Digital Holographic Interferometry - A New Method for Measuring Polymerization Shrinkage of Composite Materials

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Summary

Polymerization of composite filling is considered to be an important factor in achieving longevity of the restorative treatment. Quality of the polymerization influences physico-mechanical characteristics, color stability, volumetric changes of restoration and biocompatibility of the material itself. Polymerization shrinkage is an unavoidable consequence of setting of the composite material. So far, it has been measured by several different methods. Digital holographic interferometry, a method described in this study, is the only procedure that enables direct monitoring of the polymerization shrinkage for every second of polymerization of the composite material. Values of the polymerization shrinkage obtained by this method correspond with the values obtained by other methods.

Key words: *composite materials, polymerization shrinkage, digital holographic interferometry, program for analysis of interferograms.*

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Introduction

Polymerization shrinkage or resin contraction is a chemical reaction characteristic of resins. It is the dimensional instability of composite materials caused by polymerization of the organic matrix. According to Feilzer (1), the total amount of shrinkage for non-filled resins amounts to 7.9%, and for filled 2.5-5.5%, or according to Seguera 1.67-5.65% (2).

Several factors participate in polymerization shrinkage: cavity shaping and preparation, composition of the composite material, method of placing the material in the cavity and light source.

Because polymerization shrinkage influences the quality and durability of composite restoration, many different techniques are used in an attempt to compensate for, or reduce, the shrinkage. For example, use of dental adhesives and hybridization of dentin, layered placing of the material in the cavity, introduction of new monomer systems and choice of the optimal light source.

Value of the polymerization shrinkage can be measured directly, by measuring the dimensional changes in the material created during the polymerization process or indirectly, by studying microleakage.

Direct methods encompass: dilatometric method (mercury or water dilatometer), linometer, comparison of the differences in specific weight and density of the material, "strain-gauge" method and others (3-5).

Indirect methods encompass: determining depth of bacterial or liquid penetration, air pressure measurements, and use of radioisotopes, electromechanical studies and termic and mechanical termo-cycling (3, 4).

In this study, we implemented the newest direct method for measuring polymerization contraction, digital holographic interferometry. Interferograms are created by superimposing two or more wavefronts of the coherent light. Independent of the type of interferometer, the obtained record is a two dimensional light distribution of light and dark stripes that exhibit some relative change. It is possible to explain the change by analyzing stripe distribution (density and orientation) with the given wavefront starting conditions (light wavelength, divergence) and knowledge of the nature of the expected change (translation, rotation, deformation, diffraction index).

Wave interference is a phenomenon created at intersection points of the coherent light waves. It expresses modification of the intensity crated by superposition of wave intersection points and is manifested as the appearance of light and dark stripes on the screen. Stripes are a result of both constructive and destructive interference. Constructive interference is created when waves are in phase, and destructive when the wave phase is shifted for the π value, so that the hill of one wave coincides with the valley of another and they cancel each other out. Visibility of interference stripes depends on the following parameters: light coherence, system stability, differences in the optical pathways, etc. It is necessary to coordinate all the parameters in order to achieve more exact stripe distribution.

Classic holographic interferometry assumes recording the hologram (wet procedure) and then observing the deformity in real time or using some other technique. However, it is possible to directly record a holographic image using the CCD camera and then to store it in computer memory. Because of the small spatial resolution of the CCD camera there are limitations concerning the angle between the beams and openings in the system. Image stored in this fashion contains the information analogue to the classical holographic interferometry method. Digital approach to the interferogram analysis has many advantages because of the possibilities of the computer program. For instance, observation of the shifting of the sample can be simply automatized. Finally, all parameters of the shift can be obtained quickly and objectively (6-8).

The purpose of the study was to present a holographic interferometry and computer program for analyzing interferograms as the only method to date that allows observation of polymerization shrinkage for every second of composite material polymerization.

