

The Effects of Temperature and Light Intensity on the Polymerization Shrinkage of Light-cured Composite Filling Materials

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Abstract

The aim of this study was to investigate the effect of light intensity on polymerization shrinkage of resin composites. The five materials tested were Lite-Fil (A) (LFA), Lite-Fil (P) (LFP), Z100, Palfique, (PAL) and Tetric (TRC). Two types of measurements were made to determine the effects of environmental temperature and light intensity. Results were analyzed by two-way analysis of variance (ANOVA) and Newman-Keuls procedure at ($P < 0.05$). The changes in temperature and/or light intensity strongly affected composite shrinkage. The amount of shrinkage for LFP at 100% light intensity in room temperature (23°C) was 1.26% and increased up to 2.29% at 60°C . By raising the temperature to 37°C and dropping the light intensity to 42% ($459\text{mW}/\text{cm}^2$), the amount of polymerization shrinkage was similar to that with full light intensity, and statistically there were no significant differences between 37°C , 45°C , and 60°C . Lower temperatures and lower light intensities produce lower values of shrinkage. However, if the light intensity is below 28%, higher temperatures (60°C) must be applied to obtain a comparable amount of shrinkage.

Clinical Relevance: Pre-heating composite resin materials at body temperature (37°C) is likely to improve physical properties and compensate any drop of light intensity.

Keywords: Light-cured, composite, polymerization shrinkage, light intensity, temperature

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Introduction

Existing types of photo-curable resins and composites, especially those based on acrylates and methacrylates, exhibit polymerization shrinkage during the setting process.¹ The polymerization shrinkage strain of resin-composites, particularly contraction in a direction normal to the interface of the material with dental tissues, is of critical importance.² Polymerization stresses in a bonded structure may cause adhesive or cohesive failure and interfacial gap formation or, if adhesion is maintained, deformation of residual tooth structure.³

Once initiated, the initial polymerization of visible light-curing (VLC) resin-composites proceeds rapidly shrinkage reaction. For most materials, however, the normalized overall decrease in free-volume (V), i.e., shrinkage, is approximately represented by the Kohlraush-Williams-Watts [KWW] stretched-exponential relaxation function shown here.⁴

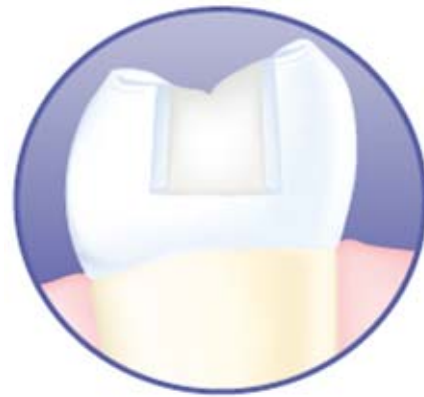
$$\Delta V / V_o = 1 - \exp(-(t/\tau)^\beta)$$

Legend:

- Where $0 < \beta \leq 1$, typical values for β being 0.3-0.6.
- ΔV is the change in molar volume and V_o the molar volume of the monomer.
- t is the shrinkage at time, the over all time-constant, the time for the shrinkage to attain a fraction $(1-e^{-1})$ of its final time.
- These time constants (τ) are obtained for a thin specimen geometry ($L_o < 1.5$ mm) in which light irradiation is optimal.⁵

Visible light, of the proper wavelength range and sufficient intensity, is essential for adequate polymerization of light-activated composite resins.⁶ The effect of exposure durations and distance of curing tip on light intensity was reported.^{7,8,9} It was concluded duration and intensity of visible light exposure are both important factors in composite resin polymerization. The polymerization contraction strain is time-dependent, and a methodology for measurement and analysis of the kinetics of this phenomenon has been described.⁷ This study was based on a deflection disc system which was first described by Wilson.¹⁰ The method has been further refined since 1991 to permit control of the ambient temperature (between 0-60°C) and of the light intensity.¹¹

