Viscoelastic behavior of dental restorative composites during setting

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Abstract

Much attention has been directed toward developing dental restorative composites that generate less shrinkage stress during setting. The aim of this study was to explore the viscoelastic behavior of a new class of low-shrinkage dental restorative composites during setting. The setting behavior of an experimental oxirane composite was investigated by analyzing stress-strain data by means of 2-parametric mechanical models. The experimental data were obtained using a dynamic test method, in which the setting light-activated composite was continuously subjected to sinusoidal strain cycles. The material parameters and the model's predictive capacity were analyzed by means of validated modeling procedures.

The light-activated oxirane composite exhibited shrinkage delay and lower polymerization shrinkage strain and stresses than conventional light-activated composites. Due to noise in the stress data, the predictive ability of the Maxwell model was restricted to the elastic modulus development of the composite. The shrinkage stress development makes this composite a candidate for use in restorative dentistry. In determining their potential as restorative material evaluation tests will be necessary to establish whether the mechanical properties of oxirane composites are acceptable for dental use.
Introduction

BisGMA is often employed as the principal monomer in present day commercial dental restorative composites [1]. From the development of these dental monomers by Bowen [2] in 1962, polymerization shrinkage (7-14 vol%) of this conventional monomer system is still of great concern [3]. Composite shrinkage, in combination with an increasing stiffness, inevitably leads to mechanical stresses, which ultimately can cause enamel fractures, microleakage, and degradation of the restoration [4-7]. The advent of low-shrinking monomers with good mechanical strength would improve longevity of composite restorations, as they would lessen the demands on dentin-bonding agents and lead to reduced marginal leakage.

Figure 8.1 (a) Schematic representations of the polymerization of several monomers. Methacrylates are commonly used in dentistry, whereas the other monomers are under development. (b) Several oxiranes (cyclic three-membered ether) monomers [8].

In the past, many efforts have been made to develop new resin systems with reduced shrinkage. One approach focuses on preparing a new family of (multi)-methacrylates, which have higher molecular weights than the bisGMA based systems [9]. Another approach focuses on the synthesis of nonshrinking or expanding monomers. For this goal, one class of new monomers, termed oxybismethacrylates, was developed.
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Cationic (ringopening) polymerization:
- Blue light (400-500nm)
- Proton initiation
- Propagation
- Expansion
- Shrinkage

Free radical polymerization:
- Free radical photo initiator
- Radical initiation
- Propagation
- Shrinkage

Figure 8.2 Comparison between free radical polymerization and cationic polymerization [9].
Depending on polymerization condition, oxybismethacrylates exhibit cyclopolymerization (Fig. 8.1). In this polymerization process, cyclic structures are introduced into the polymer backbone [10]. Stansbury reported a 30 to 40% reduction in shrinkage upon the use of oxybismethacrylates compared with dimethacrylates commonly used in dentistry [11, 12].

Simultaneously, an another class of new monomers, termed ring opening monomers, was developed. Bailey reported a variety of bicyclic monomers that can undergo double ring opening with either no change in volume, or an actual expansion in volume, or an actual expansion [13]. The class of bicyclic monomers studied most extensively is the alicyclic spiro orthocarbonates (SOCs). An alicyclic SOC consists of four rings, two on each side of the spiro carbon (Fig. 8.1). The expansion of the SOC on polymerization is attributed to a double-ring opening of this spiro molecule; that is, two bonds are cleaved for each new bond formed. Stansbury and Bailey studied free radical polymerization of SOC s with vinyl functionality [14]. Although this monomer showed some reduction in polymerization shrinkage when compared with conventional dimethacrylate resins, their shrinkage was still substantial, because the shrinkage of vinyl polymerization was more than the expansion encountered by the SOC. An other research group has studied some SOC structures that are cationically light-initiated [15-16]. Cationic polymerization resulted in net expansion (3.5 vol%) of the resin system, due to the absence of the shrinkage contribution of the vinyl functionality present in the monomer. Up to this moment, the potential of these kinds of monomers as candidates resins for formulating dental composites has not been utilized.

Cationic ring opening polymerization continue to enjoy increasing interest. Beside low polymerization shrinkage, the use of cationic ring opening polymerizable monomers exhibit several advantages compared to the free radical polymerizable conventional dimethacrylate monomer system (Fig. 8.2). First, the cationic polymerization reaction is not inhibited by oxygen [17]. Furthermore, very high degree of monomer conversion can be achieved, because the protons (H+) are highly mobile compared to radicals. At this moment, the only drawback of the cationic polymerization reaction is that it is inhibited by basic materials and high humidity.

