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ORIGINAL ARTICLE

Light polymerization during cavity filling: Effect of 'exposure reciprocity law' and the resulted shrinkage forces on restoration margins

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Abstract

Objective. To evaluate shrinkage development and marginal integrity of a micro hybrid restorative composite as a function of irradiance. ***Materials and methods.*** Linear displacement and shrinkage were measured with custom-made devices for irradiances of 100, 200, 400, 800 and 1600 mW/cm^2 at a constant radiant exposure of 16 J/cm^2. Marginal adaptation (MA) of composite restorations performed with a self-etch adhesive (Syntac Classic, Ivoclar Vivadent) and a micro hybrid composite (Tetric, Ivoclar Vivadent) was evaluated before and after mechanical loading with 300,000 cycles at 70 N. ***Results.*** The highest percentage of MA was attained by the group light cured with an irradiance of 100 mW/cm^2 for 160 s. No significant differences were observed between the rest of the groups. Shrinkage development was similar in all groups. ***Conclusions.*** For the material tested in this study, the reciprocal relationship between irradiance and time of exposure had no significant effect on restoration margins and shrinkage stress development within the range of 200–1600 mW/cm^2 with a constant radiant exposure of 16 J/cm^2.

Key Words: *curing protocol, irradiance, linear shrinkage, polymerization force, marginal integrity*

Introduction

Setting of light cured composite resins occurs by radical polymerization of methacrylate monomers and proceeds via adding monomers to the growing polymer chains [1]. The main purpose of the light source is to initiate polymerization by emitting a certain number of photons per second; these will excite the photo initiator molecules (camphorquinone/amine system) present within the composites mass to produce amine free radicals [2]. As long as monomers are available, propagation of polymerization continues when amine radicals add to the double bonds of methacrylate groups and generate new radicals that will further interact with monomers to form a cross-linked network. This reaction continues until no additional monomer is able to react or if the propagation reaction is stopped by termination, i.e. when two radical centers come into contact and interconnect together. In this context, the influence of different parameters of the light-curing source, i.e. *radiant exposure*, *power density* and *exposure time*, are of major importance and have been the subject of numerous investigations [1].

Some authors reported that *radiant exposure* (*power density* multiplied by *exposure time*) is the main determining factor of the degree of conversion and mechanical properties of the light cured material [3]. In this respect, a critical *radiant exposure* of ~ 16 J/cm^2 has been advocated as the minimum necessary to ensure adequate mechanical properties of the restorative composite [4–9]. Following this concept, for a given composite resin and *radiant exposure* the same total number of radicals should be generated under the conditions of both high *power density* and low *exposure time* or low *power density* and high *exposure time* [2]. This relationship between *power density* and *exposure time* is known as the *exposure reciprocity law*.

Previous reports have evaluated the influence of *irradiance* or *power density* variations on microleakage, microtensile bond strength, volumetric shrinkage, flexural strength, modulus of elasticity and degree of cure. Some of them observed no effect of *power density* variations if compensation with curing time exists [5,10,11], while others did not find this reciprocity [4,12–15].

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Table I. Description of the experimental groups.

Groups	Irradiance (mW/cm^2)	Exposure time (s)	Radiant exposure (J/cm^2)
100/160 s	100	160	16
200/80 s	200	80	16
400/40 s	400	40	16
800/20 s	800	20	16
1600/10 s	1600	10	16

In addition to the above-mentioned evaluations, little information is found in the literature regarding the effect of light curing with different *power densities* on marginal integrity. Marginal adaptation together with the assessment of polymerization shrinkage can add important information to the clinician in respect to the relationship between light intensity and curing time, and its impact on restorations' long-term performance. Marginal integrity is one important parameter contributing to the long-term success of any dental restoration. Monitoring of polymerization shrinkage can provide the information on the amount of contraction stresses that will be generated during polymerization. It is known from previous studies that contraction stresses have a direct impact on the adhesive interface; being responsible for cusp movements and marginal gaps [16].

