

# The Effects of Various Light Intensity on the Polymerization of Resin Composites

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## ABSTRACT

### 광조사 강도의 변화에 따른 광중합 복합레진의 중합반응에 관한 연구

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기존의 광중합기는 높은 광강도를 제공함으로써 광중합 복합레진을 최대한 단축된 시간내의 중합을 목표로 하였다. 이러한 높은 광강도는 복합레진의 중합깊이, 중합률면에서는 우수하나 중합 반응속도가 빠름으로 인해 중합시 응력 발생이 높아진다는 일련의 보고가 있다. 최근에는 광중합 속도를 늦춤으로써 변연적합도 및 중합시 응력 발생을 낮추는 새로운 중합방법들이 제시되고 있다. 이에 본 실험에서는 광조사 강도의 변화가 광중합 복합레진의 중합반응 과정에 미치는 영향 및 중합된 복합레진의 중합률에 대한 영향을 분석하고자 하였다. 5개의 혼합형 광중합 복합레진 (Z-100, Spectrum, Z-250, Clearfil AP-X, P-60)을 사용하였으며 중합시 적용된 광조사 강도에 따라 6개의 실험군으로 정의하였다. 실험군과 이에 따른 광조사 방법은 다음과 같다. 1군은 110mW/cm<sup>2</sup>로 40초 중합, 2군 210mW/cm<sup>2</sup>로 40초 중합, 3군 410mW/cm<sup>2</sup>로 40초 중합, 4군 620mW/cm<sup>2</sup>로 40초 중합, 5군 110mW/cm<sup>2</sup>로 10초 중합 후 1분 뒤 620mW/cm<sup>2</sup>로 30초 중합, 6군 210mW/cm<sup>2</sup>로 10초 중합 후 1분 뒤 410mW/cm<sup>2</sup>로 30초 중합하였다. 광중합시 중합반응 양상에 관한 분석은 시차주사 열계량기를 이용하여 37℃ 항온상태에서 10분간의 열흐름곡선을 기록하였다. 기록된 열흐름곡선에서 중합 반응시 나타나는 중합열 및 최대 중합열에 이르는 시간을 기록하여 중합반응 속도를 측정하였다. 중합된 복합레진의 중합률은 Fourier Transform Infrared Spectrometer (FTIR)를 이용하였으며 2mm 두께의 복합레진 하방에서의 중합률을 측정하였다. 측정된 결과는 ANOVA 및 Student-Newman-Keuls 방법을 이용하여 유의성을 검증하였다. 실험결과 다음과 같은 결론을 얻었다.

1. 광중합 복합레진 중합시 광조사 강도가 증가할수록 중합열은 증가하였으나 통계적 유의성은 보이지 않았다 (p) 0.05).
2. 최대 중합열에 이르는 시간은 광조사 강도가 증가할수록 단축되었다. 이단계 중합방법을 사용한 경우 중합반응 속도를 감소시킬 수 있음을 보였다.
3. 광조사 강도가 증가할수록 중합률은 증가하였다. 이단계 중합방법을 사용한 경우 연속적인 고광강도를 사용한 경우와 유사한 높은 중합률을 보였다.
4. 중합률면에서 광중합 복합레진의 중합시 400mW/cm<sup>2</sup> 이상의 광강도가 필요한 것으로 나타났다.

**주요어 :** 광중합 복합레진, 중합열, 중합반응속도, 중합률

## I. INTRODUCTION

Using visible light to initiate the polymerization process of composites has changed operative den-

tristry. Light cured composites have become an indispensable tool in the dental office. Early deficiencies of composites included color stability, incomplete cure, microleakage, low wear resistance and high stresses

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caused by polymerization shrinkage<sup>1)</sup>. To resolve some of those problems, it has been suggested that a curing unit with high light intensity should be used in the curing of visible light-activated resin composites. Lutz(1992) proposed that a subsequent period of higher light intensity is necessary for achieving a sufficient depth of cure, a high degree of polymerization and high quality mechanical properties<sup>2)</sup>. These traditional concepts, however, are being questioned. Recent publications have shown that initial high light intensity during curing may not produce the best results with regard to polymerization shrinkage stress or marginal adaptation<sup>3,6,8-11)</sup>.