Recently, an experimental restorative composite based upon oxiranes has been developed [8]. The oxiranes are cyclic three-membered ether monomers that undergo cationic polymerization (Fig. 8.2). The initiation system was chosen so, that this condensable composite can be polymerized with conventional quartz tungsten halogen light source [18].
With special attention in the handling of this experimental composite, the inhibition of basic materials and high humidity can be avoided.

The aim of this study was to explore the viscoelastic behavior of this oxirane composite during setting. For this goal, dynamical stress-strain data on the setting composite were measured and analyzed with the Maxwell and Kelvin model by means of a validated modeling procedure.

Materials and methods

Experimental composite

The oxirane composite was prepared by 3M (Pluto, batch EXL546, exp 01/2002 3M). The composite was stored in plastic capsules, sealed with a foil pouch, at 6 °C. According to the manufacturer’s instructions, the composite was light cured for 60 s (Elipar Highlight, standard mode, ESPE) with a light intensity of 600 mW/cm² (radiometer, model 100, Demetron) at the light exit tip (Ø=8.95 cm). The composite was handled with a plastic spatula to avoid polymerization inhibition by the (basic) passive layer (Fe(OH)₂) on steel instruments [19].

Dynamic test: oscillatory deformation cycles

To elucidate the linear viscoelastic behavior by mechanical models, stress-strain data on the oxirane composite were obtained by an oscillatory sinusoidal strain test on an automated testing machine (ACTAIntense, ACTA). Since major features of this machine have been described in chapter 3 of this thesis, only refinements made in the specimen mounting device (Fig. 8.3) will be described here.

The glass plate was secured against the metal basement by two fixed toggles (213-U, De Sta Co, RS components) with a preset pressure of 700 N. The glass plate was temperature-controlled by a home-build temperature system. This system consist of 2 Peltier elements (L100, LC-electronics) with fans (Pentium II, LC-electronics) mounted on the backside of the metal basement (Fig. 8.3), and a thermostat placed, via a thermocouple, on top of the metal basement. The temperature of the specimen was monitored continously by a thermocouple (type K (NiCr-NiAl), d= 0,25 mm, Thermo-electra), as close as possible to the specimen.

The composite was placed on the glass plate and condensed with a disposable plastic spatula, after which the cross head was lowered until
the extensometer displayed the pre-adjusted distance (h=1.6 mm) between the upper steel rod (d=3.2 mm) and lower glass plate. The excess composite was then removed from the circumference of the cylindrical specimen. Bonding between the oxirane composite and the steel rod was achieved by applying the Silicoater procedure (5 minutes, Kulzer) to the sandblasted surface of the steel rod (Korox® 50 μm, 2 min/5 bar pressure, Bego). After that, an oxirane based adhesive resin

![Diagram of specimen mounting device and dynamic test protocol](image_url)

**Figure 8.3** (top) Specimen mounting device and (bottom) dynamic test protocol for the oxirane composite. ① Oscillatory deformation with amplitude 1.00±0.01 μm and frequency=0.1 Hz ② constant original specimen height (deformation=0 μm) ③ unload shrinkage load on specimen in prescribed time period of 200 seconds ④ constant load signal (0 N) ⑤ tensile loading (120 μm/min) until fracture.
(experimental adhesive EXL520, 3M) was brushed on the silanized surface to realize good bonding to Pluto. The adhesive was light-cured for 60 s (Elipar Highlight, standard mode, ESPE). For the same reason, the glass surface was lightly sandblasted (Korox® 50 μm, 10 s/5 bar pressure, Bego), primed (RelyX ceramic primer, 3M), and finally coated with a light-cured adhesive layer (experimental adhesive EXL520, 3M) [20].

In the dynamic test method, the steel rod performed an oscillating vertical sinusoidal deformation on the setting composite around its original height. The test method was programmed with the following protocol (Fig. 8.3). First, an oscillatory deformation was applied for approximately 100 minutes to the setting composite, followed by a time period of 300 seconds, wherein the original height of the specimen was kept constant. After the cross head moved towards the composite in a prescribed time period of 200 seconds to relief the shrinkage load, the load signal was maintained zero for 300 seconds. Two hours after the start of the experiment, the oxirane composite was subjected to tensile loading until fracture. The measurements on the oxirane composite (n=3) were performed at C-factor 1.0 (h=1.6 mm) and 3.85 (h=0.65) at room temperature (23±0.3 °C).