Therefore, the main purpose of this investigation was to evaluate the marginal adaptation of class V restorations delivered by a restorative composite light cured with different *power densities* by keeping *radiant exposure* constant and, additionally, to assess if *power density* variations would have a significant effect on shrinkage development. The research hypotheses tested were that, for a given *radiant exposure* (16 J), (1) There would be no significant effect of *power density* variations on marginal adaptation, (2) There would be no effect of the different *power densities* on linear shrinkage and (3) There would be no effect of the different *power densities* on polymerization force.

Materials and methods

Assessment of marginal adaptation

Fifty caries-free human molars stored in 0.1% thymol solution were used for the experience within 1 month following extraction. After scaling and pumicing, the teeth were mounted on custom-made holders using a cold-polymerizing resin (Durafill VS, Heraeus Kulzer Gmbh, Hanau, Germany), with the cervical surface parallel to the holder and then randomly assigned to five experimental groups (Table I).

A round standardized cavity with half of the margins located on enamel and half on dentin was prepared in the cervical part of the tooth with cylindrical burs (Diatech Dental, Coltène Whaledent, Altstätten, Switzerland) under continuous water-cooling. The dimensions of the cavities were 4 mm in diameter and 2 mm in depth (c-factor of 3, i.e. ratio of bonded to non-bonded or free surface). A three-step adhesive system (Syntac Classic, batch number M21403, Ivoclar Vivadent, Schaan, Liechtenstein) and a hybrid restorative composite (Tetric A2, batch number M21414, Ivoclar Vivadent) were used for the restoration of all cavities. The adhesive system was light cured for 20 s with a halogen light source (Swiss Master Light, EMS, serial n° M1053, Nyon, Switzerland) at a constant relative irradiance of 800 mW/cm^2 that was periodically verified with a radiometer (Curing Radiometer Model 100, Serial No. 134089, Demetron Research Corp. Danbury, CT). Then the composite was inserted into the cavity in one layer and light-cured according to the different curing protocols with the same halogen light source.

An energy density of 16 mJ/cm^2 was used for all groups by inducing variations in the *time of exposure* and *irradiance*, that is, light curing for 10 s with an irradiance of 1600 mW/cm^2, for 20 s with 800 mW/cm^2, for 40 s with 400 mW/cm^2, for 80 s with 200 mW/cm^2 and for 160 s with 100 mW/cm^2.

After filling, the restored cavities were polished with decreasing grain size discs (Soflex Popon, 3M ESPE AG, Seefeld, Germany). Before performing the replicas the restorations were cleaned with nylon rotating brushes (White Polish brush, 3M ESPE) embedded firstly with tooth paste (Signal anti-caries, Thayngen, Switzerland) and then with water. Then impressions with a polyvinylsiloxane material (President light body, Coltène-Whaledent) were made of each restoration. The impressions were poured with epoxy resin to obtain individual models which would be further gold-coated for enabling observation in a scanning electron microscope (XL20, Philips, Eindhoven, The Netherlands) at 200× magnification and quantitative assessment of the marginal quality. Percentages of continuous margin (%CM) were reported for the entire margin length of each restoration before and after loading.

After storage in the dark for 1 week, the restored cavities were submitted to 300,000 cycles at 70 N of loading force applied to the center of the restoration in a loading chamber filled with room tempered tap water. To ensure that the antagonists would load in the center of the restoration, the cavity emplacement was marked with a pencil directly on the tooth's' surface before mounting the tooth on the support. Then the tooth was placed in the mounting device in such a way that the antagonist would have contact on the center of the cavity emplacement and be fixed to the support with the above-mentioned cold-polymerizing resin. The axial loading force was exerted at a 1.5 Hz frequency with antagonists made of stainless steel. The diameter of the metal cusps was of 4 mm. After loading, the teeth were cleaned with rotating brushes embedded with

Table II. Percentages of marginal adaptation before and after load. Results of linear shrinkage and polymerization force for the five different groups tested.

