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Strain determination of self-adhesive resin cement using 3D Digital Image Correlation Method

Утврђивање оптерећења самовезујућег композитног цемента применом методе 3Д корелације дигиталних слика

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SUMMARY

Introduction/Objective In an attempt to simplify dental procedures, a new group of resin cements, self-adhesive resin cements (SARCs), have been introduced. Performances of SARCs can widely vary. One of the main reasons of adhesion failure is polymerization shrinkage.

The aim of this study was to determine, evaluate and measure strain field of self-adhesive dual cure resin cement during polymerization in self-cure mode using 3D Digital Image Correlation (DIC) method.

Methods The self-adhesive cement Maxcem Elite (Kerr, Orange, CA, USA) was tested in five cylindrical samples (5 mm in diameter and 2 mm thickness) prepared by filling plastic ring-type molds. Digital images were recorded immediately after sample preparation.

Results Non-uniform strain distribution was found in resin cement with higher strain values along the periphery (up to 15 %) and lower strain values in central parts (around 4 %) of each sample.

Conclusion It can be concluded that DIC is powerful tool for full-field strain measurement in material characterization.

Keywords: self-adhesive cement; digital Image Correlation; strain; polymerization shrinkage; self-cure mode

САЖЕТАК

Увод/Циљ У настојању да се поједноставе стоматолошке процедуре уведене су нове групе композитних цемената – самоадхезивни, двојновезујући композитни цементи. Својства ових цемената могу да варирају у великој мери. Један од главних разлога адхезионих мана је полимеризацијска контракција.

Циљ овог рада је да се одреди и измери деформационо поље самоадхезивног двојновезујућег композитног цемента током хемијске полимеризације користећи експерименталну технику – методу 3Д корелације дигиталних слика.

Методе Самоадхезивни композитни цемент, *Maxcem Elite (Kerr, Orange*, САД) испитан је на пет узорака пречника 5 мм, дебљине 2 мм, који су припремљени пуњењем пластичних калупа прстенастог облика. Дигиталне слике су забележене непосредно после припреме узорака.

Резултати Неуниформно деформационо поље је показало веће вредности деформација на периферији узорака (до 15%) и мање вредности деформација у централним деловима узорака (око 4 %).

Закључак Може се закључити да је 3Д корелација дигиталних слика прецизна и поуздана метода за мерење деформационих поља у области карактеризације материјала.

Кључне речи: самоадхезивни композитни цемент; метода 3Д корелације дигиталних слика; деформација; полимеризацијска контракција

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INTRODUCTION

Recently, dual-cured resin cements were being developed for dental application due to their ability to be self-cured under indirect esthetic polymeric or ceramic restorations [1, 2]. Compared to conventional luting agents, current resin cements can achieve better marginal seal, show retentive capability and possess adequate physical and mechanical properties, such as increased fracture resistance of overlying restorations along with an optimal esthetic result [3].

Self-adhesive dual-cured resin cements become commonly used in dentistry for bonding the indirect restorations [4]. They have been invented to simplify the resin bonding process and minimize the steps as well as the time consumed during the bonding procedures in dental practice. Their advantage is being dual-cured and easy to use with no need for dental tissues pretreatment. These cements are manufactured on a new technology of monomers, fillers and initiators.

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According to manufacturers, the adhesive properties of self-adhesive resin cements (SARCs) include acidic and hydrophilic monomers in their composition, which simultaneously demineralize and infiltrate enamel and dentin, resulting a strong bonding [5]. The organic matrix of some of them is based on a newly developed multifunctional phosphoric-acid methacrylate system (phosphates and phosphonates groups) [6]. Also acidic monomer groups allow adhesion to the tooth surface through micromechanical retention [7]. Furthermore, these acidic monomers are claimed to interact chemically with the basic inorganic fillers of the material, leading to an additional acid–base setting reaction, apart from the free radical polymerization of the material [8].

