Dental tissue - inlay interface structural shift at mechanical and thermal stresses

B. M. GALBINASU[*], F. MICULESCU[a], D. PIRVU, L. T. CIOCAN, D. HERSCHBACH[b], A. TONKO, I. PATRASCU,
F. STOICIU[c]
"Carol Davila" University of Medicine and Pharmacy, Bucharest, Romania
[a] *"Politehnica" University of Bucharest, Romania*
[b] *Private Medical Practice, München, Germany*
[c]*The National Institute of Research and Development of Rare and Nonferrous Metals, Bucharest, Romania*

All indirect ceramic dental restorations represent a challenge, both in terms of aesthetics and interactions with the adjacent biological tissues, the ceramic mass being considered a biotolerated, bio-prophylactic, and bioinert material. The interface between advanced ceramic restorative materials, fixing resins and dental hard tissues has always been considered a sensitive domain in terms of maintaining lasting prosthetic restorations and also of the predictability of restorations included in the treatment plan. The objective of this study is to *in vitro* investigate the changes occurring at the adhesive interface, tooth-whole ceramic inlay, after subjecting the assembly to thermal and mechanical stresses that have outcomes on the restoration clinical behaviour. In order to obtain the best and reproducible results, we used a thermo-cycler system, a computer assisted mechanical testing system, and the electron microscopy for testing of all-ceramics inlays fixed on permanent intact teeth.

1. Introduction

Patients' perception of aesthetics and biocompatibility offered by all ceramic restorations has led to an increase in all ceramic restoration numbers in all quadrants and areas of oral cavity. Therefore all ceramic restorations can be regarded as sustainable and predictable treatment options not only aesthetically, but mostly functionally [1]. Many research evaluations focused on the importance of the assessment on the interface between all ceramic inlay restorations (in the present study called dental inlay) and the dental tissue [2-7]. The focus is based on its importance in terms of durability and aesthetic outcome. Adhesive techniques represented a significant step for patients who needed reconstruction of lost hard dental tissues. Given the continuous development of advanced materials of all ceramic restorations and of fixing adhesive resins, it is required a constant assessment and research on the interface between the above mentioned ceramic and the hard dental structures.

Clinical studies emphasized the importance of marginal accuracy preparation for attaining the clinical success. On the other hand, a sparse adaptation of all ceramic restorations can affect both the tooth and the periodontal tissue that could lead to the damage of the cement agent on the edges, thus allowing bacterial infiltration. In addition, the restoring strength may be influenced by poor marginal adaptation [7, 8]. It is therefore of a major importance the proper way of obtaining all ceramic restorations, in terms of mechanical qualities and especially of their adaptation to the geometry of the dental preparation. Therefore by using the Computer-Aided Design/Computer-Aided Manufacturer (CAD/CAM) technology, it may be possible to eliminate both variables entered by the operator in the laboratory stage and reducing the number of clinical steps necessary to achieve the final prosthetic restoration. The technical publications present various methods for evaluating the marginal closing, with both advantages and disadvantages. Sorensen *et al.* [9] present a classification of the available methods, into four basic categories: direct, through cross sectioning, impression, and exploration or visual examination (the radiological method) [8]. The qualitative techniques, such as exploration or visual examination are not accurate *per se*, often being subjective, inevitably introducing the operator opinion. The method is less accurate when the margins of the restoration are sub gingival [10]. As for radiographic techniques, besides the fact that they are not precise, they are not always applicable to all ceramics because they do not offer a proper resolution of the details. A common method used to assess marginal deficiencies is the impression method, using fluid impression materials [11, 12]. This non-invasive method is difficult to be applied for restorations with an excellent marginal and internal adaptation, because of the distortion or degradation risk due to the material edges that can alter the results [13, 14]. Profilemetrics is another non-invasive method used to

