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Dynamics of composite polymerization mediates the development of cuspal strain

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ABSTRACT

Objectives. In the current study, we used electronic speckle pattern interferometry (ESPI) to measure tooth deformation in response to polymerization of five resin composites with a range of polymerization shrinkage. Our hypothesis was that composites with higher polymerization shrinkage should cause more cuspal strain as measured by ESPI.
Methods. Standardized MOD cavities were prepared and placed into the ESPI apparatus before the cavities were filled with composites ($n = 10$). The ESPI apparatus was constructed to measure the out-of-plane displacement of the lingual cusps of the teeth during the polymerization of the restorative material. A thermocouple was attached to the specimen to monitor thermal changes throughout the polymerization process.
Results. Experiments with empty preparations demonstrated that the ESPI technique was temporally responsive and sensitive to dimensional changes. However, the correlation between polymerization shrinkage of composite resins and ESPI-measured tooth deformation was not straightforward. In particular, a flowable material did not deform the tooth significantly more that a conventional hybrid. Further, an experimental silorane material (with the lowest axial shrinkage) induced the least tooth deformation.
Significance. We concluded that ESPI is a viable method for assessing cuspal strain induced by shrinkage of bonded composite restorations, but that polymerization shrinkage data may overestimate shrinkage-induced tooth deformation. The rate of polymerization shrinkage appeared to mediate the development of cuspal strain.

1. Introduction

Polymerization shrinkage of resin composites remains a major obstacle to their clinical success as dental restorative materials. All contemporary composite materials shrink during polymerization, resulting in a volumetric reduction ranging from 1.5 to 5% depending on the molecular structure of the monomer, the amount filler, and the rate of cure [1]. Photo-initiated polymerization, which occurs more rapidly than chemically initiated reactions, may produce more shrinkage stress [2]. Polymerization shrinkage is clinically undesirable because it stresses tooth-composite adhesive interfaces and deforms the tooth itself [3]. These stresses may cause microfractures in the tooth enamel, marginal gap formation and subsequent microleakage, or pain [4,5]. All of these factors limit the longevity and success rate of resin com-

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posites, particularly in posterior teeth where the restorations are largest and shrinkage is therefore the greatest [6–7].

Unfortunately, measuring polymerization shrinkage in a clinically meaningful context is extremely difficult. Several experimental methods have been used to assess polymerization shrinkage including dilatometry, linear displacement, strain gauges, and the bonded disc method [8–11]. Each of these methods have advantages and disadvantages, but all are greatly influenced by the testing configuration (such as the direction of light, dimensions of the specimens, or specimen constraint) [12]. Furthermore, none of these tests predicts the consequences of polymerization shrinkage on tooth-restoration stresses, which is the clinical "bottom-line".

The dimensional complexity of intracoronal dental restorations is one reason why laboratory methods that measure polymerization shrinkage are poor predictors of clinical outcomes. As the composite polymerizes and shrinks, the stresses that develop at the tooth-composite adhesive interface and within the tooth itself depend upon the cavity shape, size, C-factor, modulus of the tooth, the developing modulus of the composite, and the rate of polymerization [1,2,10,13]. These factors combine and interact simultaneously in complex ways, translating polymerization shrinkage into tooth stresses. The deformation of the tooth is a clinically significant outcome, as well as being a reasonable indicator of other effects of polymerization shrinkage such as interface stress [14].

Electronic speckle pattern interferometry (ESPI) was developed in the late 1970s as a method to measure minute changes in dimension in real time [15]. In this method, a laser beam is split and directed half to a plane of reference and half to the surface of interest. The reflected light from both surfaces is then recombined and allowed to optically interfere. As the surface of interest changes with time, the interference patterns produce optical fringes in a so-called speckle pattern. This speckle pattern can be converted to distance based on the wavelength of the laser. Electronic speckle pattern interferometry has been previously used in biomedical research to measure deformation in composite materials, dentin and bone [16–18]. The purpose of the current study was to apply ESPI to the problem of polymerization-induced tooth deformation, thereby providing a real-time assessment of the complex development of stresses that accompanies the polymerization of a bonded resin composite. We selected five resin composite materials with a range of known polymerization shrinkage, then measured tooth deformation with ESPI when these materials were bonded into a standardized cavity preparation and polymerized. Our hypothesis was that ESPI could successfully measure tooth deformation resulting from polymerization shrinkage and that we could relate polymerization shrinkage to tooth deformation.