![Figure 8.4 Dynamic test protocol for measuring elastic shrinkage strain of oxirane composite.](image-url)
Dynamic test: pulse load cycles

To reveal the elastic shrinkage strain development of the oxirane composite during setting, a second dynamic test was performed. In this test, the cross head kept the original height of the composite constant during setting. Periodically, the cross head cycled down and up to unload the composite and restore original composite height respectively (Fig. 8.4).

The period of the cycle action was approximately 20 s, whereas the period between cycling was 300 s. The measurement was performed once at two configurations of the oxirane composite (C=1.0 and C=3.85) at room temperature. All dynamical tests were performed with ACTA application software (version 3.14)

Shrinkage measurement

During the dynamic test, the axial shrinkage strain of the specimen was not measured, because the steel rod performed oscillating deformation cycles around the original height of the specimen. The axial shrinkage strain of the specimen under bonded conditions is of interest, because calculating the material parameters from the measured stress data, the strain caused by axial shrinkage must be taken into account. Since the test system is not capable in determining the axial shrinkage of composites accurately [21], the bonded axial shrinkage strain (ε_{axial}) for the oxirane composite at the chosen configuration was derived from the volumetric shrinkage strain (ε_{vol}) by the conversion factors in Table 3.1. In this approach, we assumed that the shrinkage behavior of the bonded oxirane composite was similar to conventional resin composites, as measured by Feilzer et al. [22]. The (free) volumetric shrinkage measurements (n=3) were performed with a mercury dilatometer at 23±0.1 °C, using the procedure described by De Gee et al. [23]

Light microscopy

The fractured surface of the composites was examined with an Olympus PM-10AK light microscope.

Stress-strain analysis

The data obtained from the oscillatory deformation measurement
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consisted of an array of load and displacement values for a large number of points in time. The normal stress ($\sigma$) and strain ($\varepsilon$) were calculated from the load and displacement values by Equation (8.1) and (8.2) respectively:

$$\sigma = \frac{F}{A}$$

(8.1)

$$\varepsilon_{\text{axial}} = \frac{\Delta L}{L_0}$$

(8.2)

in which $A$ is the cross-sectional area of the cylindrical specimen ($m^2$), $F$ the recorded load response of the specimen (N), $\Delta L$ is the displacement recorded by the LVDT transducers (m), $L_0$ the height of the specimen before setting (m). The shrinkage stress response was isolated from experimental stress data via the standard Fast Fourier Transform (FFT) smoothing procedure in Origin (version 5.0, microcal).

The data obtained from the volume shrinkage measurement were averaged, and the axial shrinkage strain development of the oxirane composite at C-factor 1.0 and 3.85 was calculated by multiplying the mean volumetric shrinkage strain with the conversion factor in Table 3.1. For the identification of the material parameters, the functional expression of the obtained axial shrinkage strain curves was calculated by a cubic spline fit, and was added to the oscillatory strain for all the points in time of the dynamic test measurement.

**Parameter identification**

The Maxwell and Kelvin model were investigated in one dimension only, because the experimental stress-strain data were monitored in only one direction. The models are described in detail in chapter 4 of this thesis. In the setting period of 5 to 100 minutes after light-initiation, time intervals of approximately 10 seconds were isolated from the experimental data, and applied to the parameter identification procedure. As the shrinkage strain of the composite in the isolated interval behaves linearly with time, the total strain in the interval can be described analytically:

$$\varepsilon(t) = \varepsilon(t_0) + At + B \sin(\omega t)$$

(8.3)

in which $\varepsilon(t_0)$ is the strain at begin interval, $A$ is the slope of the shrinkage strain (1/s), $B$ the amplitude, and $\omega$ the angular frequency (rad/s) of the applied oscillatory strain.
Since the functional form of the strain was known, the differential equation for the Maxwell was solved analytically (appendix A), which in every case yielded the stress as a function of strain and the unknown material parameters. The initial stress ($\sigma(t_0)$) was obtained from experimental stress data. The parameter identification procedure is described in detail in chapter 4 of this thesis.