quantify the limits of restoration [15]. Although profilemetrics is considered to be an adequate method, it was proved that if there is a vertical over contouring of the all ceramic restorations, pictures cannot be obtained, misleading to interpretation of likely results. Another frequently used method to measure the marginal lack of adaptation is the optical microscopy or stereomicroscopy. The main advantage of the latest methods is the accuracy, since the integrity of the restoration is not of interest. On the other hand, if there is a slight deviation of the angle to which the examination is made, the parameter changes and consequently, distorted results, could appear. In the eventual case of the specimens subjected to sectioning when the specimen section is examined either optically [16, 17] or by electronic microscopy [18-21], the examination result is not affected by the angle at which the microphotography is made. After the specimen section preparation for analysis, there are not needed any other additional actions. Another major advantage offered by electron microscopy consists in the possibility of performing repeated analysis, without damaging the samples. This study is supposed to analyze the interface between hard dental tissues and fixing adhesive resin using scanning electron microscopy (SEM), after the joining area was subjected to similar mechanical stresses and thermal conditions as in the oral cavity. In terms of thermal conditions, the present method used a thermo-cycler. With its aid temperature variations were obtained similar to those existing during physiological mastication and deglutition. As for the mechanical stress test, we used an ensemble, originally named "Equipment for Testing the Cohesion of Mixed Dental Materials", an invention that belongs to three co-authors of this paper [22]. The ultimate goal of the present study was the selection of the materials, the appliance of advanced products and technologies, in terms of physical-chemical parameters and especially emphasized on the clinical results.

2. Materials and methods

To obtain statistically accurate results 15 teeth (molars and premolars) free of carious lesions were used. Immediately after extraction, the teeth were washed with ultra pure deionised water. Periodontal tissues, plaque, and traces of proteins were removed and teeth were maintained for 24 to 48 hours in containers with chloramine T 0.5 % (chloramine T-SIN, Sintofarm, Bucharest). Subsequently, dental structures were refrigerated for less than 6 months, at 2- 4 ° C in ultra pure deionised water, regularly changed. Sampling protocol and preservation of the dental structures necessary to this survey were conducted in accordance to ISO/TS 11405:2003 (E) "dental materials-testing the adhesion to tooth structure" [23]. The required study groups were obtained by dividing the random batch of 15 teeth in 3 groups of 5 teeth each. In our study, group A (A 0) is the control group. Group B (B 90) is subjected to vertical forces, parallel to the tooth long axis and perpendicular to the occlusal surface, while group C (C 45) is subjected to forces with to an angle of 45 degrees along the tooth axis.

According to the literature [5, 6] it has been established a force (F) of 100 N, requesting a tooth occlusal demand. Groups B 90 and C 45 were subjected to this force during 20 cycles. In the case of group C 45, due to tooth inclination at 45 degrees to the direction of the applied force, using the parallelogram rule (Fig. 1) the vertical force, F, can be decomposed into the components, F_1 and F_2, as: $F_1 = F \cos 45 °$, $F_2 = F \sin 45 °$. Therefore it results that $F_1 = F_2 \approx F \times 0.7 = 70$ N.
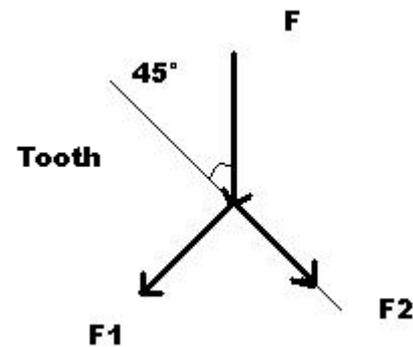

Fig. 1. Decomposition of F down force, when tooth is inclined with 45 degrees to the direction of force.

In the C 45 group there are two forces acting on dental structure, one force with the vector direction parallel to the long axis of the tooth and a force perpendicular to the axis of the tooth, both of 70 N. On each tooth (of the 15 group) a Mesial Occlusal Distal cavity was realized (Fig. 2) according to the protocol of preparation of these cavities, in particular for all ceramic inlay restorations, made by milling, using the CAD-CAM technology device CEREC 3D (*Sirona Dental Systems GmbH, Bensheim, Germany*).

Fig. 2. Preparation for Mesial Occlusal Distal (MOD) inlay type.

Fig. 3. The inlay obtained after milling.

Fig. 4. The all ceramic inlay adhesively cemented.