As mentioned, SARCs are a heterogeneous group of materials that possess a complex composition with presence of conventional mono-, di-, and/or multi-methacrylate monomers, phosphate and phosphonate acid-functionalized monomers, fillers, a redox and a photoinitiator. The selection and concentration of each component is relevant for the final performance of these materials. Specifically, acidic monomers must be strong enough to produce an adequate etching of dentin with stable salt formation and low enough to avoid excessive hydrophilicity, which may affect the physical properties of the cements [9]. Other studies have reported widely varying performances of self-adhesive cements, not only regarding bond strength to dentin but also shrinkage behavior [10], physical properties [11], pH values and film thickness [12], water sorption and solubility [5]. Such cited properties could explain variability in adhesive performance [13]. Possible consequences of insufficient adhesion are microleakage, secondary caries, pulp reaction, plaque accumulation and periodontal disease. One of the main reasons of adhesion failure is polymerization shrinkage.

Polymerization shrinkage, as one of the most important disadvantages of dental composites, causes the loss of marginal integrity of the tooth-restoration interface [14]. This process is caused by shortening the intramolecular distances between monomer units in the polymer compared to intermolecular distances between free monomers. However, polymerization shrinkage has not been eliminated even in the most advanced available materials [15].

Different contact or non-contact methods have been used to study polymerization shrinkage of dental composites [16]. One of the non-contact methods for measuring polymerization shrinkage is Digital Image Correlation (DIC) method, a current technique used in biomechanical investigations, material and structure testing, fracture mechanics, etc. So far in the dental and bone biomechanics, DIC was used for researches to study bone reaction on loading impact, interactions between jaws and dentures, designs of different dental restorations, etc [17]. DIC is based on tracking the position of surface markers by specialist software before, during and after polymerization and enables full-field displacement and strain measurement.

The aim of this study was to analyze strain field of the self-adhesive resin cement, Maxcem Elite (Kerr, Orange, CA, USA), during the polymerization (in self-cure mode) using 3D optical system based on DIC method.

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METHODS

Strain field was measured using 3D optical system Aramis 2M (GOM, Braunschweig, Germany) based on DIC method. Prior to experiment, system calibration was performed using the calibration panel for corresponding measurement volume. This volume was chosen based on the dimensions of the measured area on sample surface. After the successful calibration, the measurement could commence.

Calibration is a measuring process during which the measuring system with the help of calibration objects is adjusted such that the dimensional consistency of the measuring system is ensured. The calibration object also contains the scale bar information. The scale bar information is the specified distance between two defined reference points. During calibration, the sensor configuration is determined. This means that the distance of the cameras and the orientation of the cameras to each other are determined. In addition, the image characteristics of the cameras are determined (e.g. focus, lens distortions). Based on these settings, the software calculates from the reference points of the calibration object in the 2D camera image their 3D coordinates [18].

Five samples (ø5 x 2 mm) each of Maxcem Elite (Kerr, Orange, CA, USA) were prepared by filling plastic ring-type molds. The top surface of each sample was sprayed with fine black and white spray (Kenda Color Acrilico, Kenda Farben) to create a stochastic pattern with high contrast for image analysis. Digital images were recorded immediately after sample preparation and then automatically every 10 s. Polymerization shrinkage was monitored over a period of 20 min and 120 pictures were taken. The images were then analyzed using specialist software (Aramis 6.2.0) to determine von Mises strain. Von Mises strain was chosen as it represents an index gained from the combination of principal strain at any given point at which strain occurring on the X-, Y-, and Z-axis will cause failure [19]. Analysis of the strain fields was done using sections and stage points created by software. Five concentric circular sections (Sections 1 to 5) were positioned in the center of each sample. Sections 1-5 were 1 to 5 mm in diameter, respectively. Six stage points were positioned at the distance of 1 mm from each other, where Stage point 0 was positioned in the centre of the sample and Stage point 5 on the circumferential, peripheral segment of each sample. For the purpose of statistical analysis, von Mises strain values were plotted as mean values with standard deviation (SD) for Sections 1 to 5 with 95 % confidence intervals (Table 1).

