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Impact of similarity in chemical composition of light-polymerized resin composites on post-gel strains and interface integrity

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Abstract The post-gel strains of two light-polymerized resin composites having similar chemical composition, Filtek P60 and Filtek Z250, was assessed by strain-gauge analysis at the ex vivo level. The restoratives were tested in cavity factor 5 during light-polymerization and water-soaking for 24 h. Strain-gauge signals were digitalized by a data acquisition system and were displayed in a computer by corresponding software at a sample rate of 20 Hz. The strain data were used to compare post-gel strains, polymerization velocity, and total volumetric change of both materials. Evaluation of microleakage and scanning electron microscopy was also undertaken to elucidate effects of post-gel strains at the tooth-restorative interface. Microstrains of Filtek Z250 were lower than those of Filtek P60 and the differences between post-gel strains at several different time intervals were significant ($P < 0.009$). There was a correlation between polymerization velocity and total volumetric change. Microleakage evaluation and scanning electron microscopy did not reveal any signs of debonding at the interface for both materials. We conclude that the similarity in chemical composition of light-polymerized resin composites is not a determinant for post-gel strains. The tooth-restorative interface can withstand high post-gel strains arising from polymerization of resin composites.

Introduction

One of the main drawbacks of present-day dental resin composites is polymerization shrinkage, which leads to marginal gap formation, marginal discoloration, and secondary caries [1, 2]. These clinically manifested problems frequently compromise long-term efficacy of resin composites and indicate replacement of the restorations [3]. In essence, for all commercially-available resin composites, volumetric shrinkage is theoretically unavoidable, because the closer packing of the molecules in a polymer network versus monomers inevitably leads to a reduction of volume. Accordingly, shrinkage-dependent clinical complications have been attributed to development of contractive forces during polymerization, resulting in internal stresses within the restorative that tend to disrupt the bond to cavity walls [4–6].

At present, light-polymerized resin composites have replaced chemically-polymerized resin composites, mainly because their rapid polymerization reaction and dentist-controlled initiation of polymerization upon placement of the restorative into the cavity. Despite several cited advantages and extensive clinical use of light-polymerized resin composites, there is a potentially narrow gap between their unsurpassed benefits and adverse effects in the context of long-term clinical effectiveness. Because the gel stage is

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limited for light-polymerized composites, the polymerization reaction immediately converts the viscous-plastic material into a rigid-elastic phase, inhibits resin flow, and leads to built-in stresses at the tooth-restorative interface [7–10]. Eventually, adhesive failure and consequent marginal leakage, secondary caries and even cusp fractures may occur, if shrinkage stresses exceed the dynamic mechanical properties of the interface or tooth [10, 11].

The magnitude of polymerization shrinkage stress (or strain) is significantly influenced by material properties such as resin composition, monomer size, filler type and loading, stiffness of the material, and clinical application techniques, such as the volume of polymerized material, mode and velocity of polymerization and direction of light application [12–20]. The geometric configuration of the tooth cavity, "c-factor": the ratio of bonded to unbonded external surface area of the specimen also has a decisive effect on polymerization shrinkage [21, 22]. Hence, polymerization shrinkage is a multifaceted phenomenon, which is dependent on several factors including material properties of the resin composite and dentist-related factors.

In the last 10 years, the clinical performance of composites been amended markedly through use of more stable polymerization promoters for enhanced color stability, integration of high-concentration finely ground fillers to improve mechanical properties, and application of dentin bonding agents to grant long-term biomechanical performance of the tooth-restorative interface [14, 23]. Despite the large pool of evidence regarding the physical properties and clinical outcome of light-polymerized resin composites, it is unknown whether composites having very similar chemical compositions possess similar shrinkage characteristics. With regard to the clinical significance of shrinkage potentials of resin composites, the purpose of this study was to compare the post-gel characteristics and consequent effects at the tooth-restorative interface of two commercially-available light-polymerized resin composites having similar chemical composition.