The optical footprint of mesial occlusal distal cavity was followed by its transformation, using CAD software, into a virtual model and processing carvings were made by milling unit, IPS Empress CAD blocks (*Ivoclar Vivadent Schaan, Liechtenstein*) made of ceramic reinforced with leucite (Fig. 3). The inlay was etched with 5 % hydrofluoric acid (*IPS Ceramic Etching Gel, Ivoclar Vivadent*) for 20 seconds, followed by a thorough rinse with water-spray and dried with air spray. Monobond S silane (*Ivoclar Vivadent Schaan, Liechtenstein*) is applied by brush and allowed to react for 60 seconds and then dried by air-jet.

The Multilink Primer A and Primer B (*Ivoclar Vivadent, Schaan, Liechtenstein*) were mixed and applied, for 15 seconds, with a brush firstly on the enamel, then on the dentin for another 15 seconds, while the excess is air-jet dispersed. From each side of the tooth a light-curing was applied, using the Bluephase curing lamp, for two seconds.

All three groups were subjected to thermo-cycling by successive immersion in water at approximately 5 and 55 °C during 500 cycles using the LTC device thermo-cycling 100 (*LAM Technologies, Florence, Italy*). The force of 100 N required for occlusal demands, in the case of the groups B 90 and C 45, was generated by the "Equipment for Testing the Cohesion of Mixed Dental Materials" (Fig. 5). The down force was transferred to the tooth *via* a rod fixed in the device that comes in contact with the occlusal surface of the tooth. For fixing the teeth into the testing device that generates the forces of cohesion, a clamping system was imagined, as shown in Fig. 6.
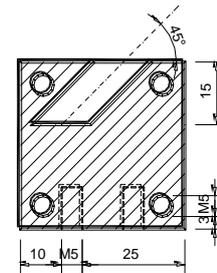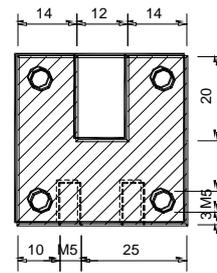

Fig. 6. Schematic representation of the lateral view of the parts for vertical (up) and at 45 degrees (down) attachment of the tooth.

For one track, the vector of the down force, generated by the "Equipment for Testing the Cohesion of Mixed Dental Materials", is perpendicular to the occlusal surface of the tooth, while for the other one, the down force vector makes an angle of 45 degrees with the vertical tooth axis (Fig. 7). After screwing the clamping piece with the tooth fixed inside (at the device to test the cohesion), the push rod is modelled as a lowered cusp until contact with the occlusal surface of the tooth is realised. The device is then turned on and set the applied force on the tooth (Figure 8). The down force control was achieved by means of a measuring circuit. This circuit consists of a half-bridge of pressure transducers, an amplifier to measure the role of the carrier frequency of transducers, half-bridge AC power and highlights the imbalance half-bridge signal converter designed to convert voltage variation signal received by a digital computer. The data were graphically displayed in real time (Fig. 9) by adequate software.
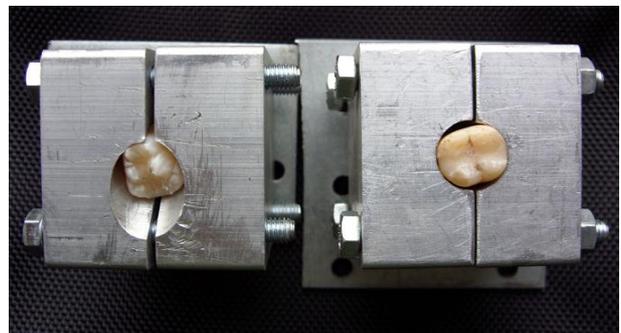

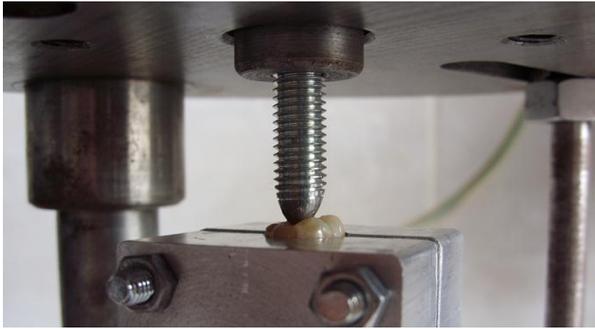*Fig. 7. Teeth set in clamping pieces.*

Fig. 5. The device to test the cohesion.