The tested self-adhesive cement was Maxcem Elite (Kerr, Orange, CA, USA). It contains glycerol phosphate dimethacrylate (GPDM), co-monomers (mono-, di- and tri-functional methacrylate monomers, water, acetone, ethanol, barium, glass, fumed silica, and sodiumhexafluorosilicate) and ytterbiumfluoride mineral fillers. Filler loading is 69 wt% and 46 vol%. Maxcem Elite is dual cure material and in this study, self-curing mode was used. The experiments were done at room temperature.

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Table 1. Mean, standard deviation and 95 % confidence intervals of von Mises strain (%) values.

MaxCem Elite		Stage 6 (1 min)	Stage 30 (5 min)	Stage 60 (10 min)	Stage 90 (15 min)	Stage 120 (20 min)
Section 0	Mean±SD	0,507±0,230	1,677±0,800	3,205±2,240	4,025±3,283	4,492±4,036
	Upper 95% CI	0,583	1,946	3,958	5,129	5,849
	Lower 95% CI	0,429	1,408	2,452	2,921	3,136
Section 1	Mean±SD	0,444±0,126	1,712±0,802	3,082±1,481	3,610±1,723	3,942±1,866
	Upper 95% CI	0,503	2,087	3,775	4,416	4,815
	Lower 95% CI	0,385	1,336	2,388	2,804	3,069
Section 2	Mean±SD	0,673±0,475	1,828±0,984	3,046±1,608	3,646±1,644	3,949±1,778
	Upper 95% CI	0,821	2,135	3,567	4,158	4,503
	Lower 95% CI	0,525	1,521	2,525	3,134	3,395
Section 3	Mean±SD	0,517±0,400	1,340±0,427	2,321±0,912	2,937±1,168	3,555±1,438
	Upper 95% CI	0,620	1,450	2,557	3,239	3,930
	Lower 95% CI	0,413	1,230	2,086	2,635	3,180
Section 4	Mean±SD	0,554±0,443	1,872±1,084	3,675±2,310	4,316±2,823	4,685±3,183
	Upper 95% CI	0,651	2,110	4,211	4,984	5,571
	Lower 95% CI	0,456	1,633	3,140	3,648	3,799
Section 5	Mean±SD	0,507±0,494	2,192±1,307	6,226±3,375	8,476±4,269	9,862±4,259
	Upper 95% CI	0,603	2,454	7,043	9,501	10,901
	Lower 95% CI	0,411	1,930	5,409	7,450	8,823

RESULTS

Polymerization shrinkage of Maxcem Elite is illustrated in Figure 1 using von Mises strain. Von Mises strain values were plotted as a function of strain stage i.e. time along each stage point. The results showed images for stage 0, 6 (1 min after the beginning of recording), 30 (5 min after the beginning of recording), 60 (10 min after the beginning of recording), 90 (15 min after the beginning of recording) and 120 (20 min after the beginning of recording).

The highest values of von Mises strain were measured peripherally, on the cement-mold interface. Mean values of von Mises strain for Section 5, positioned on the interface itself, were 9.862 ± 4.259 % in the Stage 120. Mean values of von Mises strain for Sections 1 to 4 decreased significantly compared to Section 1. There were no significant strain differences for centrally positioned Section 1 (3.942 ± 1.866 %) and Section 2 (3.949 ± 1.778 %). Furthermore, the lowest values of von Mises strain were measured on Section 3. Mean values of von Mises strain for Section 3 were 3.555 ± 1.438 % in the Stage 120. It should be noted that peak values of von Mises strain measured on the periphery of the sample reached almost 15%.

Von Mises strain values for Section 0 represent a mean strain value for the entire specimen surface, as Section 0 is positioned across the diameter of the specimen (Figure 1). Mean values of von Mises strain for Sections 1 to 3 (3,942 %, 3,949 % and 3,555 %, respectively) are showing slightly lower values than Section 0 at stage 120 (4,492 %), while Section 4 is showing slightly higher values (4,685 %). However, mean value of von Mises strain for Section 5 (9,862 %) is significantly higher.