Materials and methods

Test materials

A packable composite Filtek P60 (3M Dental Products, St. Paul, Minn.) and a hybrid Filtek Z250 (3M Dental Products, St. Paul, Minn.) were used in this study. According to the information released by the manufacturer, Filtek P60 is a light-activated, radiopaque restorative composite designed for use in posterior and indirect restorations. It contains BIS-GMA (Bisphenol A digylcidyl ether dimethacrylate), UDMA (urethane dimethacrylate), and BIS-EMA (Bisphenol A polyethylene glycol diether dimethacrylate); the filler is zirconia/silica. The inorganic filler loading is 61% by volume (without silane treatment) with a particle size range of 0.01–to 3.5 μm. The average particle size is 0.6 μm. UDMA and BIS-EMA resins have a higher molecular weight and therefore fewer double bonds per unit weight. These resins impart a greater hydrophobicity, and the higher molecular weight results in decreased shrinkage. Filtek Z250 is a light-polymerized, radiopaque composite specifically designed for anterior and posterior direct or indirect restorations. The composition and particle size range of Filtek Z250 is the same as Filtek P60, but filler loading is 60% by volume. Although these composites have the same chemical composition, their indications for use and static and dynamic elastic moduli of elasticity are different [24].

Cavity preparation and experimental set-up

The experiments were undertaken on 10 freshly harvested bovine central incisors selected among 42 teeth on the basis of dimensional requirements, lack of any carious lesions, microfractures, or abrasion cavities. The teeth were randomly divided into two equal groups and assigned for the experiments for the two composites. A two mm-deep occlusal cavity having 6 mm × mm dimensions and without bevel was prepared on the labial surface of each tooth by use of high-speed handpiece burs under copious water cooling. Then, each tooth was secured into a petri dish with a pattern resin (GC Pattern Resin, GC Europe N.V., Leuven, Belgium) to ensure optimum immobility of the tooth during the experiments. Each cavity was etched with Scotchbond etchant (3M Dental Products, St. Paul, Minn.) for 15 s, rinsed with water, and dried gently with an air syringe. Two consecutive coats of Single Bond adhesive (3M Dental Products, St. Paul, Minn.) was applied to enamel and dentin, dried gently by air for 2–5 s and light-polymerized for 10 s.

First, an approximate thickness of 0.2 mm composite resin was placed on the base of the cavity followed by placement of a linear strain-gauge (EA-06-015CK-120; Micromeasurements Group, Raleigh, NC; resistance 120.0 ± 0.3% Ω; gauge factor: 2.04 ± 2.0%). The surface tack of the resin composites was adequate to ensure appropriate adhesion with the polyimide backing of the gauges [20]. Then, resin composite was placed incrementally over the strain gauge, while extensive care was taken not to displace the gauge and to avoid entrapment of air. The measuring grid of the gauge was aimed towards the occlusal part of the cavity (Fig. 1). The rationale behind this application was to qualify and quantify the post-gel shrinkage strains within the resin composite in cavity configuration 5 [c-factor 5: a composite resin filling having 5 walls bonded to the cavity and 1 unbonded surface (top)]. Each gauge was wired separately into a Wheatstone bridge. The lead wires of the gauge were waterproofed by application of an air-drying nitrile rubber, an air drying acrylic, and an air drying polyurethane in the given order (M Coat B, M Coat D, and M Coat A, respectively; Micromeasurements Group, Raleigh, NC) [25]. During experiments, the strain-gauge signals were digitalized by a data acquisition system (ESAM Traveller 1, Vishay Micromeasurements Group, Raleigh NC, U.S.A) and were displayed in a computer by a corresponding software (ESAM; ESA Messtechnik GmbH, Olching, Germany) at a sample rate of 20 Hz.

