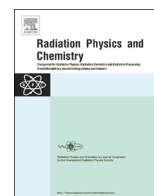




ELSEVIER

Contents lists available at ScienceDirect

## Radiation Physics and Chemistry

journal homepage: [www.elsevier.com/locate/radphyschem](http://www.elsevier.com/locate/radphyschem)

## DH and ESPI laser interferometry applied to the restoration shrinkage assessment

L.M.P. Campos <sup>a,\*</sup>, D.F. Parra <sup>a</sup>, M.R. Vasconcelos <sup>b,1</sup>, M. Vaz <sup>c,2</sup>, J. Monteiro <sup>c,2</sup><sup>a</sup> Institute of Nuclear and Energy Research, University of Sao Paulo (IPEN/USP), Av. Lineu Prestes, 2242 Cidade Universitária, São Paulo, SP 05508-000, Brazil<sup>b</sup> School of Dental Medicine, University of Porto (FMDUP), Rua Dr. Manuel Pereira da Silva, 4200-393 Porto, Portugal<sup>c</sup> Faculty of Engineering, University of Porto (FEUP), Rua Dr. Roberto Frias, s/n. 4200-465 Porto, Portugal

## HIGHLIGHTS

- Both of holographic techniques were able to measure the polymerization shrinkage.
- The entire tooth surface was deformed during the polymerization shrinkage.
- The group with greater percentage of filler showed the lowest value of deformation.
- The values of displacement ranged from 0.9 to 3.4  $\mu\text{m}$ .

## ARTICLE INFO

## Article history:

Received 21 December 2012

Accepted 2 May 2013

Available online 11 June 2013

## Keywords:

Digital holography

Dental composites

Shrinkage polymerization

## ABSTRACT

In dental restoration postoperative marginal leakage is commonly associated to polymerization shrinkage effects. In consequence the longevity and quality of restorative treatment depends on the shrinkage mechanisms of the composite filling during the polymerization. In this work the development of new techniques for evaluation of those effects under light-induced polymerization of dental nano composite fillings is reported. The composite resins activated by visible light, initiate the polymerization process by absorbing light in wavelengths at about 470 nm. The techniques employed in the contraction assessment were digital holography (DH) and Electronic Speckle Pattern Interferometry (ESPI) based on laser interferometry. A satisfactory resolution was achieved in the non-contact displacement field measurements on small objects concerning the experimental dental samples. According to a specific clinical protocol, natural teeth were used (human mandibular premolars). A class I cavity was drilled and restored with nano composite material, according to Black principles. The polymerization was monitored by DH and ESPI in real time during the cure reaction of the restoration. The total displacement reported for the material in relation of the tooth wall was 3.7  $\mu\text{m}$  (natural tooth). The technique showed the entire tooth surface (wall) deforming during polymerization shrinkage.

© 2013 Elsevier Ltd. All rights reserved.

## 1. Introduction

Polymerization shrinkage is the main limitation of resin composites for dentistry applications. Their polymerization is initiated by the photo-activation in the spectral band between 300 and 500 nm (Rueggeberg and Jordan, 1993). After its insertion in the dental cavity, during cure, the composite is subject to the opposing forces of polymerization shrinkage and adhesion to the dental structure (Carvalho, 1996). The problem caused by polymerization

shrinkage is critical, as the composite must remain tightly bound to the dental cavity while it grows rigid and reduces its dimensions. However, should the restoration material be allowed to detach from the walls of the dental cavity, the resulting rupture would cause a marginal microleakage, leading to further issues such as: secondary caries, postoperative sensibility and pulp alteration (Kleverlaan and Feilzer, 2005; Venhoven et al., 1993).

The loss of marginal integrity is one of the main causes for failure during resin composite restoration. An improper procedure of adhesion and residual stresses around the restoration interface are among the main causes for an eventual breakage of the bond between restoration substrate and dental tissue. As seen in literature, interface displacement can follow mainly from stress generated by the resin composite polymerization. (Davidson and Davidson-Kaban, 2000; Ferracane, 2005; Calheiros et al., 2004; Braga and Ferracane, 2004; Davidson and Davidson-Kaban, 2000).

\* Corresponding author. Tel.: +5511 3133 9313.