The effects of light intensity, temperature, and composition on the polymerization behavior of Bis-GMA/TEGDM copolymerization has been reported.¹ They concluded the maximum rate of polymerization was significantly affected by the intensity of the light, and the temperature of the polymerization affected the conversion at which the maximum rate occurred. However, polymerization shrinkage could contribute to improving mechanical properties such as stiffness or hardness of composites resin. Unfortunately, stiffness and shrinkage could cause several clinical problems, such as gap formation at the restoration-cavity wall interface and/or build up stress, which could cause caspal deflection.¹²



Although polymerization shrinkage of composite resin has been extensively investigated, few studies have been conducted on the effect of temperature and light intensity on polymerization. Therefore, the purpose of this study was to evaluate the effects of temperatures on the polymerization shrinkage of five light-cured composite materials cured at full light intensity, and one representative material was selected to evaluate the effect of temperature and light intensity on the polymerization shrinkage.

Methods and Materials

Test Fixture and Specimen Preparation

Five types of VLC composite restorative materials listed in Table 1 were included in this study.

The modified apparatus (Figure 1) consisted of an aluminum stand incorporating a horizontal Table (A) and capable of clamping a LVDT (linear voltage displacement transducer) (B) (Type GT200, RDP Electronics Ltd., Wolverhampton, UK) in axial alignment with a disc shaped

Table 1. VLC Composite materials investigated.

Code	Material	Manufacturer	Batch No.
LFA	Lite-fil (A)	Shofu – Japan	Pn 1200
LFP	Lite-fil (P)	Shofu – Japan	Pn 1200
Z100	Z 100	3M Dental Products, St. Paul – USA	P920316
PAL	Palfique	Tokuyama Soda Co. Ltd.	28019Y
	Liteposterior	Tokyo-Japan	
TRC	Tetric	Vivadent-Schann-Liechtenstein	440153

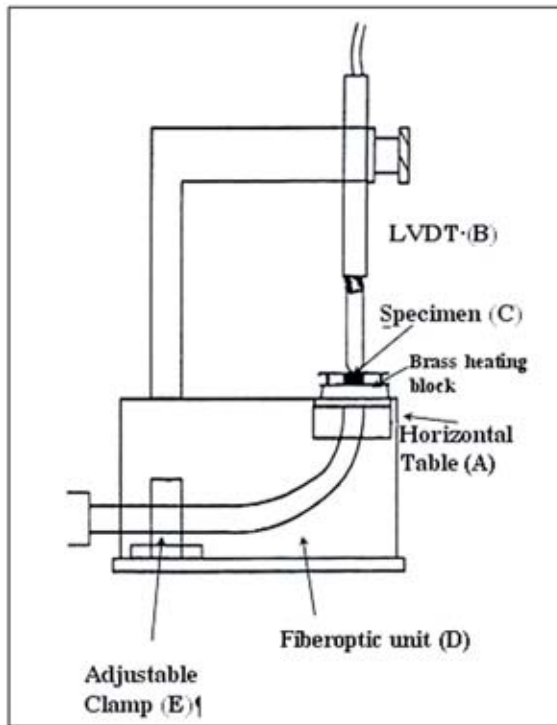


Figure 1. Shrinkage apparatus.

specimen (C). A clinical fiberoptic unit (D) was positioned below the table by an adjustable clamp (E). A “table insert” approximately 40 mm in diameter was positioned in access in the horizontal table.