Quantification of post-gel strains

After final placement of the resin composite, pre-gel strains occured within the material due to the external force applied by the hand instrument. Instead of calibrating the gauges, relief of pre-gel strains was monitored using the data acquisition system with corresponding software at a sample rate of 250 Hz. Upon relief of pre-gel strains, the gauges were calibrated to zero. Consecutively, each specimen was light-polymerized at a distance of 0.5 mm from the surface of the composite for 40 s using a light-polymerizing unit (Hilux; Benlioğlu Dental Inc, Ankara, Turkey). The light-polymerizing unit was calibrated before polymerization procedures; the intensity of the polymerizing light source was 400 mW/cm^2 as measured by a polymerizing light tester (Hilux Dental Curing Light Meter; Benlioğlu Dental Inc, Ankara, Turkey). The diameter of the light tip was 1 cm. During polymerization, the strain-gauge signals were simultaneously digitalized by the data acquisition system and displayed in the computer by the corresponding software. After light-polymerization, each specimen was left exposed to air for 6 min to observe post-gel changes. Then, the petri dish was immediately filled with water at 24 °C to explore time-dependent hygroscopic expansion of the composite resin, if any. Simultaneously, data acquisition (strain versus time curve) was undertaken for 24 h for each specimen.

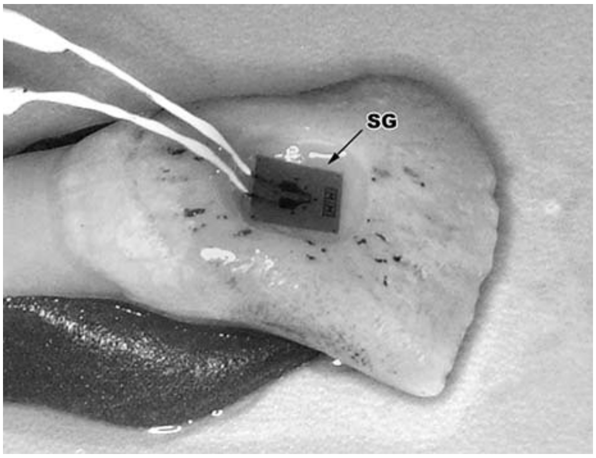


Fig. 1 The linear strain-gauge (SG) with isolated lead foils placed in the base of the cavity having a c-factor of 5

Determination of shrinkage (or expansion) velocity

To compare early and late polymerization velocities (unit per second) of each restorative, the curve sections between 20–40, 80–100, 300–400, 900–1000, and the last 500 s (85,900–86,400) were evaluated using linear regression analysis. The slope of the curve (derivative) between the determined time intervals was used as an indicator of dimensional change velocity and type (shrinkage versus expansion) [20].

Measurement of total volumetric change

The area under the curve of post-gel strain versus time of each test material was calculated using the trapezoidal method. The area was used as an indicator for volumetric change (shrinkage or expansion) and used to compare both materials. Because there was a drift from compressive to tensile strains in the early stages of the experiment for Filtek P60 specimens, the difference between both areas were calculated and compared with that of Filtek Z 250.

Determination of microleakage

The strain gauge wires of the specimens were cut. The entire tooth and root surface excluding the composite restoration and the interface was coated with two consecutive layers of M Coat A (Micromeasurements Group, Raleigh, NC) and the specimens were stained with crystal violet for 24 h. The specimens were then embedded in clear autopolymerized methylmethacrylate resin and several sections were obtained from the middle of the cavity using a high-speed sawing machine (Isomet 4000, Buehler GmbH, Düsseldorf, Germany).

The sections were examined and photographed in an inverted microscope (Olympus IX70, Tokyo, Japan).

Scanning electron microscopy analysis

Because the restorations used for strain-gauge experiments were assigned to microleakage experiments, scanning electron microscopy analyses were undertaken on six specimens randomly selected among 10 freshly prepared specimens following the same techinque used for strain-gauge experiments. Following removal of the roots with a water-cooled high-speed handpiece, the specimens were sectioned longitudinally, bisecting the restorations in the inciso-cervical direction. An individual impression of each interface was taken with an elastomeric material (Aquasil-LV, Dentsply Caulk, Milford, DE) and replicas were created by casting the impressions with an epoxy resin (Epo-thin, Buehler Ltd., Lake Bluff, IL). The sectioned surfaces were then polished with waterproof papers of decreasing abrasiveness up to 4,000 grit (Buehler GmbH, Düsseldorf, Germany). After polishing, specimens were soaked in 6 mol/L HCL for 30 s, followed by immersion in 5% sodium hypochlorite for 5 min to remove collagen that was not resin protected. The specimens were dehydrated in ascending grades of ethanol (50, 75, 95 and 100%) followed by immersion in hexamethyldisilazane for 10 min, placed on a filter paper inside a cover glass vial, and air-dried at room temperature [26]. Thereafter, the specimens were gold sputter-coated and the adhesive interface at the underlying dentin was observed by scanning electron microscope (JEOL 6400, Tokyo, Japan). Epoxy casts were investigated as with actual specimens to control for artifact formation.