Because conventional light curing units provide the maximum light intensity instantly, they produce a large number of free radicals that cause hardening of the resin composites in a few seconds. This mass polymerization brings about an increase in the stiffness of the material too early. Thus, the ability of the material to flow is reduced<sup>9)</sup>.

The recommendation to use high light intensity during curing has been based on physical properties of cured materials. However, it fails to consider the negative influence of continuous high intensity lights on stress development<sup>8)</sup>. Furthermore, a linear relationship between light intensity and polymerization contraction has been demonstrated by Sakaguchi et al. (1992)<sup>4)</sup>. A more intense light source may produce greater polymerization shrinkage stress<sup>4,5)</sup>. Nonetheless, high intensity light is necessary to achieve good depth of cure and a high degree of polymerization in the material. The degree of polymerization is important for several reasons: A high percentage of unreacted methacrylate groups may result in poor mechanical properties, chemical degradation and color changes in polymerized resin composites<sup>22)</sup>.

To ensure optimal polymerization of resin composites without sacrificing the ability of the composite to flow during the curing process, several new approaches are being investigated.

Initial curing with low intensity light followed by full curing with high intensity of light (so called two-step curing<sup>10)</sup>, soft start polymerization<sup>11)</sup> or pulse delay curing<sup>13)</sup> has the advantage of reducing the speed of polymerization in the early setting phase. Molecules then have the possibility of moving into a

new position to compensate for internal stress.

Recent studies have shown that the marginal adaptation of light-activated resin composites can be improved by curing the material slowly with reduced intensity light<sup>3,6,8,9)</sup>. Curing reactions are accompanied not only by significant polymerization stresses, but also by heat generation due to the exothermic nature of the polymerization reaction and the heat output of the light curing unit<sup>12-14)</sup>. By analyzing heat changes during the curing process of the resin composite, polymerization dynamics can be obtained, e.g. speed of polymerization and heat of cure.

To evaluate the effects of light intensity variation on the polymerization of composite resins, two experiments were performed in this study. First, thermal analysis of composite resins during the polymerization reaction was recorded by a differential scanning calorimeter (DSC). DSC is probably the most widely used and most familiar of all the thermal analysis techniques<sup>13)</sup>. This equipment measures heat flow as a function of time<sup>16)</sup>. It allows for rapid assessment of speed of polymerization and is ideally suited for the study of curing kinetics of composite resins (Abadie, 1989). Second, the degree of polymerization was analyzed with a Fourier Transform Infrared Spectrometer (FTIR)<sup>19,21-23)</sup>.

The purpose of this study is to use these two methods to determine the effect of light intensity variation on the polymerization process.

## II . MATERIALS AND METHODS

Materials tested in this study are listed in the Table 1.

The A2 shade of each product was used in this experiment except for P-60 because the A2 shade is not available in this product.

In each test, 6 experimental groups were defined according to the light intensity applied during the polymerization process. The visible light curing unit used for composite resin curing was the Variable Intensity Polymerizer (VIP, Bisco, Schaumburg, IL, USA), which is programmable in both light intensity and polymerization time. The light intensity was measured with a Demetron radiometer (Demetron Research Co., Danbury, CT, USA).

Test protocols for the polymerization of composite resins in each group were the following. Groups 1 and 2 were low light intensity polymerization groups and groups 3 and 4 were high light intensity polymerization groups. Groups 5 and 6 were two-step polymerization groups, which were polymerized with low light intensity followed by high light intensity. The total curing time for all the groups was 40s. Before final cure with high light intensity, there was a 1 minute waiting period.

The reason for the waiting period is that preliminary study with DSC showed the time to return to base line in heat flow is about 1 minute later after light irradiation has stopped. This waiting period may also allow for stress reduction in the material itself.

1. Thermal analysis with DSC system

A model DSC 7 system (Perkin-Elmer, Norwalk, CT, USA) was used to measure the thermal changes during the polymerization process in a static air environment while a 37C isotherm was maintained.