Model evaluation

To evaluate the appropriateness of the two mechanical models under shrinkage strain conditions, the measured axial shrinkage stress development of the oxirane composite was compared with the model response. In chapter 4 of this thesis, a shrinkage stress procedure to estimate the model response on basis of the input of the axial shrinkage strain and the calculated material parameters, is described.

A second evaluation procedure was performed with the Maxwell model only. In this procedure, the elastic shrinkage strain ($\varepsilon_{\text{spring}}$) and the viscous shrinkage strain ($\varepsilon_{\text{dashpot}}$) development of the composite was calculated from the measured shrinkage stress data and Maxwell’s Young’s modulus ($E$) and viscosity ($\eta$) respectively (Fig. 8.5). This approach is valid, as the architecture of the Maxwell model - spring and dashpot working in series - provides the following coupling conditions for the stress and strain:

$$\sigma_{\text{shrinkage}} = \sigma_{\text{spring}} = \sigma_{\text{dashpot}} \quad (8.4)$$

$$\varepsilon_{\text{shrinkage}} = \varepsilon_{\text{spring}} + \varepsilon_{\text{dashpot}} \quad (8.5)$$

![Figure 8.5](image-url) (left) Elastic shrinkage strain and (right) viscous shrinkage strain calculation from experimental shrinkage stress data and Maxwell’s parameters Young’s modulus ($E$) and viscosity ($\eta$), as calculated with the parameter identification procedure.
To be able to perform calculations with experimental shrinkage stress data, a cubic spline fit was applied to the identified Young’s modulus and viscosity values. With spline interpolation, the parameter values were chosen at the same points in time as in the case for the experimental shrinkage stress values. The parameter identification procedure and evaluation of the models were performed with the software Matlab (version 5.3, Mathworks) on a desktop computer (Windows® 98 platform).

Results and discussion

Tensile loading

Figures 8.7a and 8.7b show the applied strain and stress response of the oxirane composite during the initial 10 minutes of the setting process at room temperature. None of the specimens fractured spontaneously prior to tensile loading. During tensile loading, the fracture always occurred cohesively, was flat shaped and perpendicular to the direction of loading, and was situated near the glass plate side (Fig 8.6b). The result of the stress-strain behavior of the composite during tensile loading (Fig. 8.7f) agrees with the microscopic observed fracture pattern of the composite. Both results indicate a brittle failure of the composite without any significant plastic (necking) deformation.

Figure 8.6 Light microscope pictures (magnification x10) of the fracture surface of the oxirane composite after dynamic testing at (a) steel rod side and (b) glass plate side. Dash lines indicate circumference of specimen. Black arrows indicate empty spherical holes in the composite. Notice the flaw pattern in the composite and the damage at the jacket side of the composite.
Stress-strain data

The stress-strain curves of the composite show hysteresis, which indicates energy loss of the composite during cyclic deformation. This still seems to be the case for the composite at 100 minute setting, the

![Stress-strain data graph](image)

**Figure 8.7** (a) Strain and (b) stress data of the oxirane composite collected with the dynamic test method. For clarity, only the data for the first 10 minutes of the setting reaction is shown. The thick black solid line in (b) represents shrinkage stress, which was obtained by FFT smoothing of the experimental stress data. (c) Axial shrinkage for the oxirane composite, as calculated from mean volumetric shrinkage data. Error bars indicate the relative standard error in the calculated mean (n=3). For the parameter identification procedure, the total strain curve was constructed by a linear combination of (a) and (c), resulting in (e). (d) The signal of the light irradiation process is superimposed on the glass temperature, measured as close as possible to the specimen. (f) Stress-strain behavior of the oxirane composite of the last three oscillatory cycles and tensile loading.
point at which the last three cycles in Figure 8.7f were recorded. This energy dissipation is due to viscoelastic behavior [21].

Light-activated dental resin composites exhibit shrinkage strain kinetics and magnitudes that constitute a major challenge to polymer chemists and dental scientists [24]. While the principal research target remains that of reducing the final shrinkage value, it is also a benefit to delay the onset of shrinkage strain. This ‘soft-start’ shrinkage may be obtained by varying light intensity on the composite, either by reducing the output of the curing light or by increasing the distance between the light exit tip and the composite [25-27], or using fixed light intensity via suitable changes in the formulation chemistry, as with this oxirane composite (Fig. 8.8a).