2. Materials and methods

2.1. *ESPI experiments*

Fifty human, non-carious, erupted, lower third molars of known and similar dimension (11.4 ± 0.44 mm/9.1 ± 0.34 mm) were clean by pumicing. The teeth were examined under 10X magnification (Wild, Heerbrugg, Heerbrugg, Switzerland) for the presence of microfractures or other structural defects, and only teeth without visible defects were included in the study.

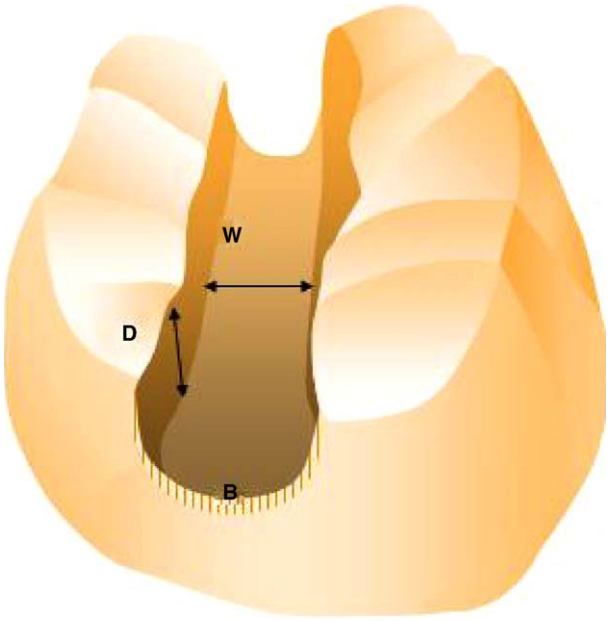

Fig. 1 – A schematic representation of the MOD cavity used in this study. The occlusal preparation is 2 mm wide (W) and 2.5 mm deep (D). A 1 mm bevel (B) was prepared at the enamel margins to maximize composite-enamel bonding in these areas.

Standardized MOD cavities (2 mm × 2.5 mm × 11 mm) were prepared using coarse then superfine diamond burs operated in a high-speed hand-piece using copious air-water spray (Fig. 1). Restoration's depth was chosen to ensure that composite restorations could be polymerized in a single increment [19]. The MOD configuration (without approximal boxes) was chosen to conservatively allow cuspal flexure yet retain a standardized preparation size. A 1 mm bevel was prepared at the enamel margins to maximize composite-enamel bonding in these areas. The buccal cusp of each tooth was etched for 60 s, rinsed, then air dried. The entire surface of the etched cusp was then bonded to a steel base that provided maximum stability of each specimen during ESPI measurements. The teeth were randomly assigned to receive different restorative materials ($n = 10$, Table 1).

The materials used in this study were chosen on the basis of common clinical use and a spectrum of polymerisation shrinkage values previously reported in the literature [20,21]. Furthermore, a new experimenal low shrinking material based on silorane chemistry was compared to conventional dimethacrylate resins. Although shrinkage data collected from different litterature sources has several limitations (e.g. variable test methods, irradiation conditions, curing interval, operator), all shrinkage data used in the current study were calculated using the bonded disk method [9] and comparable time intervals (Table 1), faciliting reasonable comparison among materials. Each material was used with a compatible, recommended self-etching adhesive system (Table 1) that was added to the tooth, then cured immediately in situ (20 s; Free-

Table 1 – Materials used in the study

Adhesive system	Composite resin	Company	Code	Description	Shrinkage value (%)
AdheSE LOT # F44228	Tetric Flow LOT # C34930	Ivoclar-Vivadent Schaan, FL	F	Flowable, free-radical polymerization.	3.8[a]
AdheSE LOT # F44228	Tetric Ceram LOT # E1083	Ivoclar-Vivadent Schaan, FL	C1	Conventional hybrid, free-radical polymerization	2.3[a]
OptiBond Solo LOT # 205974	Premise LOT # 011567	KerrHawe SA Bioggio, CH	C2	Tri-modal fill hybrid, free-radical polymerization	1.6[b]
Xeno III LOT # 0410002526	QuiXfil LOT # 0412000805	DeTreyDentsply Konstanz, D	N	Nanoparticle-enhanced fill free-radical polymerisation	1.7[a]
Hermes Bond LOT # HA-395	Hermes LOT #151174-2	3 M-ESPE AG Seefeld, D	Si	Silorane-based, cationic polymerisation	0.9[a]

[a] Data compiled from references [20,21].
[b] Manufacturer information.

light 2, 3 M-ESPE AG, Seefeld, Germany). However, the enamel margins of the preparations also were acid-etched before the application of the adhesives, to maximize bonding in these areas of the preparation.