Samples of composite resin were placed in a pre-weighed aluminum sample pan. The weights of the sample used was 48.50.5mg. After preparation, each sample was transferred immediately to the calorimeter cell. Composite resin previously fully cured (at 620mW/cm<sup>2</sup> for 60s) and with the same weight was placed in the reference calorimeter cell to minimize influences from heat of cure in reference sample.

For the stability of the light curing tips during irradiation, a guiding stand with two holes was made and the hole diameter was the same as the light curing tip diameter. After the guiding stand was placed above the sample and reference pans, light curing tips were positioned in each hole. Then the sample pan and the reference pan were simultaneously exposed to the curing lights for 40s according to each test protocol. After heat flow stopped, the cured sample pan and reference pan were exposed to the light according to the each test protocol two more times to measure the base line heat associated with light exposure.<sup>20</sup> The DSC curve was obtained for 10 minutes at a 37C isothermal condition. In the DSC curve, the area under the peak is proportional to the

Test protocols

Group	Curing methods	Total energy
Group 1	Cured at 110mW/cm <sup>2</sup> for 40s	4,400mW/cm <sup>2</sup>
Group 2	Cured at 210mW/cm <sup>2</sup> for 40s	8,400mW/cm <sup>2</sup>
Group 3	Cured at 410mW/cm <sup>2</sup> for 40s	16,400mW/cm <sup>2</sup>
Group 4	Cured at 620mW/cm <sup>2</sup> for 40s	24,800mW/cm <sup>2</sup>
Group 5	Cured at 110mW/cm <sup>2</sup> for 10s	19,700mW/cm <sup>2</sup>
	1 minute later	
	Cured at 620mW/cm <sup>2</sup> for 30s	
Group 6	Cured at 210mW/cm <sup>2</sup> for 10s	14,400mW/cm <sup>2</sup>
	1 minute later	
	Cured at 410mW/cm <sup>2</sup> for 30s	

Table 1. Resin composites tested in this study

Products	Shade	Type	Batch number	Manufacturer
Z-100	A2	Hybrid	5095A2	3M, St.Paul, MN, U.S.A
Spectrum	A2	Hybrid	606.05.202	Dentsply,Gmbh,Germany
Z-250	A2	Hybrid	1370A2	3M, St.Paul, MN, U.S.A
Clearfil AP-X	A2	Hybrid	00539A	Kuraray, Osaka, Japan
P-60	A3	Hybrid	8100A3	3M, St.Paul, MN, U.S.A

heat of reaction<sup>13)</sup>. The heat of cure was determined by measuring the area under the curve produced during the light curing and subtracting the average of two baseline heat output areas. The time to reach the maximum rate of heat output was determined to compare the speed of the polymerization process in each group. Each test was repeated 3 times and averaged. The results were analyzed by One-way ANOVA analysis and multiple comparison was done with Student-Newman-Keuls method.

## 2. Measurement of degree of polymerization with Fourier Transform Infrared Spectrometer (FTIR)

The degree of polymerization was analyzed with a Fourier Transform Infrared Spectrometer (FTIR). Spectra were recorded on a Spectrum One (Perkin-Elmer, Norwalk, CT, USA). The samples were examined by the Attenuated Total Reflectance (ATR) method using an ATR unit with a zinc selenide crystal. For the sample preparation on the ATR unit, a metal plate (80×10×2mm) with a window (10×8×2mm) in the center was fabricated. After the metal plate was placed on the ATR unit, composite resins were applied through the window. Then, the top of the window was covered with a mylar strip and slight pressure was applied with a glass plate in order to place the composite resin in the window evenly. Twenty scan pairs were acquired over a 440~4,400cm<sup>-1</sup> range. The spectra were recorded in transmission mode and then converted to absorbance mode. The intensities of the C=C absorbance peak at 1638cm<sup>-1</sup> and the cc reference peak at 1609cm<sup>-1</sup> were measured using a standard baseline technique (Heigl et al., 1947)<sup>21)</sup>. For groups 1 to 4, the spectra were obtained before and after curing. For the groups 5 and 6 three spectra were obtained: The first spectra was obtained before curing, the second after initial curing with low light intensity for 10s, and the third after curing with high light intensity for 30s. The peak in the absorbance band at 1638cm<sup>-1</sup> was attributed to C=C stretching vibrations of the aliphatic dimethacrylate type polymers. The aromatic CC peak at 1609cm<sup>-1</sup> originates from the aromatic bonds of the benzene rings in the monomer molecules. Its intensity remains unchanged during the