E-mail addresses: [luizamello@usp.br](mailto:luizamello@usp.br), [luizamellocampos@gmail.com](mailto:luizamellocampos@gmail.com) (L.M.P. Campos), [dfparra@ipen.br](mailto:dfparra@ipen.br) (D.F. Parra), [mvasconcelos@fmd.up.pt](mailto:mvasconcelos@fmd.up.pt) (M.R. Vasconcelos), [gmavaz@fe.up.pt](mailto:gmavaz@fe.up.pt) (M. Vaz), [jmont@fe.up.pt](mailto:jmont@fe.up.pt) (J. Monteiro).<sup>1</sup> Tel.: +351 220 901 100.<sup>2</sup> Tel.: +351 22 508 14 00.

Different methods are available to measure polymerization shrinkage: mercury dilatometer (De Gee et al., 1981; Reed et al., 1996), extensometer (Puckett and Smith, 1992; Rosin et al., 2002; Vandewalle et al., 2004), linear displacement (De Gee et al., 1993; Park et al., 2002), free linear contraction (Pereira et al., 2009), bonded disk (Watts and Cash, 1991; Watts and Al Hind, 1999). All these measurement methods are based on the same physical principle.

Out of the methods used to register alterations caused during the shrinkage process of orthodontic composites, those based on optical measured presented several advantages. As such, ESPI (Electronic Speckle Pattern Interferometry) and DH (Digital Holography) were chosen. Both are optical methods that fit well with this study, as they allow for measurement of dislocation without contact and with great precision. Dislocation (deformation) measurement is made based on the alteration of the optical path that light follows. The optical path suffers such alteration due to mechanical roughness in the surface.

This study's goal was to monitor the polymerization shrinkage of restoration composites in real-time through usage of high-resolution optical interferometry measuring devices.

## 2. Materials and methods

### 2.1. Materials

The materials used to the experimental nano composites were: **Bis-GMA** (Bisphenol A Bis(2-hydroxy-3-methacryloxypropyl) Ether). Lot: 688-51, Esstech, Essington, Pennsylvania, USA; **TEGDMA**: (Triethyleneglycol Dimethacrylate). Lot: PA02700018, Esstech, Essington, Pennsylvania, USA. **Camphorquinone**, 97%. Lot: S12442-127, Sigma-Aldrich Chemie, GmbH, Steinheim, Germany. **DMAEMA**: (2-(Dimethylamino)ethyl methacrylate), 98%. Lot: 21608009, Sigma-Aldrich Chemie, GmbH, Steinheim, Germany; **Cloisite® 10A**: MMT natural Cloisite® 10A. Lot: 09C02AAX015, Southern Clay Products, Inc. 1212 Church Street Gonzalez, Texas 78629, USA;

The experimental composites groups were prepared with Cloisite® 10A in three distinct formulations, varying according to the type and the concentration (in percentage) of inorganic filler, they were: Group 1–50% Cloisite® 10A; Group 2–60% Cloisite® 10A; Group 3–65% Cloisite® 10A.

The materials were individually weighed on an electronic scale digital precision, Shimadzu/model AY220. The goal was for each group of experimental composite had 1 g in its final mass. The manipulation of the experimental composites began by mixing the polymer matrix. The BisGMA was incorporated into TEGDMA in equal proportions in all groups, as well the camphorquinone and the tertiary amine DMAEMA. After manipulation of the polymer matrix, was added the inorganic filler in small amounts until the entire cargo incorporation in the matrix. For a correct homogenization of the mixture, the experimental composite was manipulated for 5 min. The optimum thickness of samples was defined to be 0.50 mm. A Digital Caliper, Digital 6" model (0–150 mm) was used to check the samples thickness.

In this study, human teeth (premolars) were prepared with a specific clinical protocol. A class I cavity in the occlusion side was previously drilled and restored with experimental nano composite material, according to the time-Honored principles of G.V. Black. The cavities were drilled with a 2.5 mm depth using a calibrated drill with a stop.

All the preparations (natural teeth recovered with experimental nanocomposite material) were placed in a DH or ESPI set up to obtain the experimental recordings. A blue LED curing lamp, brand Kondor-teck, model Aigh-7A (420–480 nm) is used to induce composite polymerization, and the real time deformation during resin cure was

recorded. Experimental data was post processed for the deformation assessment. Indirect measurements of the resin behavior were obtained from the teeth deformation. Direct measurements on the resin surface are very difficult due to the speckle decorrelation which occurs because the texture surface changes during polymerization.

### 2.2. Apparatus

The ESPI is an interferometry technique that uses coherent light illumination to obtain with no direct contact the displacement distribution associated to the superficial deformation of an object. DH is also an interferometry technique, similar to the ESPI, however it uses the classical off axis set up with no optical system for image formation. On DH the hologram is recorded directly on the target of a video recording system and later is numerically reconstructed. Both techniques can be used with image processing methods to extract the displacements of each point in the area of measurement. ESPI registers the superficial deformation and the HD reconstructs numerically the deformation (Monteiro et al., 2011).

