Lecture Notes in Networks and Systems 54

Nenad Mitrovic
Milos Milosevic
Goran Mladenovic *Editors*

Experimental and Numerical Investigations in Materials Science and Engineering

Proceedings of the International Conference of Experimental and Numerical Investigations and New Technologies, CNNTech 2018

Springer

Lecture Notes in Networks and Systems

Volume 54

Series editor

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ISSN 2367-3370 ISSN 2367-3389 (electronic)
Lecture Notes in Networks and Systems
ISBN 978-3-319-99619-6 ISBN 978-3-319-99620-2 (eBook)
https://doi.org/10.1007/978-3-319-99620-2

Library of Congress Control Number: 2018951894

This Springer imprint is published by the registered company Springer Nature Switzerland AG
The registered company address is: Gewerbestrasse 11, 6330 Cham, Switzerland

Preface

The book is a collection of high-quality peer-reviewed research papers presented at the International Conference of Experimental and Numerical Investigations and New Technologies (CNNTech 2018) held at Zlatibor, Serbia, from July 4 to 6, 2018. The conference is organized by the Innovation Center of the Faculty of Mechanical Engineering, Faculty of Mechanical Engineering at the University of Belgrade, and Center for Business Trainings. Over 30 delegates was attending the CNNTech 2018—academicians, practitioners, and scientists from 11 countries—presenting and authoring 40 papers. The conference program included two keynote lectures with five invited lectures, four sessions (oral and poster), and two workshops. Seventeen selected full papers went through the double-blind reviewing process.

The main goal of the conference is to make positive atmosphere for the discussion on a wide variety of industrial, engineering, and scientific applications of the engineering techniques. Participation of a number of domestic and international authors, as well as the diversity of topics, has justified our efforts to organize this conference and contribute to exchange of knowledge, research results, and experience of industry experts, research institutions, and faculties which all share a common interest in the field in experimental and numerical investigations.

The CNNTech 2018 was focused on the following topics:

- Mechanical Engineering,
- Materials Science,
- Chemical and Process Engineering,
- Experimental Techniques,
- Numerical Methods,
- New Technologies.

We express our gratitude to all people involved in conference planning, preparation, and realization, especially to

- All authors, specially keynote speakers and invited speakers, who have contributed to the high scientific and professional level of the conference,
- All members of the Organizing Committee,

- All members of the International Scientific Committee for reviewing the papers and Chairing the Conference Sessions,
- Ministry of Education, Science and Technological development of Republic of Serbia for supporting of the Conference.

We wish to express a special gratitude to Ms. Dragana Perovic for her effort in preparing and managing the conference in the best way.

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Contents

Materials Science

Thermal and Mechanical Characteristics of Dual Cure Self-etching, Self-adhesive Resin Based Cement

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Abstract. One of the main objectives in research and development of resin based cements (RBCs) is to enhance their clinical longevity and ease of use. In spite of the undeniable technological advances introduced in the last few decades, the polymerization shrinkage i.e. strain that accompanies the chain-growth polymerization of dimethacrylate monomers remains one of the major concerns for the clinical performance of composite restorations. Also, RBCs can produce a considerable amount of heat, due to the light energy from the curing lights and exothermic reaction of polymerization.

The purpose of this study was to determine the temperature changes during the photo-polymerization using thermocouples and to measure strain field of the self-etching, self-adhesive RBC, Maxcem Elite (Kerr, Orange, CA, USA) (ø5 × 1 mm - Group I and ø5 × 2 mm - Group II) using experimental technique, 3D Digital Image Correlation (DIC) method. Digital images were recorded immediately after photo-polymerization of the samples with a LED-curing unit for 20 s, according to manufacturer's recommendation. Vickers microhardness was determined after photo-polymerization and after 24 h. Temperature curves for both groups indicated similar patterns but the peak temperature of Group II was significantly higher compared to peak temperature of Group I. DIC showed that peripheral zone of the samples had the highest strain values in both groups. Group I indicated significantly higher values of hardness. All the results were material-dependent and probably correlated to the composition of each material, which is not fully disclosed by the manufacturers.