Groups (mW/cm^2/s)	% of CM before load (p = 0.995)	% of CM after load (p = 0.028)	Linear shrinkage (in µm) (p = 0.433)	Polymerization force (in Kg) (p = 0.014)
1600/10 s	66.3 (19)^a	51.1 (21)*a*,b	28.5 (1.3)^a	3.9 (0.3)^a
800/20 s	66.5 (15.8)^a	41.9 (8.6)^b	29.6 (1.3)^a	4.2 (0.3)^a
400/40 s	64.3 (13.8)^a	41.2 (15.2)^b	29.3 (0.9)^a	4 (0.2)^a
200/80 s	66.4 (16.8)^a	54.4 (27)*a*,b	29.2 (1.2)^a	3.9 (0.3)^a
100/160 s	66 (20.31)^a	65 (15.8)^a	29.2 (1.3)^a	3.5 (0.3)^b

Levels connected by different letters are significantly different and apply to each column.

tooth paste and then with water. Impressions with a polivinyloxane material (President light body, Coltène-Whaledent) were performed for each restoration following the same procedure as before loading.

Polymerization shrinkage

Measurements for *linear displacement* induced by polymerization shrinkage were carried out with a custom-made measuring device according to the protocol established by Stavridakis et al. [17]. A standardized amount of the same composite as used for cavity fillings was placed on the aluminum platelet of the device. Then the composite was flattened with a glass plate to a test height of 1.5 mm and a surface area of 50.2 mm^2 at both the top and bottom of the sample. This set-up reproduced a c-factor of ~ 3 (2.67). Groups '100, 200, 400, 800 and 1600' were polymerized for 160 s, 80 s, 40 s, 20 s and 10 s, respectively. A constant radiant exposure of 16 J/cm^2 was used for these tests. The vertical movement of the diaphragm caused by polymerization shrinkage of the composite was detected for 180 s by an infrared sensor with an accuracy of 100 nm and a sampling frequency of 5 Hz.

Measurements for *polymerization shrinkage force* were carried out with a custom-made measuring device [17]. The upper part consisted of a semi-rigid load cell to which was screwed a metal cylinder of 8 mm diameter. The cylinder was coated with the composite, which was compressed at a distance of 1.5 mm and a surface area of 46 mm^2 at both the top and bottom of the sample, on to a glass plate attached to the base of the device (c-factor ~ 3). The surface of the metal cylinder and of the glass plate were sandblasted with 50 µm aluminum oxide particles (Microetcher, Danville Engineering, Danville, CA) at 2 bars pressure and silanized (Monobond S, Ivoclar Vivadent). Groups '100, 200, 400, 800 and 1600' were polymerized for 160 s, 80 s, 40 s, 20 s and 10 s, respectively. Forces generated during polymerization shrinkage were detected for 180 s by means of the load cell at a sampling frequency of 5 Hz. The data were fed on-line by means of an A/D converter using custom-made software to a personal computer (Macintosh II fx, Apple computer, Cupertino, CA) and stored on its hard disc. Eight measurements for linear displacement and eight for shrinkage force were performed on each group and their mean values were plotted.

Statistical analysis

Analysis of percentages of continuous margins (% CM) of linear displacement and shrinkage force data was performed with one-way ANOVA and Duncan post-hoc test. The level of confidence was set to 95%.

Results

The mean %CM for each group, at the total margin length, before and after mechanical loading, is resumed in Table II. Results of marginal adaptation *before loading* were not significantly different (p = 0.995). After loading, significant differences (p = 0.028) were detected between the groups. The highest scores of marginal adaptation after loading were attained by groups light cured with irradiances of 100 mW/cm^2 (65 (15.8)), 200 mW/cm^2 (54.4(27)) and 1600 mW/cm^2 (51.1(21)); no significant differences could be detected between these three groups. Also, no significant differences in marginal adaptation were observed between the groups light cured with irradiances of 400 mW/cm^2 (41.2(15.2)), 800 mW/cm^2 (41.9(8.6)), 200 mW/cm^2 and 1600 mW/cm^2 (Table II). This indicates that, except for the group light cured with an irradiance of 100 mW/cm^2, %CM was similar in the rest of the groups.