All the dental preparations were placed in a DH or ESPI set up to obtain the holographic recordings. A blue led lamp (420–480 nm) was used to induce composite polymerization, and the real time deformation during resin cure was recorded. The experimental system of DH is represented in Fig. 1 and the experimental system of ESPI can be seen in Fig. 2. Experimental data was post processed for the deformation assessment. Indirect measurements of the resin behavior were obtained from the natural teeth deformation. The laser used for this study was a Coherent, model Verdi, with 2 W of maximum power at a wavelength of 532 nm.

For each measurement the blue lamp (420–480 nm) was used to induce polymerization in the nanocomposite previously applied

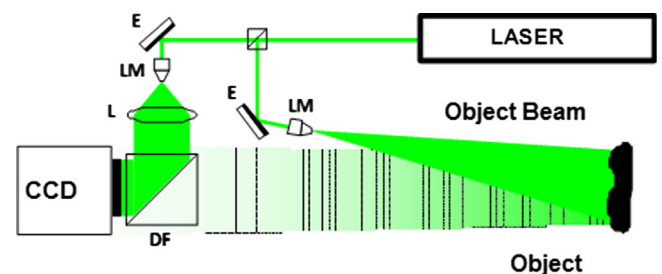


Fig. 1. Experimental system used to perform measurements with digital holography. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

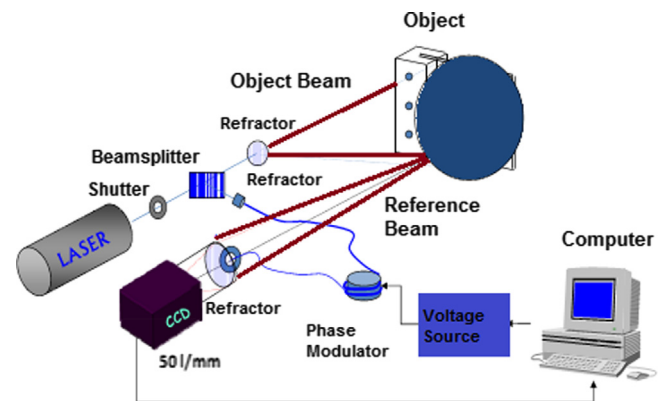


Fig. 2. Experimental set up for measurements with ESPI (Electronic Speckle Pattern Interferometry). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

on cavities prepared in the natural teeth. Each analysis had the duration of 1 min (30 s during the cure and another 30 s in the post cure period).

2.3. Displacement measurements

The deformation induced by the polymerization was reported in real time on a video monitor where the fringe pattern evolution was seen during the cure reaction of the experimental nanocomposite formulation. The measurement was done indirectly on the tooth surface and not on the resin under shrinkage. The laser

insertion was done on the natural tooth wall owing to the effect of surface roughness of the resin if directly irradiated.

3. Results and discussion

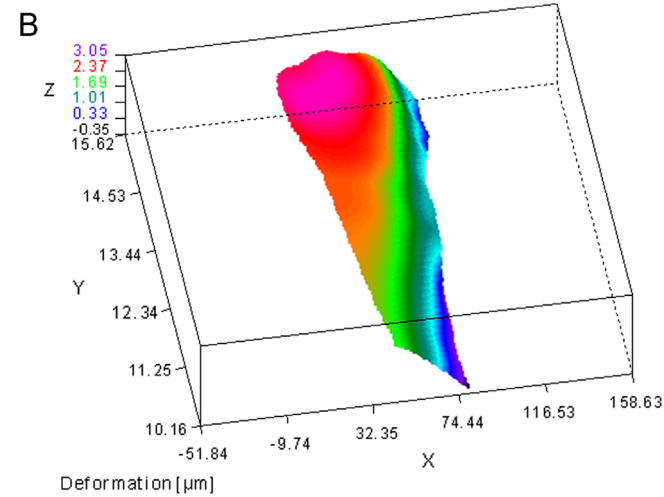
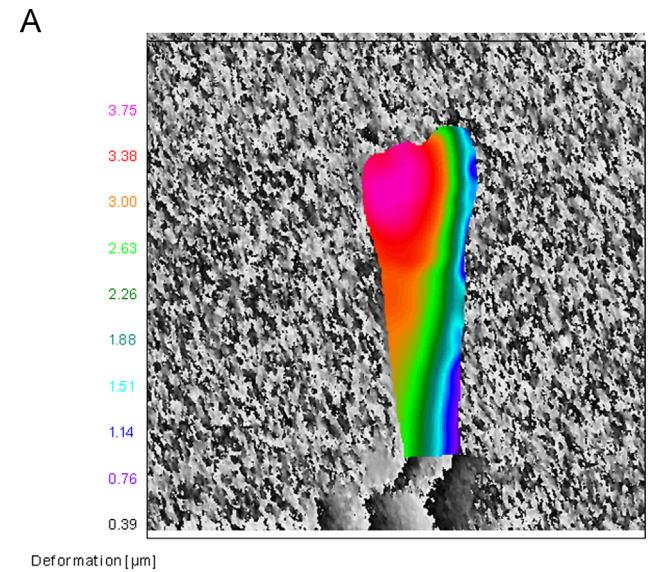
3.1. Holography results