Keywords: Resin based cement · Temperature change · Thermocouples Strain · 3D Digital Image Correlation · Vickers microhardness

N. Mitrovic et al. (Eds.): CNNTech 2018, LNNS 54, pp. 3–15, 2019.
https://doi.org/10.1007/978-3-319-99620-2_1

1 Introduction

Resin based cements (RBCs) have extraordinary aesthetic shade-matching potential and due to the higher esthetical demands of patients for dental restorations, they have become very popular [1]. In an attempt to simplify procedures, a new group of RBCs, self-etching, self-adhesive resin cements (SARCs), have been developed. According to their manufacturers, these materials are self-adhesive, including acidic and hydrophilic monomers in their composition, which simultaneously demineralize and infiltrate enamel and dentin, providing strong bonding [2]. However, one of the shortcomings of RBCs is polymerization shrinkage that accompanies the chain-growth polymerization of dimethacrylate monomers and generates stress at the tooth and composite interface. The strain induced in RBCs during the polymerization can affect therapeutic failure due to de-cementation i.e. de-bonding. This strain depends on cement-layer thickness and presents the major source of shrinkage stress [3]. It has been shown that an increment thickness of 2 mm should not be exceeded for these materials with conventional chemistry [4]. Several methods were proposed to measure shrinkage strain such as dilatometry, the bonded disc method and strain gauges. Current methods include the Digital Image Correlation (DIC) method as an optical sensor for detecting shrinkage strain [5–7]. Heretofore, the hardness tests were widely used to examine RBCs and to analyze the efficiency of the light units, its wear resistance ability to keep stabile form and to have better insight into the degree of conversion (DC) during polymerization [8, 9]. Association of these tests with optical methods for strain analysis can reveal potential negative sides of the RBCs and improve different properties of currently available dental cements.

Another disadvantage of resin based cements is heat emission during polymerization [10]. The polymerization reaction of RBCs involves rupture of the C=C bonds of dimethacrylate monomers present in their polymeric matrixes and the conversion of intermolecular distances of 0.3–0.4 nm between polymer chains, maintained by Van der Waals attraction forces, into primary C–C covalent bonds [11]. Polymerization of these materials used for the fabrication of temporary restorations is associated with an exothermic reaction since the final formed enthalpy in the network is lower than that of the amount of the initial monomer. This temperature rise may cause thermal trauma to the pulp [12]. It has been proved that the pulp chamber is sensitive to physical, chemical, biological and thermal changes [13]. An increase of the intrapulpal temperature exceeding 42.58 °C can result in serious damage of the pulp tissue. Exothermic reactions of the composite resin and radiant heat from the light-curing unit contribute to heat production [10, 14]. Any curing unit that emits radiant energy in the blue area of the electromagnetic spectrum, i.e., between 400 and 500 nm, can be used to start the polymerization reaction of RBCs. Curing units with light-emitting diodes (LEDs) have been increasingly used lately. These light-curing units emit a narrow light spectrum with wavelengths close to the absorption peak of camphoroquinone (468 nm), i.e., the photosensitizer most used in RBCs [4].

Differential thermal analyses [15, 16], differential scanning calorimetry [17], infrared thermography [18, 19], thermistors [20, 21] and thermocouples [22–26] are some of methods that have been used to measure temperature increase during the polymerization of composite resin.

This study aims to determine the temperature changes during the photo-polymerization with LED lamp in a dual-cured self-etching, self-adhesive resin based cement, Maxcem Elite, using thermocouple, as well to measure strain and microhardness of investigated material.

2 Experimental Setup and Procedure

The research was carried out as an experimental study. The tested Maxcem Elite (Kerr, Orange, CA, USA) 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.

The temperature measurement during the polymerization period was performed using a predefined procedure. The thermocouples used for temperature measurements were prepared using the OMEGA fine diameter bare wire (0.078 mm) K type (chromel-alumel) thermocouple, with special limits of error (±0.4%). The thermocouple bead was disk shaped to maximize temperature sensing surface area. The thermocouple was embedded into the center of the Maxcem Elite samples. The thermocouple voltages were acquired by HIOKI LR8431-20 high-speed multichannel data logger.