The insert (Figure 2), machined from brass, acted as a measuring surface (F), which was electrically heated. The temperature was monitored via a calibrated thermocouple (G) inserted in a hole

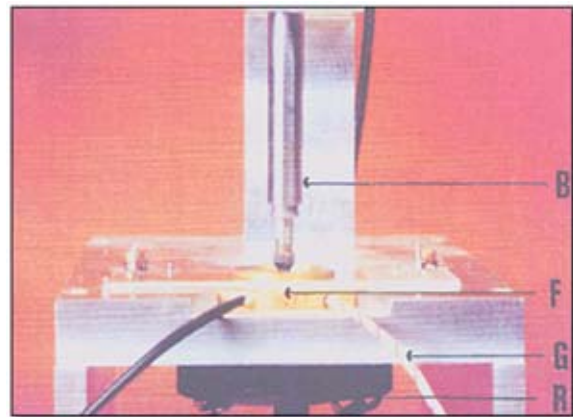


Figure 2. Table insert.

drilled into the rim. The heater consisted of two high power electrical resistors (R) fitted to the underside and embedded in an epoxy resin. When connected to an electrical supply, the table insert was heated due to the power dissipated in the resistors. The insert was constructed to loosely fit into the stand, so it was thermally decoupled from the stand. The insert was also pierced by a glass rod (diameter 7 mm), which acted as a co-axial light guide, conducting the radiation from the clinical curing light guide below.

Test Procedure

Before the start of the experiment the LVDT was calibrated by using the traceable calibration test apparatus constructed in the Unit of Biomaterials Science in Manchester University, UK. This was used to derive a calibration coefficient for the setting of the transducer amplification system.

A known volume of composite resin, approximately 12 mm³ was placed into the center of a square section (1.5 x 1.5 mm) brass ring with an internal diameter 16 mm, which was bonded to the surface of a glass microscope slide. The resin mass was covered by a microscope cover slip 25x25x1 mm and pressed down until the underside of the cover slip was in even contact with the brass ring. In this manner the resin was formed into a disc shape, about 8 mm in diameter and about 1.5 mm thick. This assembly was then positioned onto the surface of the shrinkage apparatus so the resin disc was centrally located over the glass light guide.

The LVDT was positioned in contact with the center of the cover slip, and the light unit Luxor ICI plc (Macclesfield, Cheshire, UK) was activated for 60 seconds to cure the composite from below. The deflection of the cover slip during shrinkage was monitored over time by the LVDT, which had sensitivity better than 0.1 µm. Electrical excitation for the displacement transducer was derived from an E30713 Transducer Indicator and Signal Conditioning Unit (RDP Electronics Ltd., Wolverhampton, UK), and the output of this unit after amplification was connected to an Analogue to Digital Converter [A-D], (Pico Technology Ltd., Hardwick, Cambridge, UK) for data storage via microcomputer resident software (Pico Technology Ltd.).

Measurement Procedures

Two types of measurements were taken to determine, repeatedly, the effects of environmental temperature and applied light intensity.

The first test was for polymerization shrinkage at four different temperatures for each material (Table 1):

- 23°C (room temperature)
- 37°C (human body temperature)
- 45°C (hot drinks)
- 60°C (maximum temperature)

First, the measurement was taken at room temperature (23°C) where no heat was used. For the other following temperatures 37°C, 45°C, and 60°C the plate was heated to the desired temperature using the brass heating block

(Figure 2F). The insert was heated electrically (Figure 2R) and monitored via a thermocouple (Figure 2G). At this stage, the specimen was placed on the brass heating block and heated for two minutes prior to the measurement being taken with the specimen covered with a black sheet to protect it from light. Then the covered sheet was removed and the light cure was initiated through the fiberoptic unit (Figure 1D). The polymerization shrinkage measurement was monitored spontaneously with the heat.

Five different groups, each one comprising three specimens of the same material, were measured at all four temperature levels making the number of samples 60.

The second type of measurement was polymerization shrinkage at different light intensities. Measurements were carried out on one representative material (LFP VLC composite) at four different temperature levels at four different light intensities:

- 1080 mW/cm² {maximum light intensity} (100%)
- 459 mW/cm² (42% of the maximum)
- 309 mW/cm² (28.6% of the maximum)
- 219 mW/cm² (20.8% of the maximum)

Three measurements were made at each light intensity and at each level of temperature, i.e., 23°C, 37°C, 45°C, and 60°C. The total number of samples was 48. The light intensity was measured with a calibrated radiometer of a type currently being recommended by the International Organization for Standardization (ISO) committee on powered polymerization activations (International Light Radiometer, Type IL 1350, Newport, Mass, USA).