Statistical analysis

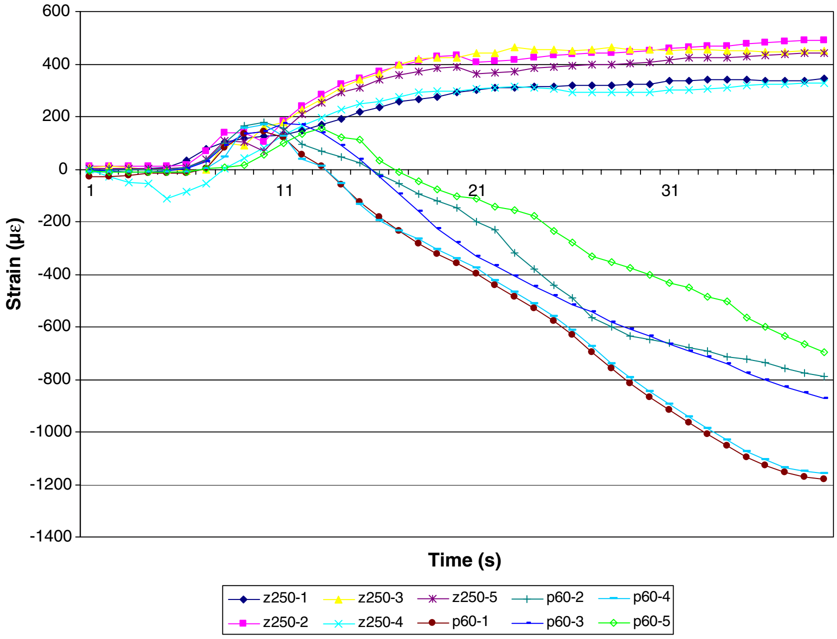
Because compressive strains occured as a sequel of polymerization for Filtek P60 absolute values were used for quantitative comparison. The median of the data at 40, 400, 1000, 76400, and 86400 s (24 h) was calculated for both materials and compared using the Mann–Whitney Test at a significance level set at 95%.

Results

Post-gel strains within materials

Time-dependent post gel strains of test materials between 0 and 40 s are presented in Fig. 2. For all specimens of P60, there was an initial expansion of the material exhibited by tensile strains below 200 $\mu\varepsilon$, which immediately converted to compressive strains within the first 20 s of light polymerization. Excluding one specimen, which exhibited initially compressive strains (shrinkage), Z250 specimens yielded tensile strains (expansion) during light-polymerization. Strain vs time curve of test materials in the first 1000 s are presented in Fig. 3. There was a high-rate increase in compressive strains in the first 100 s of P60, which

Fig. 2 Post-gel strains versus time curves of Filtek P60 and Z250 specimens (n = 5) between 0 and 40 s