The delay of approximately 20 seconds in the early shrinkage strain of the oxirane composite may be designated as intrinsic ‘soft-start’ phenomenon. So far, only one commercially available light-activated (multi-acrylate based) composite is known in dental literature (Solitaire) which exhibit intrinsic ‘soft-start’ shrinkage behavior [30].

To put the shrinkage behavior in perspective to conventional dimethacrylate composites used in dentistry, the shrinkage development of the oxirane composite in Figure 8.8a is compared with the shrinkage results of two commercially available resin composites measured in previous studies [28, 29]. As shrinkage is associated with polymerization of the monomers, the shrinkage rate curves in Figure 8.8b are good estimates for the polymerization rate of the composites. Despite the shorter time of light irradiation, the conventional dimethacrylate composite undergo much faster polymerization than the oxirane composite. This is in agreement with literature [17]. The rapid decrease in polymerization rate indicates that the conventional composite is “freezed” during the light irradiation process. Due to the rapid setting process, the termination stage (auto-acceleration) and propagation stage (autodeceleration) in the free radical polymerization reaction becomes diffusion controlled. The increasing polymerization rate of the oxirane composite indicates that the weak structure of the composite causes unobstructed propagation of the growing (cationic) polymer. As a result, the shrinkage stress development in the oxirane composite proceeds very slow, almost linear, with setting time (Fig. 8.8c). Even after the light irradiation process, the structure of the oxirane composite is still weak, as oscillatory deformation on the composite did not cause any measurable deflection of the load cell (Fig. 8.7b). This might suggest that the practioner can still sculpt, adapt, and contour the material after light irradiation.

Beyond the potential benefits of the light-activated oxirane composite
Figure 8.8 (a) Volumetric shrinkage, (b) volumetric shrinkage rate, and (c) axial shrinkage stress development for the oxirane composite (Pluto - \( C=1.0/23 \) °C). The results obtained by Dauvillier et al. [28] for the light-activated composite (Z100 - \( C=1.0/23 \) °C) and by Dauvillier et al. [29] for the chemically activated composite (Clearfil F2 - \( C=0.5/23 \) °C) are also incorporated. Z100 and Pluto were light activated (600 mW/cm²) for 40 and 60 seconds respectively. Clearfil F2 was hand-mixed (1:1 w/w). Note y-as break at (c) 0.10-0.12 MPa and (b) 0.025-0.05 %/s and x-as break at (a) & (c) 200-300 s.
that it can be cure-on-command and undergoes a polymerization rate in the range of chemically activated dimethacrylate composites (Fig. 8.8b), the oxirane composite also undergoes approximately 45% less shrinkage relatively to conventional resin composites after one hour setting. On future studies, it is advisable to perform water uptake experiments after setting on the oxirane composite, because water sorption might frustrate the high expectations of this low shrinking material.

Parameter identification

In this study, the Maxwell and Kelvin model were investigated. The slow and low shrinkage stress-strain development of the oxirane composite may be good properties for the material, but it restricted our research in mechanical modeling of the early setting period heavily. Up to 3 minutes setting, we were not able to calculate reliable material parameters, due to the high level of noise (SNR<2) carried by the stress data [29].

The computed Kelvin and Maxwell parameters vary smoothly with setting time (Table 8.1). A close look at the isolated stress curves at 609 and 1209 seconds setting (Fig. 8.9a) reveals that the Maxwell model curve is almost an exact copy of the Kelvin model curve. An explanation for this observation is that in these time intervals the composite does not undergo shrinkage. As a result, the experimental stress curves in Figure 8.9a are generated by the applied sinusoidal strain only.

Since sinusoidal stress data hide two independent variables, namely an amplitude and a phase angle, both 2-parametric models describe the experimental stress curves on a similar basis. As a consequence, the graphical results of the parameter identification procedure (Fig. 8.9a) do not provide information whether the viscoelastic behavior of the oxirane composite is liquid-like (Maxwell) or solid-like (Kelvin).