Scores of linear displacement and of polymerization force produced by the contraction of the resin composite during polymerization and registered for 180 s are resumed in Table II. Linear displacement in microns due to polymerization shrinkage was similar in all groups (p = 0.433). In respect to polymerization force, significant lower forces were experienced by the resin composite cured with an irradiance of 100 mW/cm^2. For the other irradiances (200, 400, 800 and 1600 mW/cm^2), no significant differences in polymerization force were observed between the groups. Figures 1 and 2 show the development of

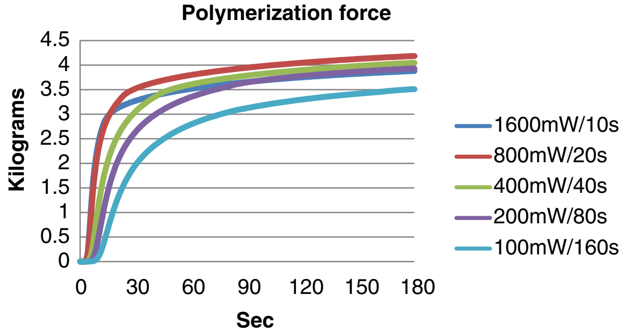

Figure 1. Contraction force developed during polymerization (Kg) registered for the five different groups since the start of light curing up to 3 min.

both properties. The curve profiles of each group showed that a slower speed of polymerization could be observed in the groups 100, 200 and 400 mW/cm^2. However, after 180 s following polymerization, no significant differences in linear shrinkage were observed between the different irradiances (Table II). Representative SEM images of enamel–resin composite and dentin–resin composite interfaces are shown in Figures 3,4,5. Minor differences in marginal morphology (SEM analysis at 200× magnification) were detected between the groups.

Discussion

The purpose of this study was to test the hypotheses that, by curing with different *irradiances*, stresses generated due to polymerization contraction and scores of marginal adaptation would be similar as a result of the 'reciprocity law' between *irradiance* and *curing time*. To this purpose only the parameters *irradiance* and *curing time* were modified, while the others, i.e. adhesive system, restorative composite, light curing device and *radiant exposure*, were kept constant. Quantitative margin analysis of composite restorations cured with different *irradiances*, identical *radiant exposure* and related to polymerization shrinkage has not been, according to the authors' knowledge, evaluated up to now [5,11,18]. The reason for choosing an irradiance of 16 J/cm^2 was based on the results of previous studies that considered this amount of energy as sufficient to ensure adequate mechanical properties of the restorative composite [4–9].

During polymerization of resin composites, intermolecular distances of 0.3–0.4 nm between dimethacrylate monomers, maintained by van der Waals attraction forces, are reduced by conversion of C=C bonds and formation of short-range covalent C-C bonds with lengths of ~ 0.15 nm [19]. In the present study, both linear shrinkage and polymerization force measurements were limited to 3 min, in order to shorten overall measurement duration. Another study using similar methodology [20] has suggested observation periods up to 24 h. Nevertheless, after performing a pilot study with observation times up to 20 min, we observed that after 3 min both properties, linear shrinkage and polymerization force, had reached over 90% of the value obtained after 20 min. While our results may not reflect the ultimate values of contraction forces due to polymerization shrinkage in absolute numbers, the use of this methodology to determine the differences between curing protocols is justified.

Before loading, no significant differences in percentages of continuous margins were observed between the groups, this was expected as specimens were not subjected to stress conditions. However, after loading, the highest results in terms of percentages of continuous margins were attained by the group light cured with the lowest power density (100 mW/cm^2) and the longest curing time (160 s). The lowest polymerization forces were also observed in this group, as shown in Table II. An increased exposure time with low light intensity has been found to improve depth of cure, because the formation of a rigid network of bonds among the polymeric chains is delayed [21]. It is possible that a better depth of cure improved the quality of the final restoration, which in the present study was traduced by an increased percentage of continuous margins. Our results are in line with the findings of Da Silva

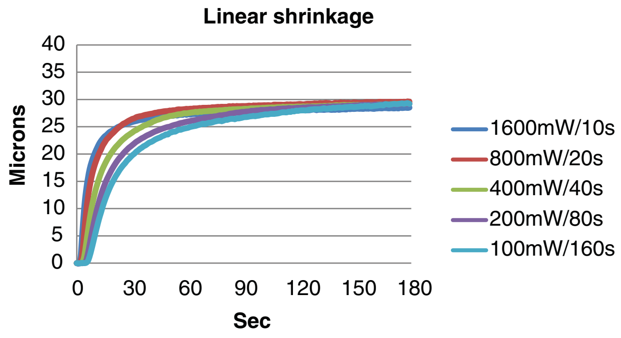

Figure 2. Displacement in microns registered for the five different groups since the start of light curing up to 3 min.