As it can be seen in Figure 2, strain values were increasing in Stage points 0–4 until Stage 90 is reached after 15 min. After that strain change is negligible. However, strain values were increasing constantly in Stage points 5 during the experiment.

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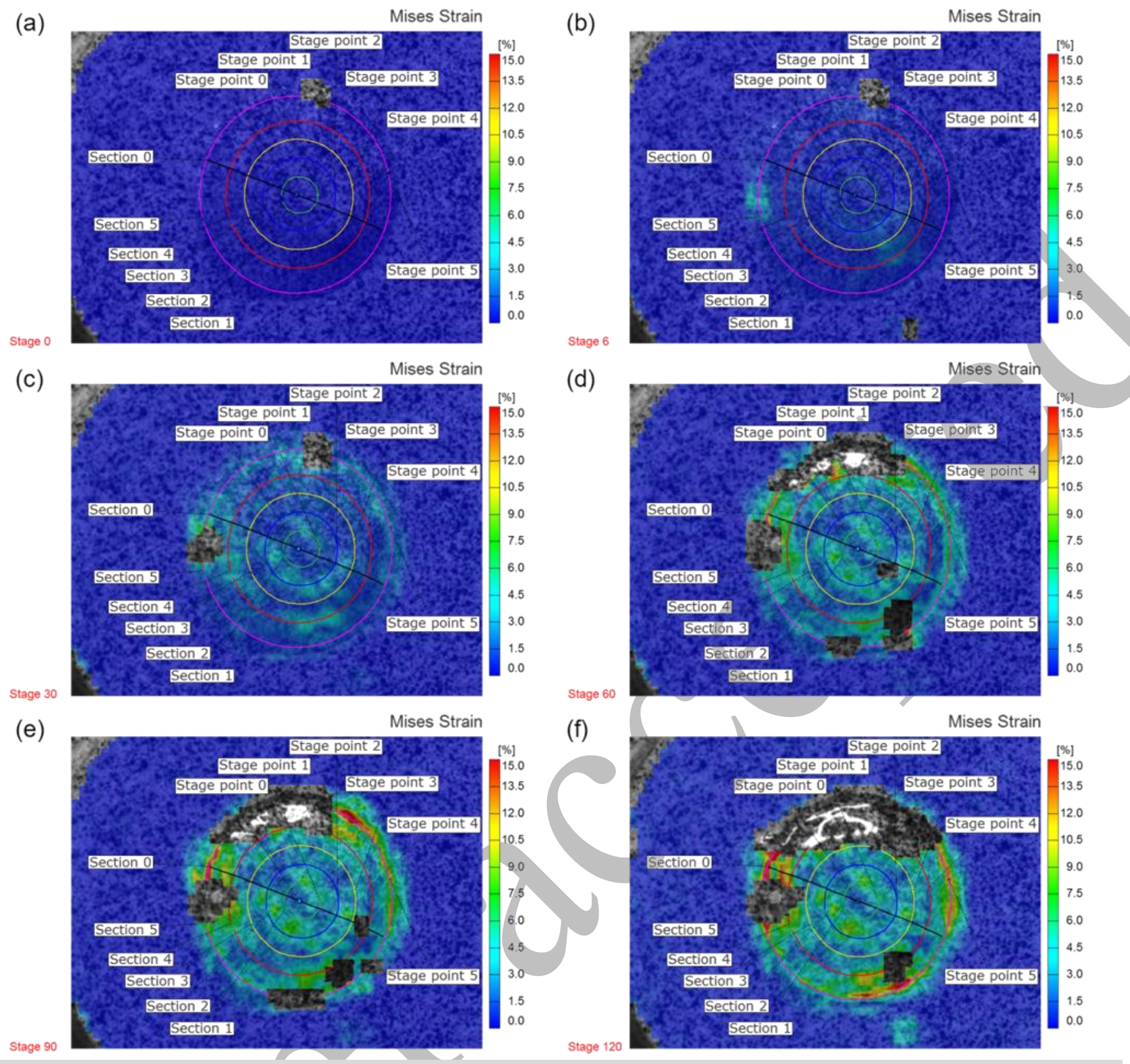

Figure 1. Von Mises strain field. (a) Stage 0. (b) Stage 6. (c) Stage 30. (d) Stage 60. (e) Stage 90. (f) Stage 120.