Materials and methods

Figure 1 is a schematic representation of the optical device. Divisor (DS1) splits the laser beam into two parts. One part, guided by mirrors (Z) and colimation by space divisor (PS1) and lens (L) reflects on the cuneiform divisor (DS2) and falls directly on the CCD camera. The second part, colimated by space divisor (PS2) and lens (L2) reflects from the mirror (Z) and falls on the sample holder mirror. The sample holder is shown in Figure 2b. It enables the beam of blue light (π = 468nm) to photo polymerize the sample from underneath, and simultaneously, using the laser red light (π = 633 nm) measures the shrinkage or stretching of the sample.

Samples 0.65 mm thick were prepared for measuring polymerization shrinkage. Each sample was first placed between two foils, each 0.75 mm thick, and then placed in an inox ring 1 mm thick. Next, the ring with the sample was compressed between two round inox plates to achieve uniform thickness of 1mm for all samples with foils. Samples of the composite material Spectrum TPH (Dentsply GmbH, Konstanz, Germany) were polymerized using low intensity program, 400 mW/cm^2, Astralis 7 halogen lamp (Vivadent, Schaan, Liechtenstein) and Lux-o-Max diode device (Akeda Dental, Lystrup, Denmark) which for the first 10 seconds emits light of intensity 50 mW/cm^2 and for the following 30 seconds light of intensity 150 mW/cm^2. Ten consecutive samples were made and the val-

ues obtained represent linear polymerization shrinkage.

The sample was placed on the glass platelet that lies on the movable part of the sample holder above the opening for the polymerization of the samples. On the sample (covered with foil) removed from the margin glass platelet was placed. The platelet was from the upper side steamed with aluminum layer (mirror) and served to direct the laser light beam. (Figure 2a and b).

Two laser light beams interfere, one that originates from the mirror placed on the surface of the sample and other that comes from the fixed mirror. The CCD camera records the image and the computer processes the information.

Interference intensity pattern can be described by the following formula:

$$I_p(x,y) = I_0(x,y) + I_1(x,y)\cos\varphi(x,y)$$

Where $I_0(x,y)$ represents background intensity, $I_1(x,y)$ is amplitude and $\varphi(x,y)$ a relative phase between the two interfering waves.

After the deformity (or the shift) we have

$$I_k(x,y) = I_0(x,y) + I_1(x,y)\cos\left[\varphi(x,y) + \Delta\varphi(x,y)\right]$$

Where $\Delta\varphi$ represents change in phase created by the sample deformation. The assumption in the upper equation is that there is a slight change that does not influence the background intensity or the amplitude of the interferometric stripes.

Change of phase and the real threedimensional vector of the shift are connected by the equation:

$$\Delta\varphi(x,y) = \frac{2\pi}{\lambda}\left(\vec{k}_r - \vec{k}_p\right)\cdot\vec{d}(x,y)$$

where λ stands for laser light wavelength, $\vec{k}_r$ and $\vec{k}_p$ are vectors representing lighting and observation and $\vec{d}$ is a shift vector. According to the figure, $\vec{k}_r$ and $\vec{k}_p$ are almost collinear so the following equation applies:

$$\Delta\varphi(x,y) = 2d\frac{2\pi}{\lambda}$$

from this follows:

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

maximum (light stripe) and for

$$d = (2n-1)\frac{\lambda}{2}$$

minimum (dark stripe).

In upper equations n stand for ordinal number of the stripe.

Shift d turns the mirror that lies on the sample, which results in an increase in the stripe density on the CCD camera. This turning can be compensated by using the micro screw with precise reading (2 μm). If the final shift (linear shrinkage) of the sample is labeled with x, then follows:

$$d = 0{,}375x$$

where 0.375 is a factor of the sample holder transfer.

Therefore, the micrometer screw (Figure 3) is used to determine the final linear shrinkage of the sample, and analysis pf the interferometric image shows the behavior of the sample thickness from the beginning to the final state.

Program for processing the interferogram

Recorded interferograms are placed in files containing about 70 images, compressed in AVI database using the available compression algorithms. Images are recorded in 1 second intervals and start of the measuring coincides with the first image.