Results

Shrinkage (%) v. Temperature

The mean values of shrinkage at maximum time (three minutes) at each temperature are tabulated in Table 2 and illustrated graphically in Figure 3.

These indicate when temperature was increased, the shrinkage also increased. This was confirmed by statistical analysis. One-way analysis of variance (ANOVA) of maximum

Table 2. The mean (X) and standard deviation (SD) values for shrinkage (%) at different temperatures.

Material Code		Shrinkage at 180 s (%)				F-Ratio	F-Prob.
		23°C	37°C	45°C	60°C		
LFA	X SD	1.40 0.05	2.00 0.05	2.18 0.07	2.35 0.15	68.2171	0.0001
LFP	X SD	1.26 0.11	1.77 0.12	1.88 0.09	2.29 0.14	37.0865	0.0001
PAL	X SD	1.48 0.09	1.70 0.05	1.94 0.06	2.18 0.07	52.9746	0.0001
TRC	X SD	1.64 0.15	2.19 0.14	2.36 0.09	2.86 0.29	22.4926	0.0003
Z100	X SD	2.10 0.14	2.42 0.18	2.83 0.02	3.38 0.0	26.5405	0.004

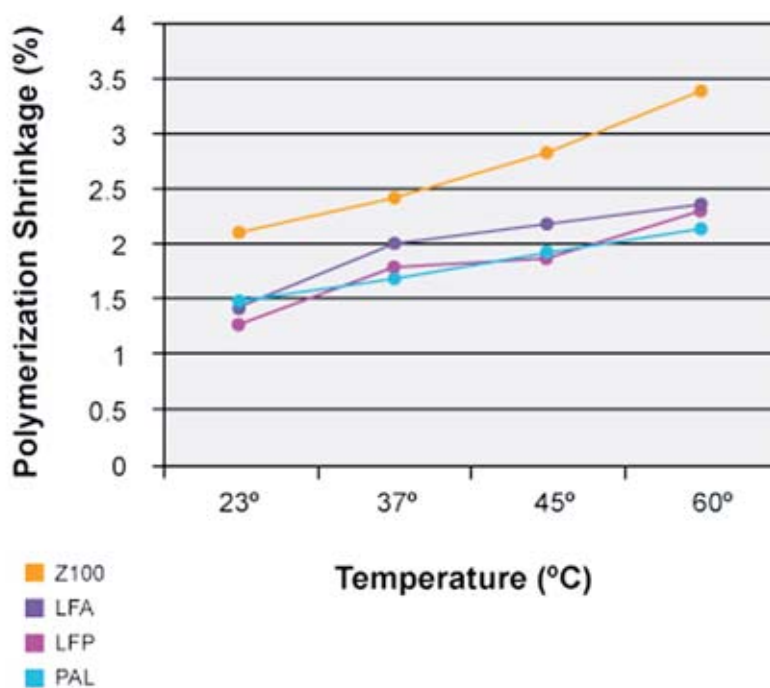


Figure 3. Polymerization strain rate as a function of temperature.

shrinkage showed significant differences ($p < 0.05$) with respect to both material and temperature. These may be summarized in a ranking manner as follows:

- at 23°C: LFP<LFA<PAL<TRC<Z100
- at 37°C: PAL<LFP<LFA<TRC<Z100
- at 45°C: LFP<PAL<LFA<TRC<Z100
- at 60°C: PAL<LFP<LFA<TRC<Z100

The polymerization shrinkage of five types of commercial VLC composite resin materials were measured (at three minutes) to range from 1.26 to 2.10% at 23°C (room temperature), 1.70 to 2.42% at 37°C (body temperature), and 2.18 to 3.38% at 60°C (Table 2).