Model evaluation

The evaluation procedure reveals that both the Kelvin and Maxwell model failed to predict the shrinkage stress for the oxirane composite (Fig. 8.9b). For both C-factors, the same two extremes were visible: the Kelvin model predicted the shrinkage stresses too high, while the Maxwell model did not predicted a shrinkage stress development at all. The incapacity of the Maxwell model to predict the viscoelastic behavior of the oxirane composite is not in accordance with the obser-
Table 8.1 Material parameters for several cycles during one measurement of the oxirane composite (C-factor=1.0) at room temperature with standard deviation in parenthesis. Material parameters: $E$=Young’s modulus, $\eta$=viscosity, and $\delta$=quantitative measure of the difference between experimental and model stress.

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<td>0.74 (&lt;0.01)</td>
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![Figure 8.9](image-url)

**Figure 8.9** (a) Parameter identification results of the (—) experimental stress intervals of the oxirane composite (C-factor=1) at (top) setting time=159 s, (middle) setting time=609 s, and (bottom) setting time=1209 s, computed with the (▲) Maxwell model and the (■) Kelvin model. Dashed line indicates shrinkage stress. (b) Axial shrinkage stress development of the oxirane composite during one hour setting: (—) experimental stress data, (■) Kelvin model, and (▲) Maxwell model. Error bars indicate the relative standard error in the calculated mean (n=3).
vation that more than 50% of the shrinkage strain was measured without generating shrinkage stress in the oxirane composite. Shrinkage without stress development is only feasible when the viscous flow behavior of the composite predominates over the elastic behavior. The reason why the Maxwell model fails to predict the viscoelastic behavior of the oxirane composite will be described in the next paragraph.

Figure 8.10a shows the elastic shrinkage strain curve of the composite, as predicted by the Young's modulus, and as measured in the second dynamic test method. With exception of the initial stage of setting, where to the noise in the shrinkage stress is relatively high, the Maxwell model is capable to predict the stiffness development of the oxirane composite on a good basis.

However, this does not count for the viscous part of the model. The viscous part of the axial (one dimensional) shrinkage strain, as predicted by Maxwell's viscosity, is many times higher than the composite undergo on a volumetric (three dimensional) basis (Fig 8.10b). It would be a misconception to believe that the composite can undergo this high level of viscous flow, because under bonded conditions, the axial shrinkage strain, which is built up from an elastic and a viscous part, is limited to

![Figure 8.10](image)

Figure 8.10 (a) Elastic shrinkage strain predicted by Equation (8.4) and measured with the pulse load cycles for the oxirane composite at (top) C-factor=1.0 and (bottom) C-factor=3.85 at room temperature. (b) Viscous shrinkage strain predicted by Equation (8.5) compared with volumetric shrinkage of the oxirane composite at (top) C-factor=1.0 and (bottom) C-factor=3.85 at room temperature. Error bars indicate the relative standard error in the calculated mean (n=3).
a maximum near the volumetric value [22]. Apparently, Maxwell predicts the viscosity value too low; i.e., predict too much viscous flow capacity for the composite under the chosen test condition. This explains why the model predicted a shrinkage stress around the zero stress level (Fig. 8.9b). A previous study showed that in presence of a high level of noise in experimental stress data influenced the viscosity identification negatively, while the identified Young's modulus value still approximated the exact value [29]. In case of viscosity identification, better results can be obtained by a different strategy: 1) measure the axial shrinkage strain under bonded conditions and 2) subtract the elastic shrinkage strain curve, as predicted by Equation 8.4 from the data obtained with 1). This will reveal, directly, the viscous part of the shrinkage strain curve, and indirectly, with the aid of the shrinkage stress curve, the viscosity development of the composite with setting time.

Conclusions

The experimental oxirane composite displays attractive mechanical properties for application as a restorative material. The light-activated oxirane composite exhibits an intrinsic 'soft start', undergoes 45 % less shrinkage strain than commercially available light-activated resin composites, and generates very low shrinkage stresses. Future studies will be necessary to reveal its potential for use in restorative dentistry. Unlike experimental stress-strain data observation, the Maxwell model cannot predict the viscoelastic behavior of the oxirane composite during setting. The high level of the noise carried by the low stress signal has a decisive influence on the value of the viscosity parameter associated with this model. On the other hand, the elastic part of the Maxwell model does predict the stiffness development of the composite during setting with a good degree of accuracy.

References

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ites in vivo, Dent Mater 7:44-49.


21. See chapter 3 of this thesis.


continuous recording of volumetric polymerization shrinkage of composite restorative materials, J Dent 9:36-42.