A thermocouple was attached to the internal face of the buccal cusp (away from the laser) to monitor thermal changes throughout the polymerization process. The lingual cusps of the specimens were sprayed with a contrasting agent (TiO_2 powder, Vita, CEREC Powder, VITA Zahnfabrik, Bad Säkingen, Germany) to compensate for the weak reflectivity of the enamel. The specimens were then placed into the ESPI apparatus before the cavities were filled with composite (0.15 g of resin per cavity). The placement of the composite was made without matrix bands to avoid interfering with ESPI measurements on the lingual cusp. This procedure worked well for all materials except the flowable because of its relatively low viscosity. Thus, for the flowable, adhesive tape was used to seal the proximal surfaces. To avoid premature polymerization of the resin, all these procedures were performed under a yellow light. The light-curing unit (Freelight 2, 1000 mW/cm^2, 3MESPE AG, Seefeld, Germany) was placed 5 mm above the cavity to allow the 8 mm diameter curing tip to deliver a 12 mm diameter beam of light. The composite resins were bulk cured for 40 s without moving the light.

The ESPI apparatus measured the out-of-plane displacement of the lingual cusps of the teeth during the polymerization of the restorative material (Fig. 2) in a manner described briefly as follows. A 638 nm helium-neon laser beam (10 mW) was passed through a collimating lens and split into two beams of equal intensity. One beam (reference beam) was directed toward a reference plane while the other beam (sample beam) was directed toward the lingual surface of the specimen. A charge coupled device (CCD) video camera captured images from the specimen. The reference beam was directed in such a way that it intersected the path between the specimen and the video camera. A partial mirror deflected the reference beam into the video camera causing it to interfere with the light reflected from the specimen. Due to the monochromatic properties of the laser light, the specimen and reference beams constructively or destructively combine to produce a speckle pattern of fringes over the lingual surfaces (Fig. 3). The speckle images were recorded by the CCD video camera, digitized and stored in a computer over 240 s at the rate of 8 images per second. The out of plane displacement was calculated using the equation:

$$d = \lambda \frac{n}{2}$$

where λ was the wavelength of the laser (632.8 nm) and n the number of fringes observed in the speckle pattern.

2.2. *Microhardness analysis*

Microhardness measurements were used as an indirect method to assess the extent of polymerization in the resin material inside the cavity [22]. Immediately after the ESPI test, each specimen was sectioned along its mesio-distal axis using a low speed diamond saw (Isomet, Buehler, Lake Bluff, USA) to allow the comparison of Vickers microhardness (VHN) at the surface and depth of the resin material. Microhardness was measured by indenting the resin material at a load of 0.5 kg for 15 s (Hauser Instruments, Biel, Switzerland).

2.3. *Statistics*

Cumulative out-of-plane displacement of the lingual cusps were compared at 240 s for the different materials with ANOVA

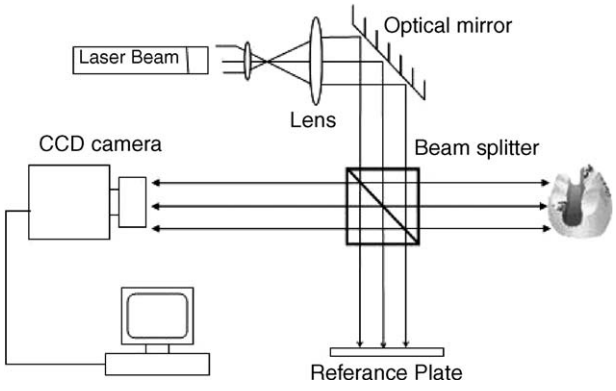

Fig. 2 – Schematic diagram of the ESPI apparatus. The laser beam is split and directed half to a plane of reference and half to the surface of interest. The reflected light from both surfaces is then recombined and allowed to optically interfere. A CCD (charged-couple diode) camera is used to capture the images. As the surface of interest changes, the interference patterns produce optical fringes in a so-called speckle pattern.