polymerization reactions. The ratio of the absorbance intensities of aliphatic C=C/aromatic CC is compared before and after polymerization by the following equation to determine the percentage of unreacted C=C bonds remaining (Ferracane & Greener, 1984)<sup>22)</sup>.

$$(\% \text{ C=C}) = \frac{[\text{Abs (C=C)/Abs (CC)}]_{\text{polymer}}}{[\text{Abs (C=C)/Abs (CC)}]_{\text{monomer}}}$$

The degree of polymerization was obtained by subtracting the residual % of carbon double bonds from 100%. The procedure was repeated 3 times for each composite resin and averaged.

## III . RESULTS

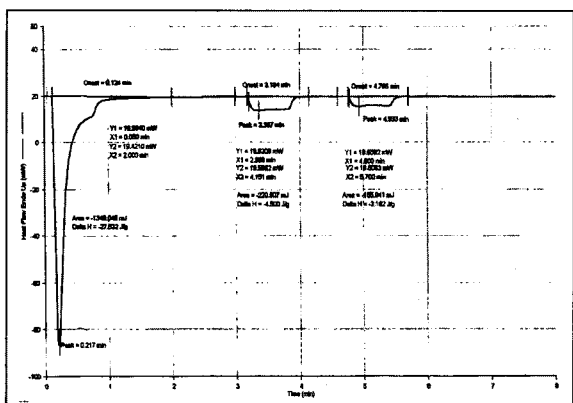
The heat of cure of the test materials measured by the differential scanning calorimeter is shown in Table 2. Typical heat flow curves during the polymerization process are shown in Fig. 1 and 2.

For groups 1 to 4, as the light intensity increased the heat of cure also increased but the change was not significantly different between group 3 and 4 ( $p > 0.05$ ). This indicates that if the light energy applied to polymerize composite resin is adequate, additional light exposure does not generate significant additional heat of polymerization. The most interesting observation in these experiments was that groups 5 and 6 (two step light curing protocol) showed less heat of cure than other groups with comparable total light exposure. Also, there was not a statistically significant difference between group 5 and 6 ( $p > 0.05$ ). The heat flow curve shows that when these groups were initially cured with low light intensity for a short time, the heat change was less than for groups 1 to 4. After the heat change associated with the initial curing period subsided, the second cure with high intensity was applied. However, when this second heat flow peak is compared to the base line heat flow from the curing light (subsequent two heat flow peaks), it is clear that there was little additional exotherm from the polymerization of the composite resin.

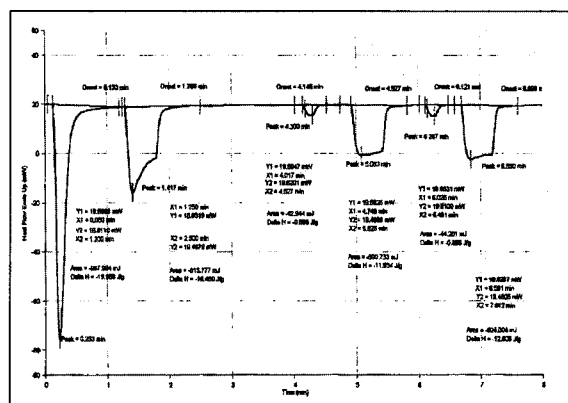
In groups 1 to 4, most of the samples showed maximum heat flow during the first 20 seconds. This