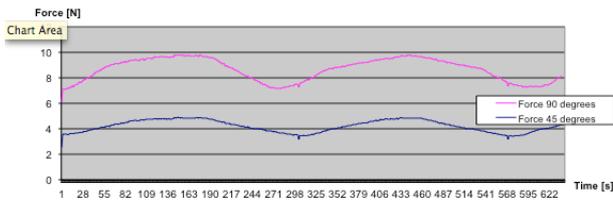*Fig. 8. The tooth fixed to be subjected to forces.*

Fig. 9. Variation of force with time during testing.

After mechanical stress, the teeth were embedded in Citofix amorphous resin then the metallographic samples were prepared for analysis by scanning electron microscopy (SEM). After the first SEM analysis, the samples were thinned to 1.5 mm and polished to identify any other areas of the adhesive interface. Using this procedure, three teeth for each group have been polished obtaining a number of 15 areas of the adhesive interface that were separately investigated. The electron microscope used was a type ESEM XL-30-ESEM TMP, "Politehnica" University of Bucharest.

3. Results and discussion

In order to obtain accurate results and to avoid additional influence of metal covering, for the analysis of the samples, the model Low-Vac (at a pressure of 0.7 Torr) was used. For this current presentation the most representative images (in the inset at low magnification and the details in main images) were chosen.
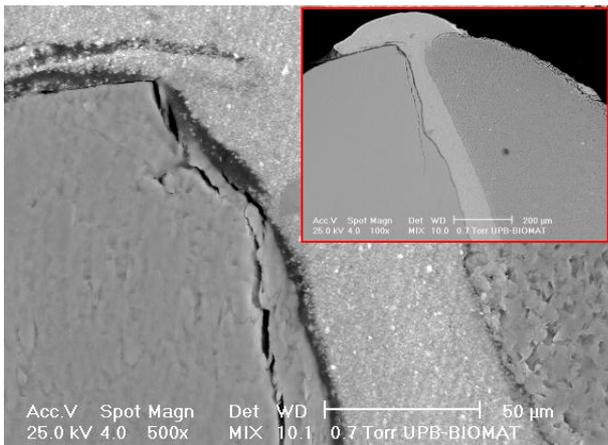

Fig. 10. Micro cracks in the enamel: appearance of incomplete enamel cracks caused by contraction during polymerization.
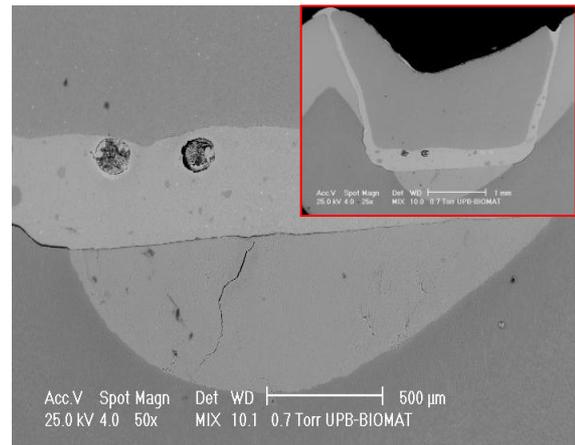

Fig. 11. Micro crack located in a portion of remaining enamel in the pulp wall caused by contraction during polymerization.
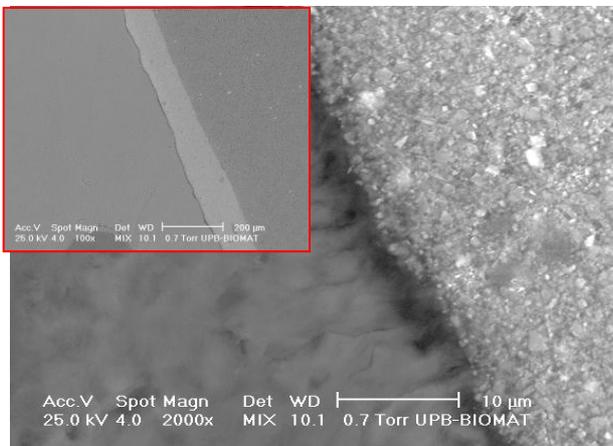