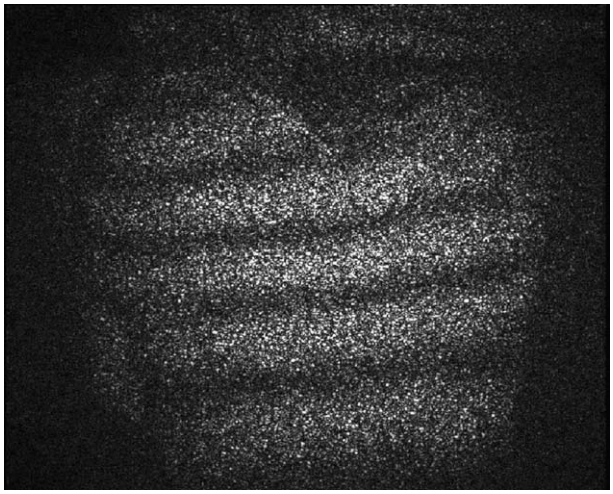

Fig. 3 – Due to the properties of the laser light, the specimen and reference beams combine to produce a speckle pattern of fringes over the lingual surface of the specimen during light irradiation.

and Tukey post hoc analysis, using $n = 10$ and an α of 0.05 as the critical value. Data for microhardness were compared at the surface and depth of each material using paired *t*-tests ($\alpha = 0.05$). No statistical comparisons among materials were done for microhardness.

3. Results

3.1. *ESPI method*

Conditions with empty cavity preparations were used to assess the responsiveness and sensitivity of the ESPI technique (Fig. 4). When empty preparations were irradiated with blue light, the temperature of the tooth rose exponentially for the 40 s of irradiation, then decayed exponentially after irradiation ceased, returning to baseline about 200 s after the light was turned off. The temperature rose as much as 6 °C, although there were reasons to suspect that the thermocouple overestimated the absolute temperature rise

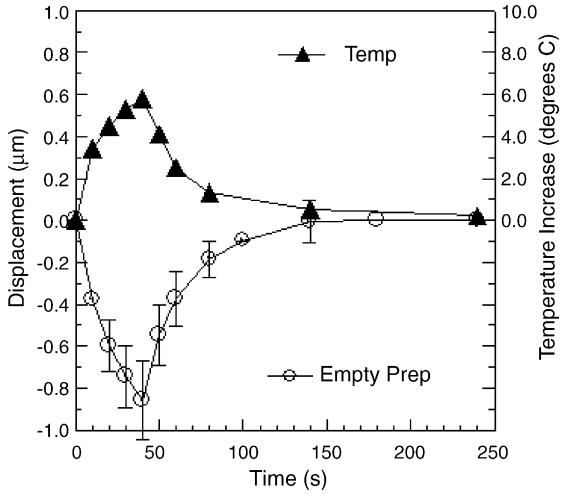

Fig. 4 – Dilatation of empty cavities during 40 s of blue light irradiation.

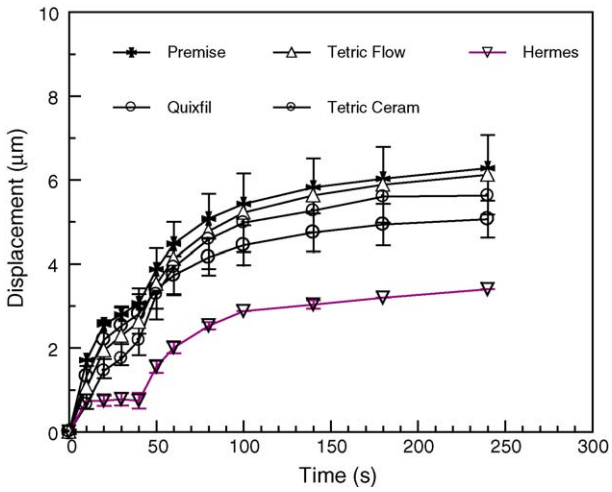

Fig. 5 – Cuspal displacement during light curing of the restorative materials.

(see Section 4). Expansion of as much as 0.9 μm coincided with the peak temperature of the tooth, and contraction of the tooth accompanied cooling in largely the same temporal pattern. These trends suggested that the ESPI measurements were temporally responsive to changes in cuspal position. The empty-preparation experiments also demonstrated the sensitivity of the ESPI technique. Changes in as little as 0.3 μm were detectable (Fig. 4) given the level of variation, which was about 10%.