The nanocomposite applied on natural tooth when exposed to blue light, suffers shrinkage due to cure reactions. The result of the displacement suffered by each group during the polymerization is described in Table 1:

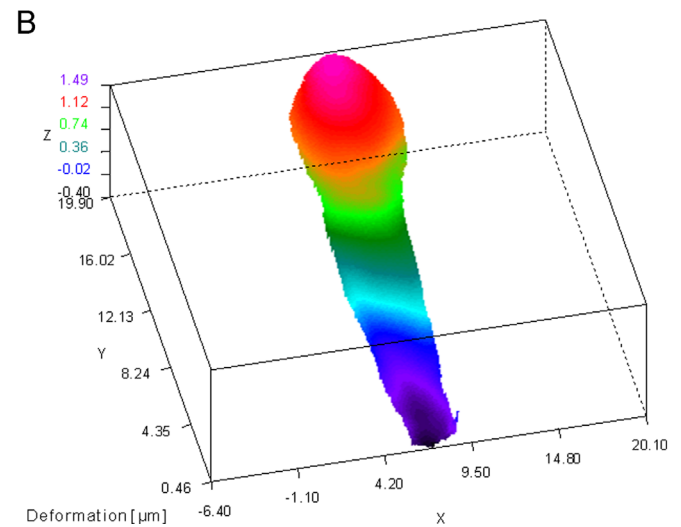
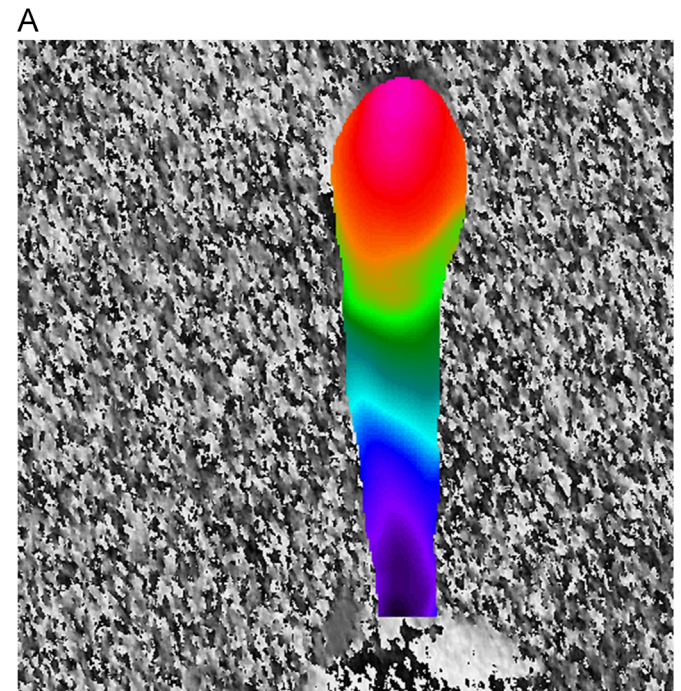
**Table 1**  
Result of the displacement suffered by each group during polymerization.

Group (% filler)	Displacement (μm)
1 (50%)	3.7
2 (60%)	1.4
3 (65%)	0.9

The entire tooth wall moves in form of frames as shown in Fig. 3 (Group 1), Fig. 4 (Group 2) and Fig. 5 (Group 3). Zones of high intensity of shrinkage are represented by red color while zones of lower intensity are blue.