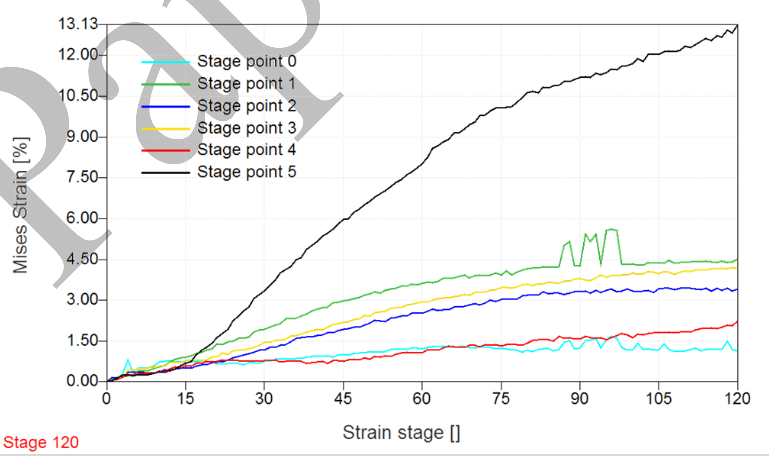

Figure 2. Von Mises strain in Stage points 0-5 as a function of Strain stage (i.e. time).

DISCUSSION

A single-camera 2D-DIC measurement system has been proven reliable for determining the polymerization shrinkage of resin-based composites. With one camera systems, in-plane displacements are measured and out-of-plane is assumed based on material properties. Two cameras allowed 3D

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measurements of dimensional changes during polymerization including out-of-plane strain and displacement values [17]. A fine spray of black paint is often used to create the stochastic surface pattern for detection by the DIC based system. It is considered to have neither an effect on the shrinkage characteristics nor on light translucency of the composite material.

The presented results indicated non-uniform strain distribution in resin cement and two distinctive zones were identified across the surface of the material, with higher strain values along the peripherally and lower strain values in central parts of each sample. Plotting strain values for Sections 1 to 5 further elucidates heterogeneous nature of strain fields. Highest strain values were measured on the periphery of the sample (Section 5), as the polymerization shrinkage is oriented towards centre of the sample. This directly influences the strain values in the central part of the sample (Sections 1 and 2), as the molecules in the polymer chains shift to center of the sample mass. This molecule rearrangement in the outer segment occurred predominantly as the in-plane shrinkage in the *x*- and *y*-axis and is increasing the strain values in the vicinity of the sample centre. Due to this fact, Section 3 has more favourable molecule arrangement compared to strain values in Section 1 and Section 2. Non-uniform strain of Maxcem Elite may be useful in clinical application as it is difficult to precisely predict the zones of the highest strain values within the material in clinical conditions where many variables are involved.

The 3D DIC method has some limitations. As the 3D computation of the measuring points is based on pixels that need to be seen from the right and left camera with the individual facet pattern, a correct 3D computation and strain computation is not possible for sample edges, specific samples shapes, poorly applied stochastic surface pattern or light reflection [20]. As strain values increased with time, light reflection occurred on cement surface and limited facet pattern visibility, which lead to data loss on the strain field.