3.2. *Cuspal deformation and composite polymerization*

As expected, all materials caused cuspal displacement (cusp tip-to-cusp tip contraction, Fig. 5). The free cusp of the system was displaced 2.5–6 μm, with at least 50% of the movement occurring during the 40 s of irradiation. Cuspal deflection occurred in an exponential pattern for all materials. However, the silorane material caused a distinctly different cuspal displacement pattern. During the first 10 s of irradiation, about 0.75 μm of cuspal displacement occurred, but change in displacement was zero fror the next 30 s and increased only after the irradiation of the material stopped. For all materials, variation of displacement was about 15% among the replicates ($n = 10$).

The total cuspal displacement (at 240 s) was different among the materials tested (Fig. 6). Premise, Tetric Flow, and Tetric Ceram were statistically equivalent with cumulative cuspal displacements of 5.5–6 μm. Quixfil caused less cumulative cuspal displacement (5 μm), which was statistically different than Premise and Tetric Flow but not different from Tetric Ceram ($p < 0.05$). Preparations filled with the silorane material (Hermes) exhibited the lowest cuspal displacement (about 3.5 μm) of the materials tested, which was statistically different from the other materials tested ($p < 0.05$).

3.3. *Microhardness*

As expected, the microhardness of the cured composites at the pulpal floor of the preparations suggested that curing of the materials was complete (Table 2). For most materials, the

Table 2 – Mean Vickers hardness (VHN) according to the composite material and the depth of measurement ($n = 10$)

	Tetric flow	Tetric ceram	Premise	QuiXfil	Hermes
Top	64 (5)	78 (7)	81 (6)	79 (6)	72 (8)
Bottom	65 (9)	72 (8)	76 (9)	70 (8)	66 (9)

Results connected by bars are statistically equivalent (paired t-tests: $\alpha = 0.05$).

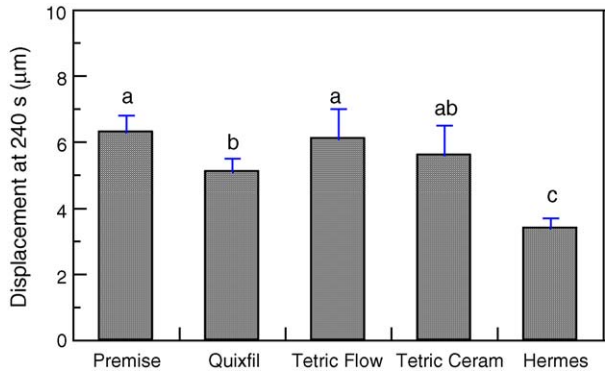

Fig. 6 – Total cuspal strain for the different materials tested at 240 s. Means denoted by the same letter are not statistically different ($p < 0.05$).

mean hardness values measured at the bottom of the cavity were 6–11% lower compared to those measured at the surface of the specimens. Premise had the highest microhardness (top: 81 VHN, bottom: 76 VHN).

4. Discussion

Based on the current results, the ESPI technology was well suited to evaluate tooth deformation induced by the polymerization shrinkage of tooth-bonded composite materials. The single biggest advantage of ESPI was its ability to assess tooth deformation without physically contacting the tooth. Using the laser and the principles of optical interference, changes as small as 0.3 μm in cuspal position could be detected. Many other techniques to measure shrinkage of this nature like strain gauges and LVDTs rely on mechanical detection devices [23,24]. These techniques are less sensitive to detect cuspal strain because the detectors are in contact or even bonded to the structure undergoing deformation. ESPI had the added advantages of being able to assess tooth deformation in real time under clinically relevant dimensional parameters [25] and can be used repeatedly on the same sample because of the non-destructive nature of the test. As an example, the visco-elastic deformation of teeth under cyclic loading could be evaluated using ESPI [26].