The teflon ring-type molds (ø 5 mm × 1 mm – Group I and ø 5 × 2 mm – Group II) were placed on a strip on the top of the quartz laboratory glass plate suspended on the laboratory stand. The LED curing light for the polymerization of both groups was placed just beneath the glass plate. The mold was filled with Maxcem Elite (Fig. 1). The samples were then cured for 20 s with a LED lamp (450–500 mW/cm^2, LEDition, Ivoclar-Vivadent, Schaan, Liechtenstein) according to manufacturer's recommendation. The temperature was recorded at 0.1 s intervals throughout the curing process and afterwards until it returned to the baseline level. The temperature of the top side of the glass plate, between the mold and the glass, was measured with a separate thermocouple in order to assess the heat input generated form the LED curing light. The extensive measurement showed that the heat input from the LED curing light was minimum and constant, contrary to the tested material polymerization temperature.

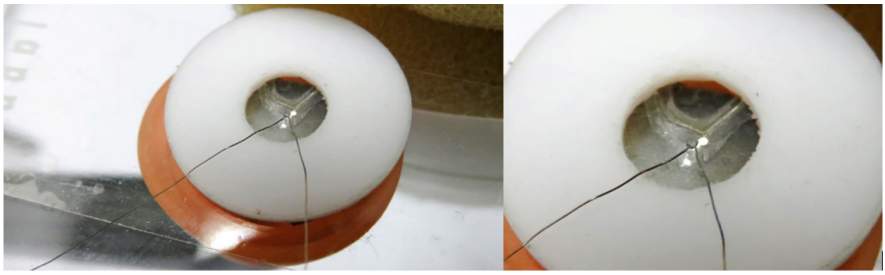

Fig. 1. Experimental setup for temperature measurement of Maxcem Elite during the polymerization.

Strain field was measured using 3D optical system Aramis 2M (GOM, Braunschweig, Germany) based on DIC method. The Aramis system consisted of two digital cameras with the resolution of 1600 × 1200 pixels and specialist software (Aramis v6.2.0). 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. Two groups of the samples were prepared. First group (Group I) included three ø5 × 1 mm sized samples of Maxcem Elite (Kerr, Orange, CA, USA) and second group (Group II) included three ø5 × 2 mm samples of the same material. All samples were prepared by filling Teflon ring-type molds. The molds were placed on a strip on the top of the quartz laboratory glass plate suspended on the laboratory stand. 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 (before polymerization, Stage 0) and immediately after photo-polymerization with LED lamp (after 20 s, Stage 1), in accordance with the manufacturer guidelines.

The images were then analyzed using software Aramisv6.2.0 to determine von Mises strain which represents criteria for the 3D deformation analysis in the x-, y- and z-axes of the photographed surface. Analysis of the strain fields was done using three sections (Sections 0, 1, 2). Circular section (Section 0) was positioned peripherally at the mold/material interface and two linear sections (Sections 1 and 2) were positioned orthogonally. Length of the Sections 1 and 2 corresponded to the sample diameter.

Microhardness measurements were performed with a digital device ECHOLAB type HTV 100 (Echo Research & Development S.p.a) that allows measurement of Vickers and Knoop micro-hardness. Device can be moved in two perpendicular directions, allowing distance measurement that ensures recommended distance (more than 2.5 d) between impressions. The force that can be applied to the device is 0.098–980.7 N (HV0.01–HV100). Measurements of the samples for both groups (N = 3) were carried out with a load of 0.49 N (HV0.05), so the ratio of sample thickness (1 mm and 2 mm) and mean-diagonal indentation was higher than 22 (min > 9) which meets the test requirements (sample thickness greater than 1.5 d). Loading time was 15 s. Vickers microhardness was measured for each group immediately after polymerization and after 24 h on the exposed surface (surface that was directly exposed to LED lamp). Mean hardness value for each sample was calculated. All experiments were performed at room temperature.

3 Results and Discussion

Thermocouples were selected to evaluate temperature alterations during the polymerization of tested material due to high precision and reliable readings. At each measurement, the thermocouples were placed in the same position to minimize variation in measurements that can be caused by changes in thermocouple position. The ambient temperature was constantly maintained because the temperature may affect the resultant net temperature rise for a given amount of energy dissipation, in accordance with Newton's law of cooling. Investigated material exhibited exothermic reaction during

photo-polymerization. The temperature measured within the samples increased quite rapidly with the initiation of light curing. Group I reached maximum peak value of 46.2 °C within 15 s and Group II 54.2 °C within 19 s (Fig. 2). In accordance with the manufacturer guidelines, photo-polymerization lasted 20 s. The mean temperature increase for the Group II was higher than the mean temperature increase for the Group I. During the next 10 min, the temperature started to decrease to the values of 27.6 °C and 29.9 °C for the Group I and Group II, respectively. The curves reached plateau after 2 min. Temperature curves for both groups indicated similar patterns, but Group I showed less sharp increase in temperature than Group II. The peak temperature of Group II was significantly higher compared to peak temperature of Group I. This can be explained by the higher concentration of the accelerator due to the greater volume of the samples in Group II.