Since resin cements are generally applied as a thin layer between an indirect restorative material and the tooth tissue, a low viscosity is required [21]. Therefore, polymerization shrinkage is an important factor, which can depend upon composite mass (or volume) [22]. Low-viscosity composites, especially flowable types, still have a relatively high shrinkage of up to 6 %, in large part due to the low filler fraction, commonly below 50 vol. % [23]. Resin adhesives can reach higher polymerization shrinkage values, up to 13 % [24]. Increased, non-uniformly distributed shrinkage strain values at the adhesive interface induced by shrinkage of flowable resin cements may compromise bonding and lead to adhesive failure. This may have a particular importance in the case of Maxcem Elite, which is intended to be used without an adhesive. Shrinkage of Maxcem Elite may compromise its bonding effectiveness due to high shrinkage values. According to manufacturer's information, as mentioned, Maxcem Elite contains an acid monomer, glycerol dimethacrylate dihydrogen phosphate (GPDM), which is partly responsible for the effect of etching and adhesion to the substrate [25]. Other studies have shown that Maxcem Elite does not have a relevant acid-base reaction while setting, as do other self-adhesive cements, maintaining a low pH for a long time [9],

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which could adversely influence the formation of an optimal cross-linked polymer network. D'Alpino et al. found a linear correlation between shrinkage strain rate and the filler-volume [26]. With increasing filler-volume percent the shrinkage strain decreased [27]. Filler content of investigated material is 69 % of weight respectively 46 % of volume. Tested material in this study was applied directly by an automix syringe. Maxcem Elite is characterized by an amine-free redox initiator system, which may help prevent chemical incompatibility between acidic groups and self-curing components [28]. Chemical and thermal instabilities at the filler/organo-silane bonds may lead to the formation of cluster agglomeration within the resin matrix. The stress development in dental composites depends on the organic matrix, the type and amount of fillers, and the filler/matrix interactions. High filler content produces stiffer polymers and higher polymerization stress levels [29].

Monomer type may also influence bond strength performance due to chemical interaction between specific monomer functional groups and the bonding substrate and chemical initiators may benefit polymerization in the absence of light [30]. The volumetric shrinkage resultant from the establishment of covalent bonding among methacrylate groups is determined by the monomeric composition, as the higher the concentration of high molecular weight monomers, the lower the amount of carbon double bonds per unit volume. Also, high molecular weight monomers in general present lower mobility, also contributing to a lower shrinkage [31].

Though, the role of each component on the final properties of the materials has not been clarified yet. The complex formulation of self-adhesive cements is only partially disclosed by manufacturers, making it difficult to explain the strain differences among commercial materials through their resin composition and inorganic content.

Although, the guideline for preparation the SARC ordains that the polymerization time last at minimum 5 min, the DIC showed that dimensional changes i.e. strain changes continue and stabilize after 15 min in all points of Maxcem Elite. Also, this fact highlights the advantage of the DIC method as a power tool for investigation in technology and dentistry research fields. Further investigation will be conducted in order to better understand in situ polymerization shrinkage of resin cements. Experiments, beside DIC method, such as hardness and degree of conversion will be done on numerous samples of Maxcem Elite but also on other SARCs. Hardness has been found to be sensitive to the residual monomer content in the polymerized resin and it is a simple and effective way to assess the degree of conversion of SARCs. Due to this fact, it will be easier to have wider picture about complexity of polymerization shrinkage of resin cements.

CONCLUSION

Our contribution in this paper consists of following conclusions:

1. A possible reference model for investigation of composite cements was presented.
2. DIC method can serve for visualization of strain generated in the samples of Maxcem Elite.

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3. Non-uniformly distribution of shrinkage strain was registered, with the highest strain generated peripherally.
4. Section 1 and Section 2 of the Mises strain field showed higher strain values than the Section 3 due to molecule rearrangement in the outer segment of Maxcem Elite that occurred predominantly as the in-plane shrinkage in the x- and y-axis and is increasing the strain values in the vicinity of the sample centre.
5. It can be argued about the increased time of polymerization in the examined self-adhesive resin cement, Maxcem Elite, and positive correlation with fillers as the key factor for the polymerization shrinkage.

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