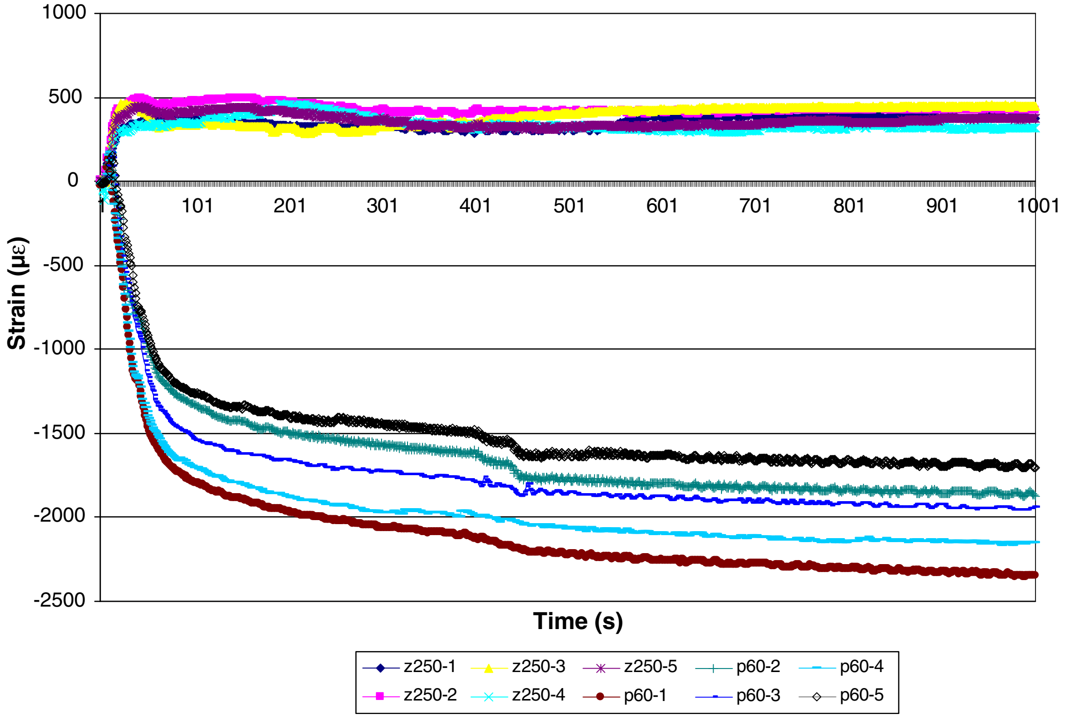


Fig. 3 Post-gel strains versus time curves of Filtek P60 and Z250 specimens (n = 5) between 0 and 1000 s

continued to decrease gradually with a steadily low rate after water soaking. The high-rate increase in tensile strains of Z250 specimens tended to reach a plateau after 50 s and gradually decreased with a steadily rate. The magnitude of strains of test materials at 40, 400, 1,000, 76,400, and 86,400 s (24 h) are presented in Table 1. The mean rank orders of microstrains of Filtek Z250 (3.00) were lower than those of Filtek P60 (8.00) and the differences between post-gel strains at 40, 400, 1,000, 76,400, and 86,400 s (24 h) were significant ($P < 0.009$).

Shrinkage (or expansion) velocities at different time intervals and total volumetric change

Early- and late-polymerization shrinkage velocities of both materials are presented in Table 2. At the outset (20–40 s), Filtek P60 experienced high shrinkage (negative slope value) associated with high velocity, whereas Filtek Z250 had expansion (positive slope value) with a relatively lower velocity. Both materials experienced shrinkage with lower velocities in consecutive intervals until the termination of the experiments.

Table 1 Microstrains ($\mu\varepsilon$) of test materials at 40, 400, 1000, 76,400, and 86,400 s (24 h)

		40 s	400 s	1000 s	76,400 s	86,400 s
Filtek P60	Mean	413	333	387	316	285
	Std. Error of Mean	29	21	22	23	35
	Median	443	322	378	327	286
	Std. Deviation	66	47	51	52	78
	Minimum	339	285	316	246	190
	Maximum	489	411	447	376	371
Filtek Z250	Mean	961	1797	2002	2120	2199
	Std. Error of Mean	88	119	110	90	76
	Median	911	1795	1941	2100	2195
	Std. Deviation	198	268	246	201	171
	Minimum	745	1476	1706	1834	1936
	Maximum	1179	2127	2342	2379	2388

Table 2 Early and late polymerization shrinkage velocities (slope of the curve) of Filtek P60 and Filtek Z250

	20–40 s		80–100 s		300–400 s		900–1000 s		85,900–86,400 s	
	Slope	R^2	Slope	R^2	Slope	R^2	Slope	R^2	Slope	R^2
Filtek P60	–34.701	0.97	–3.2077	0.98	–0.4515	0.93	–0.1612	0.54	–0.0064	0.05
Filtek Z250	2.3671	0.97	–0.0857	0.03	–0.209	0.83	–0.0033	0.001	–0.0105	0.07

This situation resulted in increase in shrinkage for Filtek P60, whereas low-rate shrinkage of Filtek Z250 tended to compansate polymerization expansion. The area under the strain vs. time curve of Filtek P60 (1773652 units) was higher than that of Filtek Z250 (371856,8 units), revealing that, the total volumetric change of Filtek P60 was approximately 4.5 times higher than that of Filtek Z250.