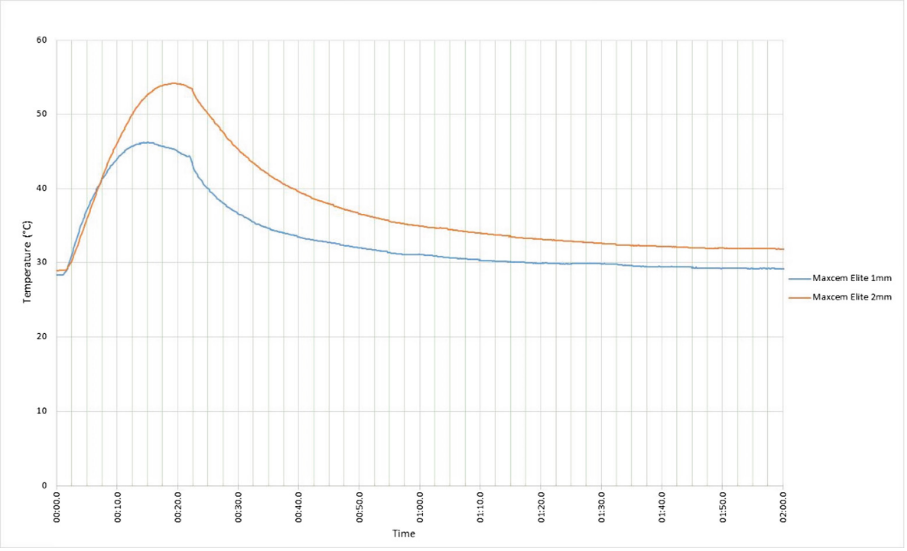

Fig. 2. Representative temperature curves for the Group I and Group II recorded during the photo-polymerization.

Both groups showed a gradual temperature reduction after the peak values due to the heat loss through dissipation to the surroundings exceeded the heat. The heat generated by the exothermic reaction mostly occurred in the initial phase of polymerization. Many studies have demonstrated a positive relationship between the light-curing unit intensity and temperature rise [19, 21, 23, 27]. Some in vitro studies reported that temperature rise depends on material used and photoactivation protocol i.e. depends, beside the light-curing unit type, on power density, exposure duration, the distance between composite surface and light guide tip end, composite shade and

thickness of composite materials [28–30]. Many authors have quantified the amount of heat generated in resin-containing material during light curing. It was found that the temperature increase with LED lamps varies from 41 °C to 53 °C [31] which is consistent with our results. However, the measured temperatures recorded by Vallittu (6–40 °C) [32] were generally lower than measured temperature for Group I and Group II. This can be explained by the differences in the test conditions because the amount of exothermic heat during polymerization depends upon the amount of materials and the ambient temperature [33].

It is necessary to emphasize the limitations of in vitro studies (methodology). A significant amount of heat was generated in both groups of the investigated material, Maxcem Elite. Clinicians should be aware of the heat generated in RBCs during light-curing, which may be a potential source of pulpal injury.

The strain fields of representative samples of Group I (Fig. 3a and b) and Group II (Fig. 4a and b), after polymerization in Teflon molds, were shown.

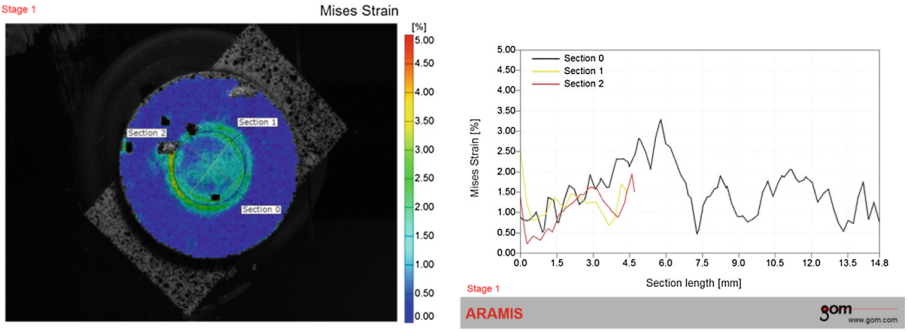

Fig. 3. (a) Shrinkage strain field at the Stage 1 of Group I; (b) von Mises strain as a function of distance for each section.