Shrinkage (%) v. Light Intensity

The mean values of shrinkage strain (%) with different light intensities at three minutes for LFP are depicted in Table 3 and illustrated graphically in Figure 4.

The following features are apparent in the data:

- (a) The stronger the light intensity, the higher
- (b) The higher the temperature, the higher the shrinkage strain (%).

Discussion

The Technique

The technique followed in this investigation appears to be suitable in yielding reproducible results and has an advantage of being rapid in terms of overall experimental duration (three minutes). In addition the technique has the ability to determine the start of the ridged contraction for photo-initiated composite resin which was hardly possible by dilatometry methods.¹¹ The specimen geometry permits ideal light irradiation and, hence, optimal investigation conditions. The apparatus has been designed to cope with:

1. Different cure temperatures in the range 0°C–60°C
2. Different light intensities

These are both important clinically relevant variables. Consequently, this apparatus allowed investigation of five representative composites at four different temperatures; one of these materials

(LFP) was studied at four different light intensities at each of the four temperatures.

There are two main factors influencing polymerization. The curing temperature, by increasing the viscosity of the resin composite, has a significant influence on the acceleration of the reaction and reduces the curing time¹⁷ while improving the flowability of the resin. In addition the light intensity which has a significant effect on the maximum rate of polymerization.¹

Temperature Dependence

The equilibrium polymerization shrinkage of five types of commercial VLC composite resin materials has been measured to range from 1.26% to 2.10% at 23°C (room temperature), 1.70% to 2.42% at 37°C (body temperature), 1.88% to 2.83% at 45°C (hot drink), and 2.18% to 3.38% at 60°C (Table 2). These results confirm temperature affects the shrinkage. From Figure 3 and Table 2, it can be seen there is a gradual increase in the strain-rate as the temperature is increased. Z100 maintains a consistently higher polymerization shrinkage rate throughout the temperature range (23°C to 60°C) compared with the other materials (LFA, LFP, PAL, and TRC). These findings suggest increasing the temperature at a constant light intensity increases the polymerization shrinkage with higher temperatures producing higher shrinkage. Freedman and Krejci¹³ reported with elevated temperatures from 23°C to 54°C, the curing time can be reduced by 50% to 80% to achieve a similar conversion rate. However, Calheiros et al.¹⁴ reported higher energy densities may cause significant increase in stress values rather than an increase in the degree of conversion.



Curing Temperature

Table 3. Mean values of shrinkage (%) with different light intensities for Lite-fil (P) LFP composite measured at maximum time (three minutes)

Light Intensity (mW/cm ²)	Temperature (°C)			
	23	37	45	60
1080 (100%)	1.26	1.77	1.88	2.29
459 (42.5%)	0.93	1.61	1.92	2.24
309 (28.6%)	0.82	1.27	1.45	1.97
219 (20.8%)	0.55	0.72	0.75	1.27

