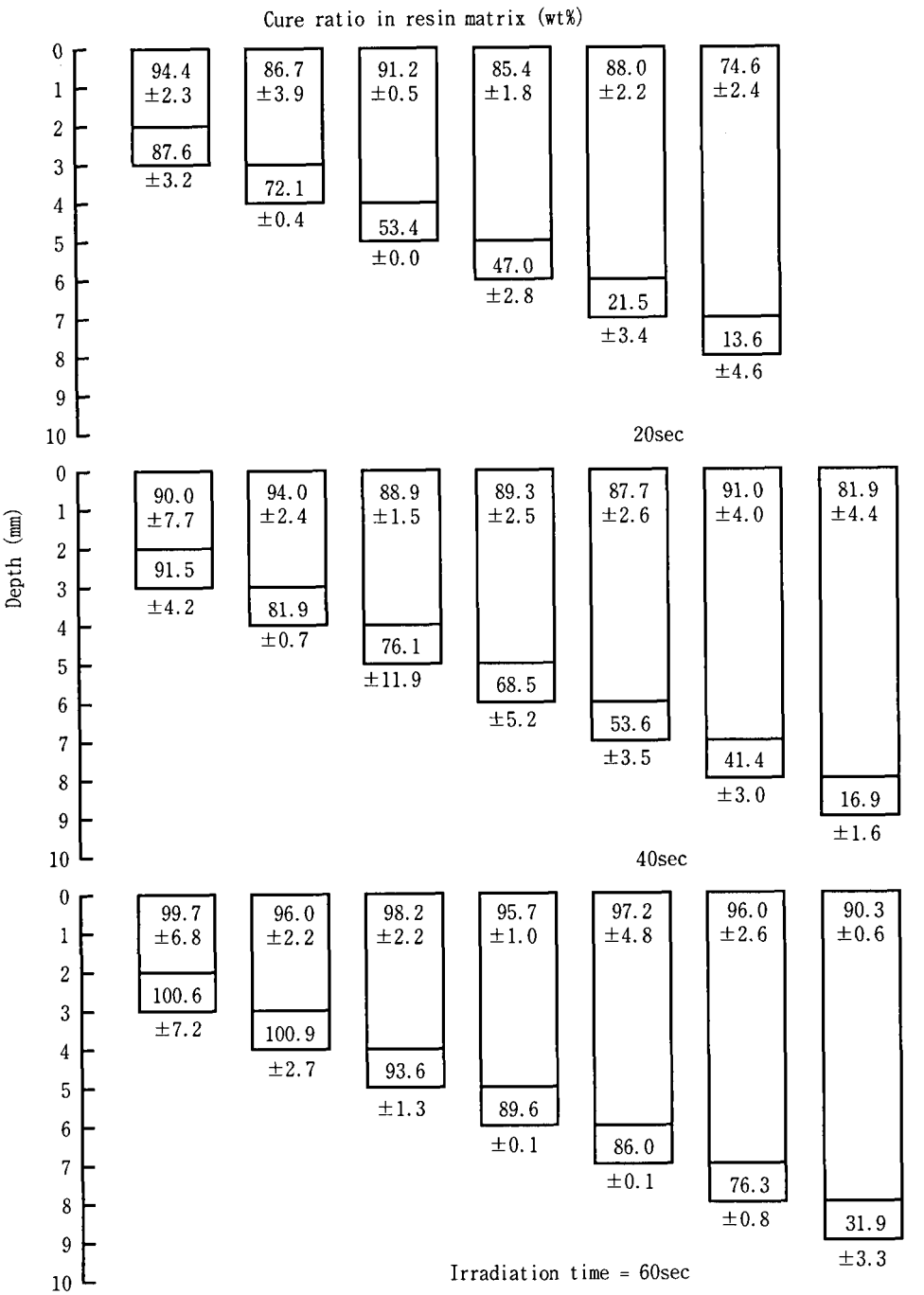

Figure 3 Cure ratio at each depth in the resin matrix.

as described already[17]. In this study the sample preparation for depth of cure was discussed by each deeper depth from the exposured surface. The VL source were examined by light intensity such as illuminating power or irradiance[17]. The extent of cure at different depths of VL-activated dental composite resins was dependent upon the irradiation time in visible light source (Figs. 2, 3), and also controlled by integral irradiance ($\int_{400}^{650} I\, d\lambda \cdot t$) including irradiation time (t) (Fig. 4).