Selected database is read and decompressed into specific interferograms, which are then ready for configuration. First step of image formation is the normalization of the level of interferogram range. Images are recorded monochromatically with vertical resolution of 8 bites per pixel. Since most images do not fall into the interval from level 0 for black to level 255 for white, the image has to be standardized to fall into the desired value interval, from 0 to 255 and also to exclude possible rise of the entire image from the background level.

The image processed in this way needs to be filtered to remove any unwanted interference disturbances and to emphasize useful signal. Filtration is performed with multiple use of low permeable Gauss matrix filter.

This filter should be used for 15 consecutive times, which gives a good result. After filtration, the interferogram normalizes again to return the maximum range of data from 0 to 255. After normalization, filtration and repeated normalization interferogram is ready for stripe counting.

Counting of interferometric stripes is performed horizontally, from left to right. At the beginning, it is necessary to determine the center of the interference circles from which the counting starts.

On Figure 4 the histogram represents the chosen row signal amplitude in black. Red vertical line represents the chosen horizontal position from which the counting to the right starts. Counting of the stripes is performed in such a way to count every crossing of histogram line over the red horizontal line representing low permeability filtered mean value of the peaks and hills of the histogram wave.

The histogram must show even and smooth waves. If the waves are uneven, it is necessary to increase the number of Gauss filter filtrations. In the case that the amplitude is too low with increase in frequency, it is necessary to decrease the number of Gauss filter filtrations. After correct determination of the center, the counting of the crossings between the curve and the mean value is noted and stored in the Excel table.

Results of the polymerization shrinkage measurement can be read using three different methods. The first method is by looking at the interferometric image on the computer screen as a film, or a sequence of 40 images, each one corresponding to 1 second of illumination.

The second method of measuring polymerization shrinkage is by recording the difference in the shift of the micrometer screw at the start and at the end of the measuring.

The third method for obtaining the linear polymerization shrinkage values is through cited equations and programs for analysis of the interferograms.

Results

Figure 5a represents polymerization shrinkage of the Spectrum TPH composite material during polymerization using “low intensity” mode of Astralis 7 halogen lamp, and on Figure 5b polymerization shrinkage created by polymerization with Lux-o-Max device. Figures 6a and 6b represent interferometric image at the start of polymerization (central circle in the center of the image) and its shift, or shrinkage of the composite material at the 10th, 20th and 40th second of polymerization.

The first figure is identical for both curing units. The polymerization shrinkage in the 10th second of polymerization process for halogen curing unit is represented in Figure 6a where the mimimal movement of the central fringe to the right or so-called “expansion in the beginning” of the illumination process is recorded. On the other hand, in the case of curing a composite resin sample with LED curing unit, the central fringe movement to the right position is more visible (Figure 6b). The light intensity of the halogen curing unit is higher and therefore expansion occurs in the initial few seconds of illumination. As a result there is a minor movement in the 10th second, which represents a shift of the central fringe to the left and the beginning of the shrinkage process.

Table 1 contains mean values of conducted measurements read by micrometer screw (μm) and calculated with the program for analysis of interferograms (%).

In the case of polymerization shrinkage values obtained by micrometer screw, it is possible to read only the final amount of shrinkage, so the results are recorded after 40 seconds of polymerization. During result analysis with the computer, program values are read in % for the 10th, 20th and 40th second.

Discussion

Setting or polymerization of the composite materials is always connected with shrinkage because of short covalent bonds forming between monomers. This usually results in microcracks between the composite filling and the cavity walls with consequen-

tial microleakage and possible pulp irritation, thermal sensitivity and secondary caries. Therefore, polymerization shrinkage is one of the main factors that determine clinical value and longevity of the composite filling (3, 9).

Conventional methods for measuring the polymerization shrinkage include water and mercury dilatometer that measures total polymerization shrinkage during illumination of the composite materials and the "strain gauge/transducter"method that measures polymerization shrinkage after the gel-point of the polymerization process (10). A method that encompasses these two methods is the so-called "bonden-disk"method used for determining "approximate"value of the polymerization shrinkage (11). In this method a disc (7-9 mm thick) of restorative material is placed on the glass plate (approx. 1 mm thick) and the bond between the sample (restorative material) and substrate (glass plate) gives the approximate result of the volumetric shrinkage towards one axes (towards the center) (11).