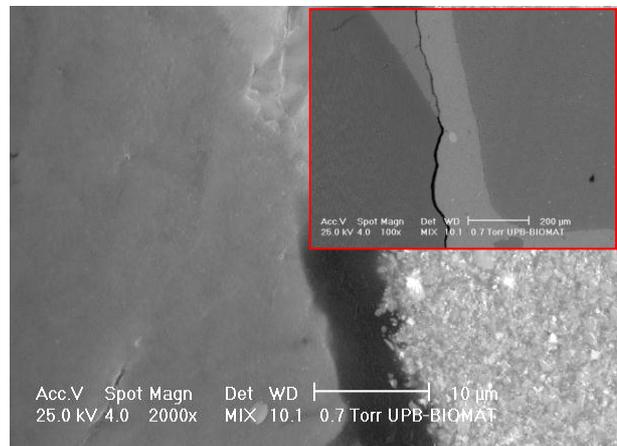*Fig. 12. Interface at the vertical wall of the cavity.*

Fig. 13. Interface at the angle between vertical and the pulp wall cavity.
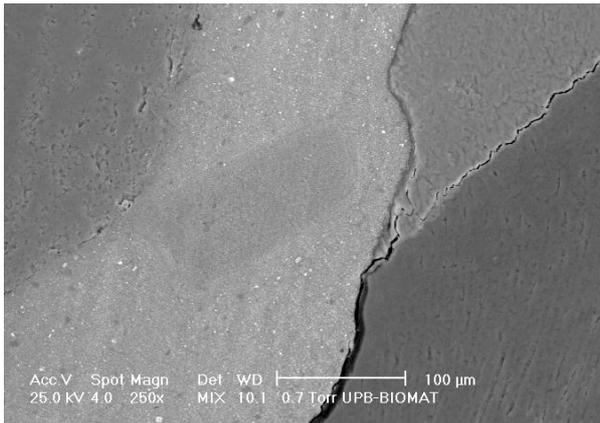
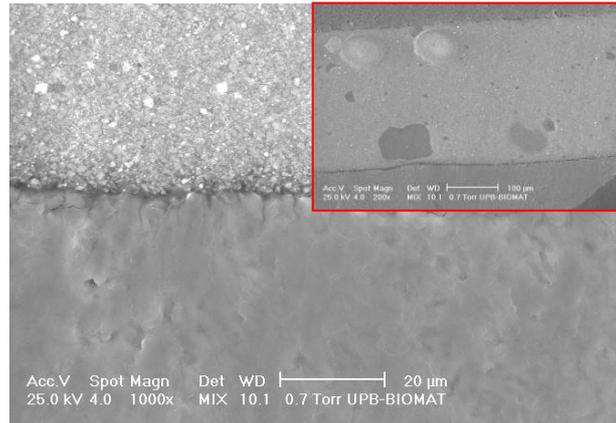*Fig. 14. Interface at the angle formed by the vertical and the pulp wall.*

Fig. 15. Interface at the pulp wall - even crossing area between the ceramics, resin cement and enamel.
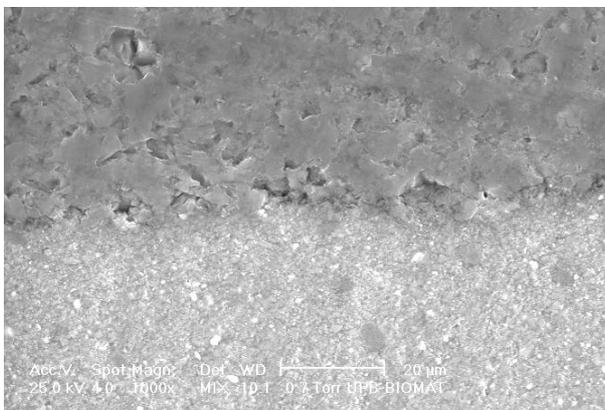

Fig. 16. Ceramic-resin cement interface.