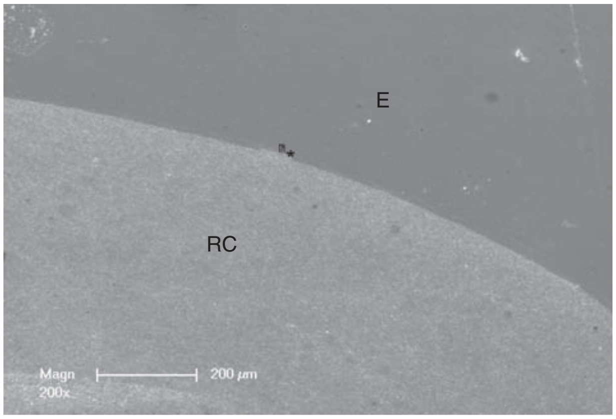

Figure 3. Representative SEM micrograph (200× magnification) of a restoration with gap-free margins. RC, Restorative composite; E, Enamel; *, adhesive interface.

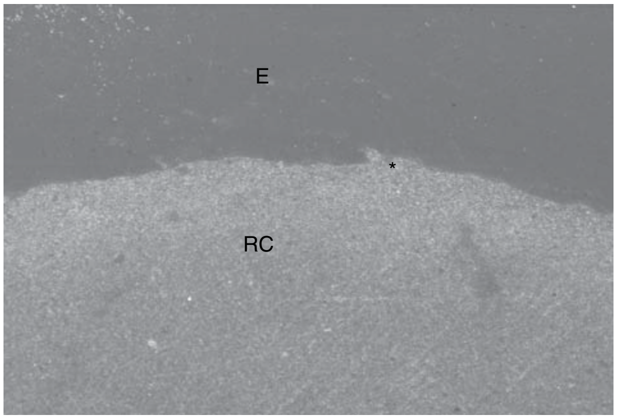

Figure 4. Representative SEM micrograph (200× magnification) of a restoration with marginal imperfections. No marginal gaps are visible, but an overhang (*) is present at the margins. RC, Restorative composite; E, Enamel; *, adhesive interface.

Segalin et al. [5]. These authors counted the number of gap widths generated after light curing with different light intensities and, for a constant radiant exposure, they observed no differences at the total mean gap width, suggesting a similar monomer-to-polymer conversion. Similarly, our results of marginal adaptation were not significantly different before loading. In terms of micro-hardness, however, they reported the highest values for the group light cured with a low irradiance. Similarly, our results showed the highest results of marginal adaptation when light curing with a low irradiance. Confirming the findings of previous studies [22–25], differences in velocity of polymerization, an extended pre-gel phase and more time available for viscous flow to relieve shrinkage stresses may have accounted for the highest results in marginal adaptation for the group cured with a power density of 100 mW/cm^2. However, translating this curing protocol to the clinical reality means that a 2 mm layer of composite should be light cured for almost 3 min. Therefore, due to practical reasons the current protocol, while delivering the best results in terms of marginal adaptation, may not be recommended.

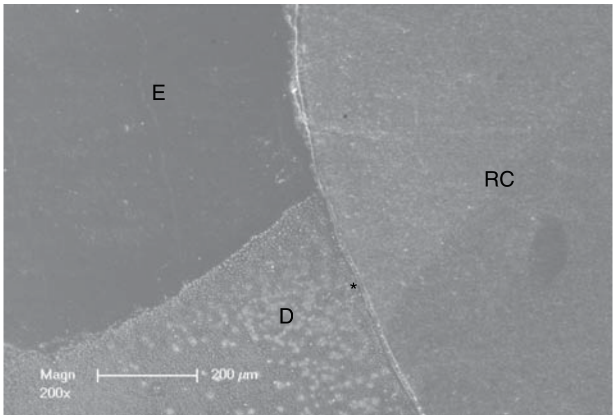

Figure 5. Representative SEM micrograph (200× magnification) of a restoration at the enamel–dentin junction. A marginal gap (*) can be observed. RC, Restorative composite; E, Enamel; D, Dentin; *, adhesive interface.