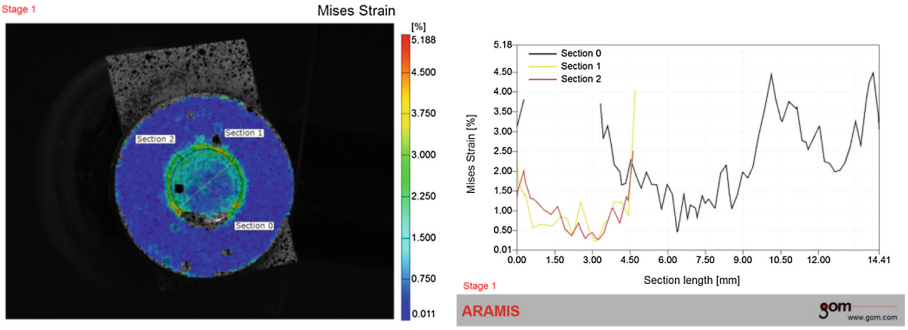

Fig. 4. (a) Shrinkage strain field at the Stage 1 of Group II; (b) von Mises strain as a function of distance for each section.

Section 0 (circular section) was placed at the mold/tested material interface. Section 1 and Section 2 represented linear sections that were orthogonally positioned. Group I and Group II showed a non-homogeneous strain field, especially in the center of the sample. Minor differences between mean strain values in the peripheral zone (Section 0) and central zone (Section 1 and Section 2) were presented for Group I samples (Table 1). The highest strain values (about 3.5%) were observed on the peripheral zone for Group I (Fig. 3a). For Group II, the highest strain values, about 4.0%, were noticed as well on the peripheral zone (Fig. 4a). However, major differences between mean strain values in the peripheral zone (Section 0) and central zone (Section 1 and Section 2) were observed for Group II. Obviously, peripheral zone was subjected to the highest strain values in both groups.

Table 1. Mean and standard deviation (SD) of von Mises strain (%) values.

Maxcem Elite ø5 × 1 mm (Group I)	Mean value of von Mises strain	Standard deviation
Section 0	2.206	0.704
Sections 1 and 2	1.578	0.709
Maxcem Elite ø5 × 2 mm (Group II)	Mean value of von Mises strain	Standard deviation
Section 0	2.084	0.843
Sections 1 and 2	0.897	0.674

It should be noted that the values in the central zone for Group I were significantly higher compared to Group II, since they had a similar value at the periphery. Possible explanation lies in fact that due to elongation, in absolute numbers, that is proportional to the material's initial dimension, thicker layers favor stress relief. Strain values reported in this study are consistent with dental composites, which range between 2% and 3% [34, 35].

Digital Image Correlation based on a two-camera 3D measurement system has been proven reliable for determining the strain i.e. polymerization shrinkage of RBCs [36–39]. In this study von Mises strain immediately after polymerization for both Maxcem Elite groups was measured. Unlike other methods for determining the dimensional stability of composite materials, the DIC method also enables the measurement of maximum strain value. In light-cured samples, the polymerization is much faster so the polymer matrix becomes semi-rigid in a few seconds. Therefore, immediately after light activation, the strain reached a high value, although the polymerization and crosslinking of the matrix were still on-going [40]. The study confirms previous findings that shrinkage behavior, including shrinkage magnitude of strain, is influenced

by the curing mode in some degree [3, 4, 41, 42]. Particularly, this means that LED lamp may modify the overall strain pattern in terms of strain distribution. Previous studies [43, 44] have been conducted on the standardized sample size showing data for mean strain. These methods excluded peripheral section (Section 0) although the peripheral strain has to be considered when interpreting the overall strain. DIC method, in this study, detected maximal von Misses strain values and determined the zones of the maximal strain through presenting images of 3D full strain field. This study revealed additional information about curing mode dependent shrinkage patterns focusing on shrinkage and strain.