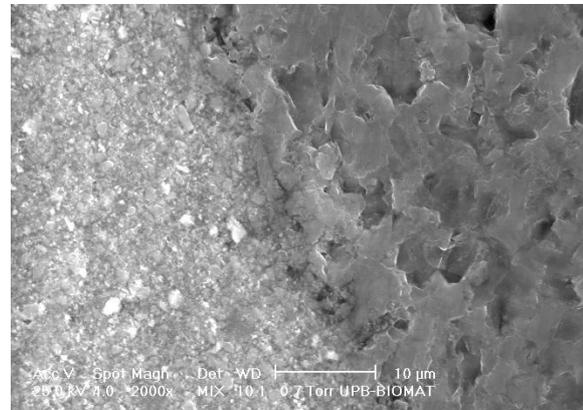*Fig. 17. Dentin-resin cement interface.*

4. Discussion

The aim of this work was to identifyinterface tooth-all ceramic inlay after subjecting the restoring teeth to mechanical and thermal stresses. In all cases of adhesive interface ceramic-resin cement, the adhesive bond showed no micro cracks connection or separation. The adhesive interface of resin cement-dentin shows a high degree of homogeneity and uniformity, with approximately 10 μm thick hybrid layer and absence of gaps or areas of dehiscence. No statistically significant differences between study groups B 90, C 45 and the control group (A 0) were observed. It was observed some incomplete enamel micro cracks of very small size (less than 1 μm), that can be attributed to mechanical stresses caused by contraction during polymerization. In addition, areas of dehiscence at the adhesive interface dentin-resin cement, with a reduced size, under 5 μm, were identified that can be also due to polymerization shrinkage of resin cement. Statistically, both micro cracks and dehiscence have occurred randomly, without a significant representation in the studied groups. The parameters used for requesting the samples were similar to those found in similar studies in the literature.

The 100 N force applied perpendicular to the occlusal surface and to an angle of 45 degrees is comparable to that used in other adhesion studies [37-39] and is relevant to the normal requirements existing in the oral cavity. It is worth mentioning that the physiological demands to whom are subjected the teeth do not follow a vertical path. This occurs only where there is a movement of maximum intercuspal position and even in this situation there is a decomposition of the vertical component caused by the implantation in different zones of the maxillary bone. Lateral movements and propulsion of the lower jaw are mediated by masticatory units, and subjected to forces that no longer act to an angle of 90 degrees, but to other angles. It is for this reason that we have chosen a group of sample, which was loaded to an angle of 45 degrees, considering that this is a conclusive value for occlusal force vertical decomposition. In this case, the resultant force will be smaller than the initial down force. The method of applying force using the "Equipment for Testing the Cohesion of Mixed Dental Materials [7, 46] has the advantage of ensuring a reproducible and controllable applied values due to the precision of this equipment. The sample loading was done progressively, starting from 0 up to 100 N and 70 N, respectively.

For each sample we have chosen a total of 100 cycles of pressure, maintained for 2 seconds trying thus to simulate the conditions of parafunctional chewing process. In order to simulate the physiological conditions in the oral cavity and attain the thermal depreciation of this interface, a thermo-cycling was performed (at 5/55 °C, 500 cycles) before loading the samples.

The obtained results have a high reliability, in terms of strict compliance with ISO/TS 11405:2003 (E) "Dental materials-The Adhesion Testing to Tooth Structure" in collecting teeth and through tooth common steps applicable to each sample to achieve steps that would generate some errors or artefacts. From this point of view, the errors and artefacts would be present in all samples studied and thus become irrelevant statistical errors.

5. Conclusions

The most important experimental issues of this work are related to the presence of micro cracks, gaps, and dehiscence on the two adhesive interfaces: ceramics-resin cement and resin cement-dental structure. The 100 N force, applied both perpendicular to the occlusal surface and to an angle of 45 degrees, with relevant value for existing applications in the oral cavity, did not cause detectable changes in tooth adhesive interface.

The Multilink Automix resin cement is able to achieve adequate adhesion to both dentin, enamel and leucite reinforced ceramics used at inlay processing by performing the CAD-CAM milling. Using the self-etch Multilink Primer one can generate a homogeneous hybrid layer and a uniform thickness of 10 μm.

The material contraction during polymerization may generate some mechanical stress translated by the appearance of micro cracks and small areas of dehiscence located on the interface between resin, cement, and dentin, which suggests a higher value of adhesive force ceramic-resin cement, than cement and resin-dentin. As a corollary, the cohesive force of cement is greater than its adhesion strength.