**Table 2.** Mean values of heat of cure of composite resins measured by DSC. Standard deviations are in parentheses.

Group	Heat of Cure (J/g)				
	Z-100	Clearfil AP-X	Z-250	Spectrum	P-60
1	29.8 (0.2)	23.4 (0.4)	25.7 (0.9)	33.8 (0.2)	24 (0.4)
2	31.6 (0.8)	23.5 (0.3)	27.5 (2.4)	34.5 (1.1)	24.3 (0.7)
3	32.6 (0.8)	25.6 (0.6)	30.2 (3.3)	34.5 (2.1)	26.9 (0.7)
4	33 (2.4)	26.7 (1.4)	29.1 (1.7)	34.3 (1.4)	27 (1.0)
5	29.8 (1.8)	24.1 (0.3)	24.4 (0.5)	30.5 (0.5)	24.6 (1.2)
6	28.2 (0.3)	21.8 (0.4)	25.5 (0.5)	30.9 (1.6)	23.7 (0.8)



**Fig. 1.** Heat flow curve of P-60 cured at 110mW/cm<sup>2</sup> for 40s.



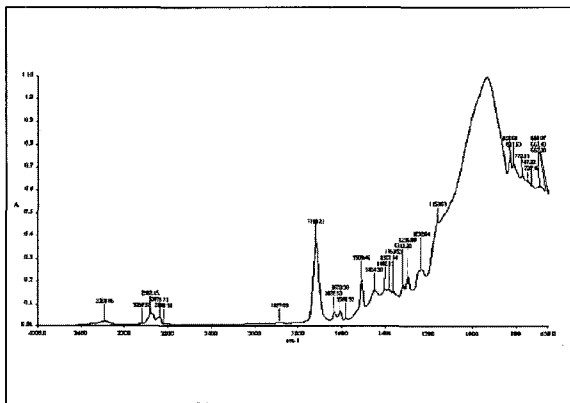
**Fig. 2.** Heat flow curve of P-60 cured with 110mW/cm<sup>2</sup> for 10s and, after 1 minute, cured again with 620mW/cm<sup>2</sup> for 30s.

means that the polymerization process is the most vigorous in the first 20 seconds so the initial curing light mode is very important. The heat of cure varied widely among the test materials because of differences in filler loading, the volume of photoinitiator, and the microstructure of the composites. Spectrum has the largest heat of cure and followed by Z-100, Z-250, P-60 and Clearfil AP-X. The time to reach peak heat output decreased with increasing curing light intensity. This means that the more intense the curing light, the faster the polymerization reaction occurred. The time to reach peak temperature in

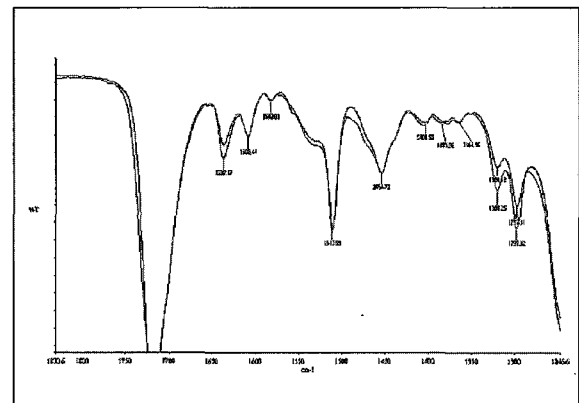
each composite is shown in Table 3. The degree of polymerization was determined with the spectra obtained by FTIR. Representative spectra of Clearfil AP-X obtained in this experiment are shown in Fig. 3. The results of the degree of polymerization measurements are listed in Table 4. As the curing light intensity increased from group 1 to 4, the degree of polymerization also increased significantly ( $p < 0.01$ ). In the two step curing groups, the initial curing with low curing light intensity for 10s produced a very low degree of polymerization in C=C bond. However, the secondary cure with high intensity for 30s produced a

**Table 3.** Mean time to reach peak heat output (in seconds)

Group	Z-100	Clearfil AP-X	Z-250	Spectrum	P-60
1	16.34	24.66	13.02	14.66	13.02
2	14.32	18.00	12.00	13.34	11.66
3	13.34	15.00	11.32	13.02	11.32
4	12.77	13.34	10.66	11.66	10.98
5	16.02	16.98	13.02	14.66	13.66
6	13.34	16.34	12.34	12.68	12.34



**Fig. 3.** Representative spectrum of Clearfil AP-X before curing. Note the C=C peak at  $1638\text{cm}^{-1}$  and the cc reference peak at  $1609\text{ cm}^{-1}$ .