Before thermo-mechanical load, the results of marginal adaptation did not differ significantly, justifying why the 1st research hypothesis that *there would be no significant effect of power density variations on marginal adaptation* could be demonstrated. After loading, however, the research hypothesis was only partially demonstrated as the highest %CM were observed in the group with the lowest power density and longest curing time (160 s). For the rest of the groups light cured in more clinically relevant time frames, increasing *exposure time* in cases in which *irradiance* was decreased has resulted in similar scores of marginal adaptation. While the reciprocity between *irradiance* and *exposure time* has been proved for properties such as elastic modulus [26], conversion rate by using Transmission Fourier Transform Infrared analysis [13], Attenuated Total Reflection F-TIR analysis [21], Raman Spectroscopy [15,27] and Knoop hardness [14], the present study showed that the reciprocity law also applies for restoration margins immediately after curing. These findings, however, should not be generalized to all composite resins, as the photo initiator type and the materials' filler content can also influence the properties of resin composites even when light cured with similar *radiant exposure* [3]. Also, our results apply for 2 mm-depth samples; therefore in cases of deeper cavities an increased *radiant exposure* than the one of 16 J used in this study may be used for light curing. It is well known that sample thickness strongly influences the degree of conversion, which is related to a decreased light transmission due to an increased cavity depth. Only by increasing energy density can an increase in depth of cure be achieved [21].

In terms of linear shrinkage and polymerization force, the main differences between groups could be observed during the first 60 s (Figures 1 and 2). In agreement with current scientific literature [28], groups cured at lower irradiances experienced less linear shrinkage and contraction forces. An extended pre-gel phase and time available for viscous flow contributed to relieve shrinkage stresses. Davidson and de Gee [23] hypothesized that, in the first stages of polymerization, only C-C chain formation initiate, and cross-linking is not yet complete, this would allow polymer molecules to adopt new positions and orientations and, therefore, to compensate for polymerization contraction without damaging the internal structure of the material. However, the results after 180 s showed that once the gel point was attained, a similar pattern of shrinkage (Table II) was observed in all groups. This observation confirms previous findings of equal ultimate polymerization shrinkages independently of the low or high light intensity that is used for light curing [29]. The significantly lower scores of polymerization force developed in the group light cured for 160 s with an irradiance of 100 mW/cm^2 (Figure 1) explain why the best results of marginal adaptation were obtained. Our results are in good agreement with those of Ferracane and Mitchem [16], showing that reduced curing rates lead to less overall stress generation and improved sealing. Therefore, the 2nd research hypothesis that *there would be no effect of the different power densities on linear shrinkage* could be validated. Due to the lower shrinkage forces generated by the group light-cured with the lowest power density, the 3rd research hypothesis that *there would be no effect of different power densities on polymerization force* could not be completely demonstrated.

Overall, a sufficient radiant exposure is needed to attain a certain depth of cure. Under this condition, this study could show that adjustment of exposure times can compensate for differences in irradiance without negatively influencing restoration margins. Under this perspective, it could be useful if manufacturers can provide information on the amount of energy density necessary to ensure, for a given composite thickness, adequate polymerization. This would enable the clinician to easily calculate, provided that the amount of *irradiance* delivered by her/his light curing unit is known, the exposure time necessary to attain a safe degree of cure.

Conclusions

For a radiant exposure of 16 J/cm^2, the highest scores of marginal adaptation and the least polymerization stresses were generated when the resin composite was light cured by using a low *irradiance* (100 mW/cm^2) and an extended *curing time*. Clinically, this curing protocol may not be practical because it requires long curing times for each layer. When irradiances of 200, 400, 800 and 1600 mW/cm^2 were used for photo polymerization, similar results of shrinkage stresses and marginal adaptation were obtained.

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