Microleakage and Scanning electron microscopy

Representative tooth-restoration sections of Filtek P60 and Filtek Z250 restorations are presented in Fig 4a and b, respectively. In all sections obtained, no signs of microleakage was observed at the tooth-restoration interface. Scanning electron microscopy findings are shown in the micrographs (Figs. 5 and 6). In either groups, bonding failures or separations at the enamel and dentin interfaces were not evidenced. The bonding interface created by Single Bond was consistently present, showing a thick hybrid layer and regularly-shaped resin tags.

Discussion

In the present study, assessment of the polymerization shrinkage characteristics of two light-polymerized resin composites, Filtek P60 and Filtek Z250, having similar chemical composition was undertaken using strain-gauge analysis. The rationale behind using strain-gauge analysis was that parametric studies of pre-polymerized and photoinitiator-free materials have already proved that strain-gauges can effectively qualify and quantify post-gel contraction [5, 9, 27]. Further, assessment of the restorative-tooth interface was performed on several sections assigned for microleakage experiments and scanning electron microscopy to determine whether strain magnitudes induced within both materials led to debonding at the tooth-restoration interface.

In the strain-gauge analysis part of the study, it was not surprising to observe volumetric expansion (tensile strains) for both materials at the outset of the experiments (0–15 s), as the intensity of the light that reaches the strain-gauge leads to thermal expansion of both the resin composite and the gauge. Accordingly, the magnitude of tensile strains was affected by the translucency and thermal diffusivity of the material [20, 28]. Upon initial expansion, both materials exhibited extremely different patterns of strain generation; a rapid conversion to compressive strains (shrinkage) was observed for Filtek P60, whereas high-rate increase in tensile strains (expansion) occured in Z250 specimens. Clearly, the similarity in chemical composition did not have any impact on initiation as well as the final strain magnitudes (volumetric shrinkage) of both restoratives. Because it was beyond the scope of this study to undertake a comprehensive evaluation of the chemical composition of the resin composites, it is impossible to determine the exact cause of this result. However, it is known that static and dynamic elastic moduli of elasticity are different and this may be one of the factors leading to differences in strain magnitudes [24]. In the present study, the final strain level was directly related to the polymerization velocity. There was a high correlation between polymerization velocity and magnitude of strains within restoratives at 40, 400, 1,000, 76,400, and 86,400 s (24 h), as higher polymerization velocities led

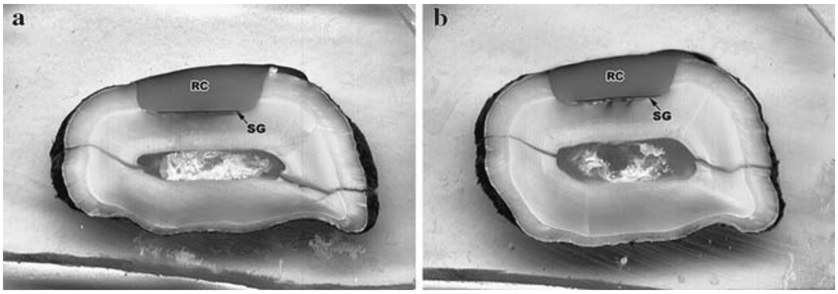


Fig. 4 Representative sections of Filtek P60 (**a**) and Filtek Z250 (**b**), showing absence of microleakage at the resin composite(RC)-tooth interface. SG = Strain gauge