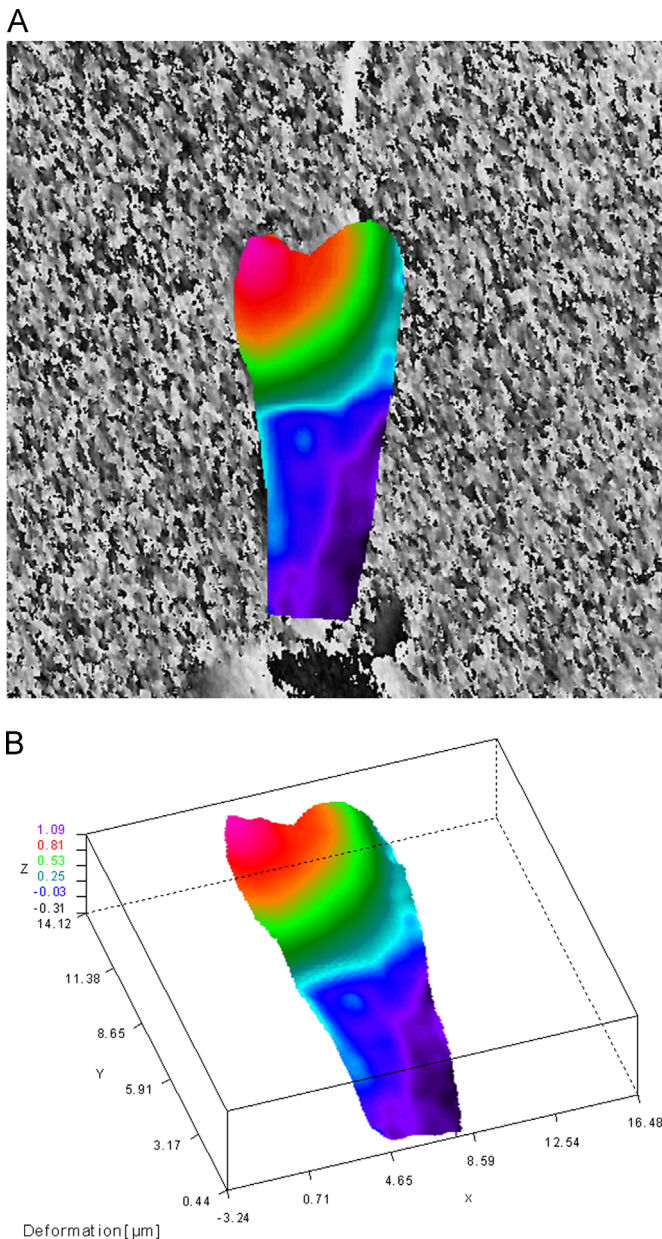


**Fig. 3.** (A) Range of colors indicating areas of higher and lower displacement. (B) Image captured at the end of the analysis with a scale indicating the final values of displacement of Group 1. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



**Fig. 4.** (A) Range of colors indicating areas of higher and lower displacement. (B) Image captured at the end of the analysis with a scale indicating the final values of displacement of Group 2. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)





**Fig. 5.** (A) Range of colors indicating areas of higher and lower displacement. (B) Image captured at the end of the analysis with a scale indicating the final values of displacement of Group 3. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

The experimental nanocomposites with higher amounts of filler, showed the lowest displacement value. The results demonstrated great potential of this technique in indirect measurement of polymerization shrinkage of dental composites. The numerical/experimental technique should be compared with other techniques, such as conventional dilatometry.

The process of polymerization shrinkage of dental composites was evaluated by authors through ESPI-HD-CI techniques. A comparative study using different composite materials, as well as a numerical simulation with finite element, was done. The indirect measurements of the composites behavior were obtained from the deformation of the tooth external surface. According to the authors, the technique demonstrated a great accuracy in obtaining results (Monteiro et al., 2011). ESPI was also used to evaluate the impact of chewing on the different restoration materials in natural teeth specimens (Lang et al., 2004) as well

as a means of determining the biomechanical behavior of a partially edentulous human mandible as a function of the alveolar process density (Campos et al., 2009).

#### 4. Conclusions

The results obtained proved the suitability of both techniques for resin shrinkage assessment through an indirect measurement. The technique showed that the entire tooth surface (wall) deforms in the shrinkage during the polymerization. The group with the highest amount of filler showed the lowest value of the polymerization shrinkage.

#### Acknowledgments

The authors thank to CAPES, CNPQ, CNEN, FAPESP (process number 2012/0236-1), Faculdade de Medicina Dentária da Universidade do Porto and Faculdade de Engenharia da Universidade do Porto, for the support during the experimental work.