A primary outcome of the current study was a lack of strict correlation between shrinkage values of the composite materials and the degree of induced cuspal strain. For example, the cumulative amount of cuspal displacement at 240 s for the flowable material, which exhibits the highest shrinkage value was 6.0 μm, whereas the conventional hybrid which shrinks less was nearly 5.6 μm (Fig. 6). The ability of the material to flow and relieve developing shrinking stresses may have reduced cuspal strain [27]. On the other hand, the silorane material, which had the lowest axial shrinkage, also caused less cuspal displacement (Fig. 6) supporting our hypothesis. Thus, the current results suggest that axial shrinkage may not be predictive of tooth deformation. This observation has significant consequences for composite development because it implies that small, incremental decreases in polymerization shrinkage may not reduce tooth deformation during polymerization [1]. Recently, Kleverlaan and Feilzer have evaluated the relationship that exists between shrinkage, contraction stress, elastic modulus and flow properties of a range of composite resins [28]. They confirmed the inverse relationship that exists between the tensile modulus and the shrinkage value of different materials but they also demonstrated a positive correlation between tensile modulus and contraction stress. Therefore, both the shrinkage value and the modulus of the composite resins are important factors contributing to stress developmment in polymerizing resins. Braga et al. also reported that the elastic modulus acquired during polymerization is important factor in contraction stress development [29].

Among the materials tested, the silorane caused significantly less tooth deformation than the other materials (Fig. 5), which is in agreement with previous reports [20,24]. Although siloranes exhibit low polymerization shrinkage, they also exhibited an atypical time-cuspal displacement curve, with a 30 s period of no dimensional change. One hypothesis for this behavior is that the siloranes were slower to polymerize, allowing time for flow of material and stress relaxation, resulting in a lower final degree of cuspal strain. If true, this hypothesis would suggest that the degree of overall axial shrinkage of these materials is not as important as the rate at which shrinkage occurs. Recent studies with siloranes have demonstrated a polymerization reaction with a slow onset because of time needed for cation formation [1,30]. Other materials in the current studies polymerized via free radical mechanisms, which are inherently faster [30]. Fig. 5 also suggests that about 50% of the total tooth deformation occurred in the 40 s irradiation period, albeit some tooth deformation continued through 240 s. The current study supported previous studies that indicate that properties of light-activated composites continue to change after irradiation stops [12].

Several alternative explanations might account for the different abilities of materials to induce cuspal strain. For example, if the material did not cure 'completely', then less polymerization shrinkage may have accounted for a lower cuspal displacement [30]. However, hardness data (Table 2) suggest that this was not the case. Further, the risk that a partial debonding of the composite material from the cavity floor has occured cannot be ruled out. Although no microleakage test was performed to assess debonding, the good bonding potential of these adhesive resins to etched enamel and the low C factor of the cavities used in this study

(<2.1) support the idea that resin-tooth debonding was not a problem. This observation is in agreement with a previous study that showed a limited reduction in bond strength for conventional methacrylate-based adhesive resins applied to MOD cavity walls compared to flat control surfaces [31]. This also apply to the adhesive resin of the silorane material which contains methacrylate groups. Furthermore, tooth size, preparation size and preparation design were all standardized or controlled to ensure that the different cuspal displacement curves for the materials were from the polymerization chemistry of the materials and not systematic artifacts.

The increased temperatures (Fig. 4) resulting from light irradiation and composite polymerization were probably accurate temporally, but the absolute temperature increases seen were probably less accurate because the thermocouple was exposed to the light. Previous reports indicate the direct exposure of thermocouples to blue light may itself induce current flow in the thermocouple and lead to inaccurately high temperature readings [32]. Of more importance to the current study was the close temporal correlation between the thermocouple and laser measurements (Fig. 4), which indicates that tooth deformation was being measured in real time and was not delayed or otherwise biased.

The tooth expansion observed in conjunction with temperature increases in the tooth may have clinical consequences for the final amount of cuspal strain and tooth stress during placement of bonded composite restorations. Although tooth expansion was only measured with empty cavities exposed to blue light, tooth expansion is just as likely to occur when the composite resin is polymerized inside the cavity. The net temperature increases from both the curing light and the polymerization reaction of the composite resin may cause a transient increase in tooth size that will essentially create a slightly oversized restoration. Upon cooling, the tensile stresses that create the contraction of tooth cusps would be partly relieved as the tooth shrunk, although the stress generated during curing undoubledly outweigh the stress relief provided by the expansion effect. Further, temperature increase in composite resins during curing may also reduce viscosity and contribute to a slower stress development [30]. This idea, although speculative, may indicate that a curing process with no net temperature change is not desirable.

5. Conclusion

The current study has shown that ESPI is a viable method for assessing cuspal strain induced by shrinkage of bonded composite restorations, but that polymerization shrinkage data may overestimate shrinkage-induced tooth deformation. The rate of polymerization shrinkage appeared to mediate the development of cuspal strain.

Acknowledgments

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