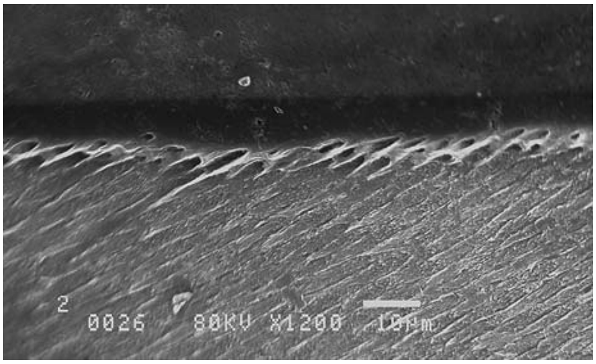


Fig. 5 SEM image at ×1200 magnification of the resin-dentin interface of a sample restored with Filtek P 60 and Single Bond. A thick hybrid layer may be observed. Note absence of bonding failure

to higher magnitude of strains. Indeed, it is well-known that the polymerization velocity of composites affects magnitude of internal stresses. In a comparative study, it was reported that autopolymerizing composites, which require more polymerization time (less polymerization velocity) than light-polymerized composites, were associated with fewer internal stresses within the composite [18, 20]. Hence, for light-polymerized resin composites, it can be estimated that the higher the polymerization velocity, the greater the total volumetric change (Filtek P60: 1773652 units versus Filtek Z250: 371856,8 units) will be and this change will presumably increase built-in strains at the interface.

After a rapid increase in strain magnitudes, the strains induced within Filtek Z250 reached a plateau after 50 s and gradually decreased with a steadily low rate. However, strains continued to increase rapidly within Filtek P60 for the first 100 s, which decreased gradually with a steadily low rate after water soaking. Importantly, the time-dependent strain generation within both materials did not exhibit clear signs of hygroscopic expansion. The nature of decrease in strain magnitudes, although resulting in increase of shrinkage for Filtek P60 and decrease in expansion for Filtek Z250, was rather like a sign of ‘‘diminish’’ in strain generation for Filtek P60 and a slight ‘‘strain relief’’ for Filtek Z250. Indeed, one should expect very low hygroscopic expansion in both restoratives, as the UDMA and BIS-EMA resin contents impart great hydrophobicity. Hence, after clinical application, the possibility of compensation in volumetric shrinkage by hygroscopic expansion is very low for these materials. Another critical issue is the impact of these drastic differences on the tooth restorative interface, particularly its effect on dynamic fatigue strength. Depending on our scanning microscopy analyses findings and evaluation of possible microleakage at the tooth-restorative interface, it seems that the bond at the interface is capable of withstanding polymerization contractive forces up to thousands of microstrains, and probably more than 15–20 MPa, unlike generally interpreted [29]. In essence, our methodology was unable to quantify actual strains at the interface, which can probably be measured only by individualized, microfocus computed tomography based, three-dimensional non-linear finite element stress analysis. However, in such models, there is also a high possibility of error, as the implementation of the dynamic behavior of the hybrid layer and dentin tubules filled with resin, including contact definition is an extremely hard task and needs several in vitro experiments for determination of the data which will be implemented into the numeric model. Therefore, strain-gauge analysis, although it quantified and qualified strains within the material, was descriptive on interface strains and suggested that the interface could withstand both contractive/expansive forces arising from polymerization shrinkage or expansion. Yet, scientifically consistent evidence from in vitro studies and epidemiological facts from human studies are scarce, and therefore, a battery of analyses are required to elucidate the maximum interface strain thresholds and the effects of resin composition on strain generation.

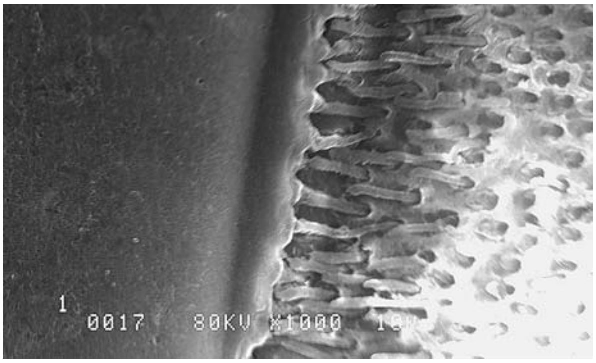


Fig. 6 SEM image at ×1000 magnification of the resin-dentin interface of a sample restored with Filtek Z250 and Single Bond. Regular, cylindrical resin tags and a thick hybrid layer are evident

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