Table 1 The amount of weight at each depth after firing at 900°C in the composite resin. Irradiation time=20, 40 and 60 sec.

Depth (mm)		Weight (wt%) Irradiation time (sec) 20	40	60
1	0-2	87.2±0.3	87.9±1.0	86.5±0.9
	2-3	88.1±0.4	91.5±4.2	86.2±1.0
2	0-3	88.3±0.6	86.8±0.8	86.8±0.1
	3-4	89.9±0.1	88.9±0.1	86.3±0.5
3	0-4	87.6±0.6	88.0±0.2	86.7±0.3
	4-5	92.3±0.1	89.7±1.6	87.2±0.4
4	0-5	88.4±0.3	88.0±0.4	87.1±0.2
	5-6	93.0±0.5	87.3±2.5	87.8±0.2
5	0-6	88.4±0.3	88.0±0.4	86.9±0.7
	6-7	96.2±0.5	90.5±0.3	88.1±0.0
6	0-7	89.4±0.4	88.0±0.4	87.0±3.3
	7-8	74.6±2.4	93.5±0.3	89.3±0.2
7	0-8	——	87.6±0.6	87.6±0.2
	8-9	——	96.4±0.0	94.8±0.2

SUMMARY

The sample to determine the curing performance at deeper layer from the exposure surface was prepared and the values of cure ratio were obtained in VLC composite resin and resin matrix. At each layer a cure ratio was measured, showing that more cure ratio was obtained at deeper layer when the larger irradiation time was exposured. These methods were effective to evaluate the curing at each deeper layer in VLC resin and to clarify the relation between depth of cure and integral irradiance. Detailed information on the other composite resins is greatly needed. This present study confirmed that the described analyses including sample preparation and cure ratio were significantly recommended for the depth of cure in dental composite resins.

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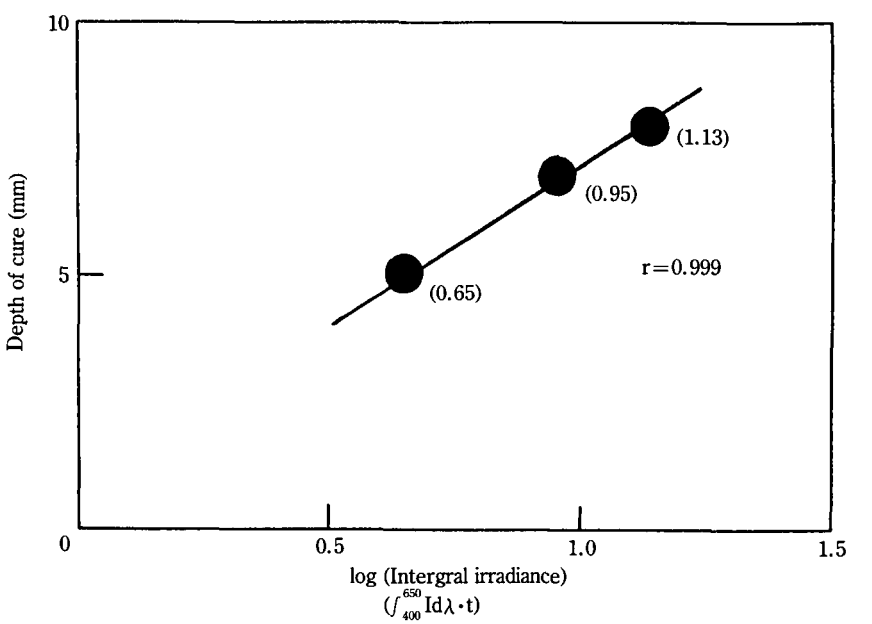

Figure 4 A relation between depth of cure in the composite resin and the logarithm of integral irradiance between 400 to 650 nm. Irradiation time=20,40 and 60 sec.

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