The obtained inlays, using the device CEREC 3, presents an optimum adaptation to the cavity walls with a uniformly space tooth-inlay, at low dimensional values. The MOD inlay teeth presented an adequate resistance for the demands of vertical and paraxial strength-activity under biting without pathological changes. Nevertheless, the fact that we only considered 15 teeth prepared and restored can be considered a limit of this study. We mitigated this risk by analysing 45 sections with adhesive interfaces obtained by successive stages of metallographic polishing samples and SEM analysis.

The results of this study open perspectives for further studies, on a larger group of teeth, as well as on cyclic fatigue strength of adhesive interface. The method of obtaining the restorations was intended to be in the limits of the preparation protocols and of mechanical properties. Therefore a computerized method that uses industrially produced ceramic blocks have to be chosen.

References

[1] M. Gresnigt, M. Ozcan, W. Kalk, Euro. J. of Esth. Dentistry, **6**(3), 298 (2011).

[2] A. Fabianelli, P. Pollington, F. Papacchini, J Dent, **38**, 39 (2010).

[3] R. Frankenberger, U. Lohbauera, B. Rainer, S. Nikolaenkob, M. Naumannc, Dent Mater., **24**, 185 (2008).

[4] C. S. Mota, F. F. Demarco, G. B. Camacho, J. M. Powers, J Adhes Dent., **5**, 63 (2003).

[5] A. Rajaput, I. Ataide, R. Lambor, J. Monteiro, M. Tar, N. Wadhawan, Eur. J. Esthet. Dent., **5**, 398 (2010).

[6] A. Mehl, K. Manfred, Cosm. Dent., **2**, 51 (2007).

[7] S. Pelekanos, M. Koumanou, S-O. Koutayas, S. Zineslis, G. Eliades, Euro. J. of Esth. Dentistry, **4**(3), 278 (2009).

[8] A. F. Quintas, F. Oliveira, M. A. Bottino, J. Prosthet Dent, **92**, 250 (2004).

[9] J. A. Sorsen, J Prosthet Dent, **64**, 636 (1990).

[10] G. J. Christensen, J. Prosthet Dent, **16**, 297 (1996).

[11] S. Reich, M. Wichmann, E. Nkenke, P. Proeschel, Eur. J. Oral. Sci., **113**, 117 (2005).

[12] K. W. Boening, B. H. Wolf, A. E. Schnmidt, K. Kastner, M. H. Walter, J. Prosthet. Dent., **84**, 419 (2000).

[13] K. B. May, M. M. Rusell, M. E. Razzoog, B. R. Lang, J. Prosthet. Dent., **80**, 394 (1998).

[14] R. G. Luthardt, G. Bornemann, S. Lemelson, M. H. Walter, A. Huls, Int. J. Prosthodont, **17**, 680 (2004).

[15] S. I. Voicu, A. C. Nechifor, B. Serban, G. Nechifor, M. Miculescu, J. Optoelectron. Adv. Mater., **9**(11), 3423 (2007).

[16] J. R. Holmes, W. D. Sulick, G. A. Holland, S. C. Bayne, J. Prosthet. Dent., **67**, 594 (1992).

[17] F. Vahidi, E. T. Egloff, F. V. Panno, J. Prosthet. Dent., **66**, 426 (1991).

[18] F. Miculescu, M. Miculescu, L. T. Ciocan, A. Ernuteanu, I. Antoniac, I. Pencea, E. Matei, Digest J. of Nanom. and Biostructures, **6**(3), 1117 (2011).

[19] F. Miculescu, L. T. Ciocan, M. Miculescu, A. Ernuteanu, Digest J. of Nanom. and Biostructures **6**(1), 225 (2011).

[20] L. T. Ciocan, F. Miculescu, M. Miculescu, I. Patrascu, Rom. J. of Morphology and Embryology, **51**(1), 117 (2010).

[21] I. Patrascu, L. T. Ciocan, B. Galbinasu, B. Iordache, Euro. Cells and Materials, **9**(1), 37 (2005).

[22] L. T. Ciocan, B. Iordache, B. Galbinasu, I. Patrascu, Invention brevet no. 122801: "Equipment for Testing the Cohesion of Mixed Dental Materials", State Office for Inventions and Trademarks, 2012-02-26.

[23] Techcal Specification ISO/TS 11405:2003(E), "Dental materials – Testing of adhesion to tooth structure", second edition, 2003-02-01.

*Corresponding author: bogdan.galbinasu@yahoo.com