Vickers microhardness (HV) in both groups Maxcem Elite were measured immediately after the photo-polymerization and after 24 h. Mean microhardness values of the tested material are listed in Tables 2 and 3.

Table 2. Values of microhardness for Group I

Sample no.	Immediately after polymerization	Exposed surface
		HV 0.05
Sample 1	1	34.71
	2	38.52
	Average	36.62
Sample 2	1	35.22
	2	38.62
	Average	36.92
Sample 3	1	36.91
	2	34.62
	Average	35.77
Sample no.	After 24 h	Exposed surface
		HV 0.05
Sample 1	1	39.32
	2	36.81
	Average	38.07
Sample 2	1	37.46
	2	37.46
	Average	37.46
Sample 3	1	35.56
	2	35.48
	Average	35.52

Table 3. Values of microhardness for Group II

Sample no.	Immediately after polymerization	Exposed surface
		HV 0.05
Sample 1	1	25.13
	2	23.13
	Average	24.13
Sample 2	1	30.38
	2	31.01
	Average	30.70
Sample 3	1	26.08
	2	27.45
	Average	26.77
Sample no.	After 24 h	Exposed surface
		HV 0.05
Sample 1	1	28.73
	2	28.73
	Average	28.73
Sample 2	1	35.30
	2	30.94
	Average	33.12
Sample 3	1	29.77
	2	31.01
	Average	30.39

Considering the results for Group I, the values of microhardness immediately after polymerization and after 24 h were similar. Measured values of hardness were consistent as well for Group II. Group I indicated significantly higher values of hardness than Group II, which is in accordance with the results in the literature [5, 8, 22, 36]. Hardness is interpreted as a statistical magnitude that practically depends on the composition of the tested material structure, particularly in this case, for dual-phase structure like Maxcem Elite is. As the HV indenter is greater compared to the size of the fillers and the space in between filled with polymer matrix, the resulting HV value is a measurement of the filler-matrix system. The filler component is dominant factor compared to the softer polymer matrix [4]. HV indirectly considers the matrix network crosslinking. With 46% of filler volume fraction, Maxcem Elite is lower filled compared to other RBCs [45].

In dual-cure RBCs, there is a post-polymerization reaction within the polymer, which results in increased strain after a few minutes and higher values of hardness after 24 h. Lower content of fillers particles may have contributed to additional crosslinking or simple physical reorganization of polymer chains, which also leads to an increase in strain and hardness [46]. Proportional relationship between microhardness and polymerization shrinkage of the material has been shown by Li and co-workers [47]. This finding was in general agreement with other studies [45, 48], which revealed that the

strain was related to the hardness. Previous studies investigated the microhardness of self-adhesive cements and found that the values greatly varied according to the brand [9], showing a very strong influence of the material [8].

4 Conclusions

Self-etch, self-adhesive resin cement, Maxcem Elite, was investigated. In the present study, the instructions of the manufacturer were followed with regard to the photo-polymerization time of the tested material. Results demonstrated that temperature increased inevitably after the initiation of the light curing for both groups. These increases were associated with damage to dental tissues. A significant amount of heat was generated in both groups. Clinicians should be aware that the heat generated in tested material during light-curing may be a potential source of pulpal injury. Group I and Group II showed a non-homogeneous strain field, especially in the center of the sample. However, peripheral zone was subjected to the highest strain values in both groups. Also, this fact highlights the advantage of the DIC method as a power tool for investigation in dentistry research fields. Group I indicated significantly higher values of hardness than Group II. The results were also material-dependent and correlated to the composition of the material. The role of each component on the final properties of the material has not been clarified yet. The complex formulation of tested Maxcem Elite is only partially disclosed by manufacturer, making it difficult to explain the strain differences through resin composition and inorganic content. It is important to bear in mind that all this study has been made in vitro and therefore, has some limitations. Further investigation will be conducted in order to better understand temperature and strain changes in the tested material.

Acknowledgement. The authors are grateful to Neodent (Belgrade, Serbia) for providing the material used in this study. This research was supported by Ministry of Education, Science and Technological Development of Republic of Serbia under Project TR35031.

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