**Fig. 4.** Close-up of spectrum after curing with  $410\text{mw/cm}$  for 40s. To calculate the degree of conversion, the spectra obtained before and after curing are superimposed. Note the decrease in the C=C peak at  $1638\text{cm}^{-1}$  area.

**Table 4.** Mean degree of polymerization for each curing protocol measured by Spectrum One. Standard deviations are in parentheses.

Group	Curing condition	Z-100	Clearfil AP-X	Z-250	Spectrum	P-60
1	110mW/40S	24.27% (1.03)	29.36% (1.04)	24.57% (4.72)	28.35% (1.67)	19.81% (1.12)
2	210mW/40S	28.93% (1.82)	37.89% (0.85)	30.68% (1.40)	34.65% (0.41)	27.94% (0.44)
3	410mW/40S	30.58% (1.14)	41.88% (0.85)	36.43% (1.40)	35.31% (1.49)	33.62% (1.97)
4	620mW/40S	33.70% (3.46)	45.89% (1.41)	37.67% (1.95)	39.04% (0.32)	34.15% (1.58)
5	110mW/10S	7.64% (1.21)	3.26% (0.58)	10.12% (2.46)	8.76% (0.31)	9.04% (1.13)
	110mW/10S, 620mW/30S	33.76% (1.17)	46.17% (1.35)	36.49% (0.17)	37.90% (0.48)	33.31% (1.48)
6	210mW/10S	15.47% (1.24)	13.65% (4.46)	19.84% (3.00)	20.60% (2.57)	18.21% (2.23)
	210mW/10S, 410mW/30S	33.57% (1.01)	43.96% (0.80)	36.95% (2.13)	38.36% (1.40)	33.20% (1.35)

higher degree of polymerization ratio. The reason is that the degree of polymerization is proportional to the total curing energy delivered to the composite resin.

In most of the test samples, group 4 has the greatest degree of conversion because its total irradiation energy is the largest among the groups (24,800mW/cm<sup>2</sup>). But the total degree of polymerization among groups 4, 5 and 6 was not significantly different. Because the measurements in this study are made at the bottom layer of a 2mm thick composite resin increment, the degree of polymerization in this test is somewhat lower than other results reported<sup>23,30</sup>. Clearfil AP-X has the largest degree of polymerization followed by Spectrum, Z-250, Z-100 and P-60. Z-250 vs. P-60 difference was probably due to the darker shade of P-60. However, Z-100 and P-60 showed a similar degree of polymerization.

#### IV. DISCUSSION

Curing composite resin optimally is an important clinical issue. However, the many factors involved in polymerization reactions make the description of the ideal curing method difficult. The importance of light intensity should be emphasized in clinical practice. It has been found that changes in light intensity may result in significant change in resin cure within the bulk of the restoration<sup>27</sup>. It has been shown that higher light intensities are needed to achieve superior physical and mechanical properties in general<sup>4</sup>. Although high curing light intensity provides better physical properties and better degree of polymerization, it also produces higher contraction stresses during polymerization<sup>5</sup>. It may lead to microleakage, marginal discoloration, and a failed restoration. The contraction rate of a light cured composite is highest during the initial stage of polymerization reaction<sup>5</sup>. This is clinically significant because it means that the integrity of the composite-tooth interface is quickly challenged during the early phases of polymerization. Investigators have suggested that a gel point exists in the polymerization process of resin composites. The gel point is defined as the moment at which the materials can no longer provide viscous flow in response to curing contraction<sup>24</sup>. Before this

gel point, the resin crosslink is relatively weak and the movement of molecules is easy enough to compensate for the polymerization stresses at the composite-tooth interfaces<sup>24</sup>. As the curing proceeds, the stiffness of the resin composite increases but the flow capacity is reduced. Reduced rates of polymerization may delay the gel point and allow increased flow of the material, which is favorable for marginal integrity<sup>3,25,26</sup>. Uno & Assumussn(1991), Goracci(1996)<sup>9</sup>, Feilzer(1995)<sup>6</sup>, Unterbrink(1995)<sup>8</sup>, and Koran (1998)<sup>10</sup> also reported that slow polymerization results in better marginal adaptation than polymerization with continuous high light intensity. It has been pointed out that composites cured at low light intensity have inferior compressive strength, flexural strength, microhardness, resistance to wear and resistance to fracture. This is caused by low degree of polymerization<sup>11</sup>.