Each of these techniques has its advantages as well as shortcomings. Mercury dilatometer is potentially harmful and very sensitive to thermal changes (12).

The "water dilatometer"method also depends on temperature and results can be influenced by water absorption to restorative material (13).

The "buoyancy method"is insensitive to temperature change but very sensitive to air bubbles absorbed on the surface of the restorative material (9).

Techniques for measuring the linear polymerization shrinkage such as the use of linometer and "contact displacement conductor" method are less sensitive to outside factors, but a potential mistake can occur under the influence of the gravitational load of the measuring system (14).

One of the often described methods for measuring volumetric polymerization shrinkage that gives the total value of shrinkage is the "picnometer" method, that is not affected by temperature change and is based on measuring resin volume before and after polymerization (9).

Results of polymerization shrinkage obtained by linear or volumetric methods coincide only in the case of so-called "homogenous shrinkage", or when restorative material shrinks uniformly along all three dimensions during illumination with a polymerization device (11).

It is well known that polymerization shrinkage depends on multiple factors, the most important being intensity of the polymerization device, composite resin composition, time of exposure and temperature (either because of the light source or chemical reactions inside the material that take place during the polymerization process) (15-17). Polymerization process can be divided into two phases: pre-gel and post-gel phase. The gel-point is the moment of "no return", or setting of the material. Polymerization shrinkage appears in boththe pre- and post-gel phase. Shrinkage that appears in the pre-gel phase can be compensated by "flowing" of the material (18).

Digital holographic interpherometry represents a relatively simple method for measuring polymerization shrinkage of composite materials. Furthermore, it enables double control of the amount of shrinkage: by direct reading of the polymerization shrinkage value on the micrometer as a result of movement of the micrometer screw before and after the end of polymerization and by the subsequent computation of the number of fringes with not only a figurative but also a numeric value for the shrinkage in every second of the illumination. All the methods mentioned provide the final amount of shrinkage but do not show the development of shrinkage during the illumination process like digital holographic interpherometry can. This is precisely the reason why this method is very significant for determining the quality of various polymerization devices (different intensities or polymerization modes) and composite materials. Therefore, to show the polymerization development shrinkage devices were chosen, one based on the cold light source of low intensity (LED device Lux-o-Max) and halogen devices of higher intensity (Astralis 7). The initial phase of the polymerization process is visible on the interpherometric image as a negative shift or expansion (-0,1±0,29% for Lux-o-Max) or shift to the right, while polymerization shrinkage is visible as a shift to the left (Figure 5a and 5b, 6a and 6b). The initial expansion was also recorded during illumination by the low intensity mode of the Astralis 7 halogen device, although because of uniform light

intensity (continuous illumination of 400 mW/cm^2) it was only observed during the initial five seconds (Figure 5a, 6a). Prolonged expansion observed for diode device Lux-o-Max was most probably because of lower initial light intensity (first 10 seconds 50 mW/cm^2) (Figure 5b, 6b). The total shrinkage after 40 seconds of illumination of the composite material sample is higher for halogen than diode device, as shown in Table 1. This presentation of the development of polymerization shrinkage was not possible in any of the previously mentioned or contemporary methods described for measuring polymerization shrinkage in professional literature. The results of polymerization shrinkage obtained in this study are in accordance with the values obtained by other authors: Meirs et al recorded values for linear polymerization shrinkage ranging from 0.8 to 1% (19); Goldman (20), Watts et al (21), Feilzer et al (22), and deGee et al (14) all obtained values from 052 to 0.99% linear shrinkage.

Conclusion

The digital holographic interferometry method enables direct reading of the total composite resin shrinkage immediately after the end of polymerization as a result of change in shift or change in volume before and after the sample polymerization on the one hand, while on the other hand it enables figurative monitoring the process on the computer screen during every second of the duration of the polymerization process. Furthermore, this method enables double control of the total shrinkage: value of shrinkage determined by the micrometer screw corresponds with the value calculated from the change in number of fringes after 40 seconds of illumination of the composite material sample by the polymerization device.