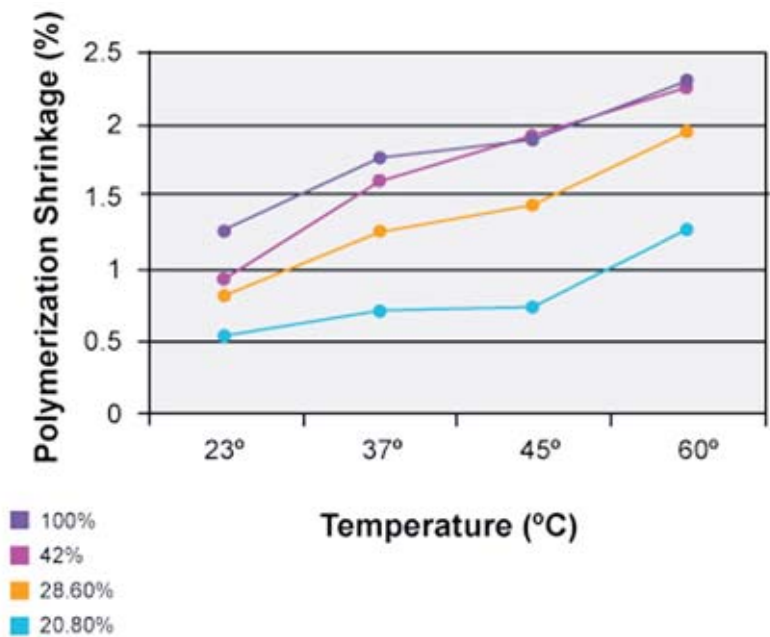


Figure 4. Polymerization shrinkage versus temperature at different light intensities. (Maximum light intensity 1080 mW/cm² = 100%).

Light Intensity Dependence

This study involved the variation in the intensity of the light emitted from an activating unit by reduction from its full light intensity of 1080 mW/cm² to values of 459, 309, and 219 mW/cm² which are equal to 42.5, 28.6, and 20.8% of the full intensity, respectively.

The temperature and light intensity affect the polymerization shrinkage. The question arises as to whether increasing the temperature of the composite specimen may compensate for a reduction of light intensity. This issue



Light Intensity

Table 4. Comparison of means (Newman-Keuls procedure) between shrinkage (%) at different temperatures and full light intensity (100%) and 42% of the maximum light intensities for Lit-fil P (LFP).

Mean(%)	Temperature	Intensity	Group	1	2	3	4	5	6	7	8
0.93	23°C	42 %	1								
1.26	23°C	100%	2	*							
1.61	37°C	42%	3	*	*						
1.77	37°C	100%	4	*	*						
1.92	45°C	42%	5	*	*						
1.88	45°C	100%	6	*	*						
2.24	60°C	42%	7	*	*	*					
2.29	60°C	100%	8	*	*	*					

(*) Denotes Significantly different at ($p < 0.05$).

was experimentally investigated by reducing the intensity of incident light on the surface of the specimen. The material studied was LFP. Interpretation of Tables 3 and 4 and Figure 4 indicate increasing the temperature at constant light intensity increases the polymerization shrinkage. Nevertheless, the effect of temperature is relatively small for a light intensity of 20.8 %, i.e., shrinkage increases from 0.55 to 1.27% as compared with at 100% light intensity when the shrinkage increased from an initial measured value at 23°C of 1.26 to a final value of 2.29% at 60°C. By raising the temperature to 37°C and dropping the light intensity up to 42.5% (459 mW/cm²), the polymerization shrinkage was similar to that with the full light intensity and statistically not significant between 37°C, 45°C, and 60°C. There is a significant difference between full light intensity and 42% light intensity at room temperature (23°C) (Table 4). Therefore, the results indicated by increasing the temperature (37°C up to 60°C), the loss of light intensity can be compensated approximately up to 70% for light intensity 309 mW/cm² (28.6%), whereas below 28.6% (309 mW/cm²) light intensity, higher temperatures must be applied to obtain a comparable amount of shrinkage.

In clinical situations composite in a deep cavity is inserted by the layering technique; the first layer is

usually away at a distance of 3 to 4 mm from the tip of the light curing unit.⁸ That would mean the light intensity decreased with increasing distance from the curing tip that would effect hardness and produce a softer composite resin.¹⁵ To overcome this problem, pre-heating of the composite resin between 37°C to 45°C may compensate for the loss of light intensity. However, Freedman and Krejci advocated warming of composite to either 37°C or slightly more than 54°C would improve the conversion rate of the material.

Conclusion

The following conclusions are drawn from the results and findings of the study:

1. Changes in temperature and/or light intensities significantly affect the shrinkage of light-cured composite ($p < 0.05$). Lower temperatures and lower light intensities produce less polymerization shrinkage.
2. By increasing the temperature, the loss of light intensity can be compensated up to approximately 70%; whereas, below 28% of normal intensity, higher temperatures must be applied to obtain a comparable amount of shrinkage.
3. Recommendation for pre-heating composite resin by 37°C to 45°C before insertion would be beneficial.

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