Rueggerberg(1994) has reported that light intensity below 233mW/cm<sup>2</sup> should not be used because it produces significantly poorer cure at a depth of 2mm than higher light intensity<sup>27</sup>. He proposed that a minimum intensity of 400mW/cm<sup>2</sup> be recommended for routine polymerization of composite resin<sup>27</sup>. This is in agreement with our results. The composites cured with the light intensity below 200mW/cm<sup>2</sup> were produced less heat of polymerization and the corresponding degrees of polymerization were lower than for the other groups. In this study, polymerization occurred more quickly with the high intensity light. The time to reach peak heat flow was inversely proportional to light intensity. Because the speed of polymerization may be the most important factor contributing to the magnitude of the internal stresses generated in the composite restoration<sup>28</sup>, any retardation of the reaction will help to reduce stress development<sup>7</sup>. The relationship between the rate of polymerization and light intensity can be described as

$$R_p = k_p / k_t^{1/2} (I_0[A]b)^{1/2} [M]$$

Where  $\eta$  is the initiator efficiency,  $\epsilon$  is the absorption coefficient of the initiator,  $I_0$  is the incident light intensity in light quanta per area second,  $[A]$  is the molar concentration of initiator,  $[M]$  is the monomer concentration,  $b$  is the thickness of the reacting sample,  $k_p$  is the propagation kinetic constant, and  $k_t$  is

the termination kinetic constant (Oadian, 1991). This relationship shows that the rate of polymerization is proportional to the square root of light intensity. In our study, low intensity curing groups (group 1 and 2) showed reduced rate of polymerization. But in two step curing groups (group 5 and 6) the time to reach maximum heat output during the initial curing for 10s was not significantly different than that of low intensity curing groups cured for 40s. Another means to reduce the speed of polymerization is chemical modification of the resin system, for example, by reducing the concentration of photosensitizer or adding a retardant. But this less reactive resin would be more susceptible to decreased mechanical properties<sup>26)</sup>.

This study showed that the total heat of cure in the two step curing groups was slightly lower than that of the low intensity curing groups. The reason is not certain but one possibility is as follows: The DSC experiments may have been carried out with too small of a sample and the initial exposure exhausted most of monomer accessible for further polymerization. When samples were irradiated second time, the resins were considerably less reactive due to monomer exhaustion. A second possibility is that, when the composite resins are irradiated the first time, the polymerization reaction continues for a long time after the light is shut off. However, the speed of polymerization reaction is greatly reduced after the gel point during the initial polymerization. Reduced rate of polymerization resulted in less heat of cure at the second exposure.

## V. CONCLUSIONS

To evaluate the effects of light intensity variation on the polymerization of composite resins, this study was performed. The heat flow of the composite resins during polymerization reaction was recorded by a differential scanning calorimeter (DSC). In this experiment, the heat of cure of the test materials was measured and the time to reach maximum heat output was measured. And the degree of polymerization was analyzed with a Fourier Transform Infrared Spectrometer (FTIR). The results are summarized as follows.

1. As the light intensity increased during curing composite resins, the heat of cure also increased. But there was not a statistically significant difference among curing groups ( $p > 0.05$ ).
2. The time to reach maximum heat output decreased with increasing curing light intensity.
3. As the curing light intensity increased, the degree of polymerization also increased. The two step curing approach does not affect the degree of polymerization compared with continuous high intensity curing approach.
4. The light curing intensity above 400mW/cm<sup>2</sup> is recommended in the aspect of degree of polymerization.

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