#### References

- Braga, R.R., Ferracane, J.L., 2004. Alternatives in polymerization contraction stress management. *Crit. Rev. Oral. Biol. Med.* 15, 176–184.
- Calheiros, F.C., Sadek, F.T., Braga, R.R., Cardoso, P.E., 2004. Polymerization contraction stress of low-shrinkage composites and its correlation with micro leakage in class V restorations. *J. Dent.* 32, 407–412.
- Campos, J.C.R., Correia, A., Vaz, M.A.P., Branco, F.M.B., 2009. Holographic stress analysis in a distal extension removable partial denture. *Eur. J. Prosthodont Restor. Dent.* 17, 111–115.
- Carvalho, R.M., 1996. A review of polymerization contraction: the influence of stress development versus stress relief. *Oper. Dent.* 21, 17–24.
- Davidson, C.L., Davidson-Kaban, S.S., 2000. Handling of mechanical stresses in composite restorations. *Dent. Update* 25, 274–279.
- De Gee, A.J., Davidson, C.L., Smith, A., 1981. A modified dilatometer for continuous recording of volumetric polymerization shrinkage of composite restorative materials. *J. Dent.* 9, 36–42.
- De Gee, A.F., Feilzer, A.J., Davidson, C.L., 1993. True linear polymerization shrinkage of unfilled resins and composites determined with a linometer. *Dent. Mater.* 9, 11–14.
- Ferracane, J.L., 2005. Developing a more complete understanding of stress produced in dental composites during polymerization. *Dent. Mater.* 21, 36–42.
- Kleverlaan, C.J., Feilzer, A.J., 2005. Polymerization shrinkage and contraction stress of dental resin composites. *Dent. Mater.* 21, 1150–1157.
- Lang, H., Rampado, M., Mullejans, R., Raab, W.H.M., 2004. Determination of the dynamics of restored teeth by 3D electronic speckle pattern interferometry. *Lasers Surg. Med.* 34, 300–309.
- Monteiro, J., Lopes, H.M., Vaz, M., Campos, L.M.P., Vasconcelos, M., Campos, J.C., 2011. Estudo do comportamento mecânico dos compósitos de restauro dentário utilizando técnicas ópticas. *Anais do 4º Congresso Nacional de Biomecânica-Coimbra, Portugal.* 275–277.
- Park, S.H., Krejci, I., Lutz, F., 2002. Microhardness of resin composite polymerized by Plasma ARC or conventional visible light curing. *Oper. Dent.* 27, 30–37.
- Pereira, R.A., Araújo, P.A., Castañeda-Espinosa, J.C., Mondelli, R.F.L., 2009. Comparative analysis of the shrinkage stress of composite resins. *J. Appl. Oral Sci.* 16, 30–34.
- Puckett, A.D., Smith, R., 1992. Methods to measure the polymerization shrinkage of light-cured composites. *J. Prosthet. Dent.* 68, 56–58.
- Reed, B., Dickens, B., Dickens, S., Parry, E., 1996. Volumetric contraction measured by a computer-controlled mercury dilatometer. *J. Dent. Res.* 75, 2184.
- Rueggeberg, F.A., Jordan, D.M., 1993. Effect of light-tip distance on polymerization of resin composite. *Int. J. Prosthodont.* 6, 364–370.
- Rosin, M., Urban, A.D., Gartner, C., Bernhardt, O., Splieth, C., Meyer, G., 2002. Polymerization shrinkage-strain and microleakage in dentin-bordered cavities of chemically and light cured restorative materials. *Dent. Mater.* 18, 521–528.
- Vandewalle, K.S., Ferracane, J.L., Hilton, T.J., Erickson, R.L., Sakaguchi, R.L., 2004. Effect of density on properties and marginal integrity of posterior resin composite restorations. *Dent. Mater.* 20, 96–106.
- Venhoven, B.A., De Gee, A.J., DAVIDSON, C.L., 1993. Polymerization contraction and conversion of light-curing BisGMA-based methacrylate resins. *Biomaterials* 14, 871–875.
- Watts, D.C., Al Hind, A., 1999. Intrinsic “soft-start” polymerization Shrinkage-kinetics in an acrylate-based resin-composite. *Dent. Mater.* 15, 39–45.
- Watts, D.C., Cash, A.J., 1991. Determination of polymerization shrinkage kinetics in visible-light-cured materials: methods development. *Dent. Mater.* 7, 281–287.