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INTEGRATED APPROACH TO THE STUDY OF BIODEGRADATION OF COMPOSITE MATERIALS FOR THE RESTORATION OF HARD DENTAL TISSUES

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КОМПЛЕКСНЫЙ ПОДХОД К ИЗУЧЕНИЮ БИОДЕГРАДАЦИИ КОМПОЗИТНЫХ МАТЕРИАЛОВ ДЛЯ ВОССТАНОВЛЕНИЯ ТВЕРДЫХ ТКАНЕЙ ЗУБОВ

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Objective. To study the stress-strain state of demineralized tooth enamel after its impregnation with a low-viscosity composite in a long-term period of functioning in the oral cavity in a series of equivalent cyclic tests; to study the correlation of the experimental results with clinical observations of the treatment of enamel caries using a modified infiltration technique.
Materials and methods. The parameters of laser holographic interferometry and atomic force microscopy, reflecting the timing, type and severity of deformation defects in samples of demineralized tooth enamel were analyzed in a series of equivalent cyclic tests. During the experiment the samples underwent classical infiltration with composite using ICON technology (main group) and a 2-stage enamel caries treatment technique, including carrying out a time-modified conditioning of classical infiltration and the final stage (stages) of enamel coating with a bioactive hybrid glass ionomer (comparison group).
Results. The first signs of the deformation defects generation in demineralized enamel filtered on the classical method (the appearance of perifocal microcracks) were recorded in 11.2 % of the tested samples after $0.9 \cdot 10^6$ cycles (equivalent to 9 months of functioning of a tooth with treated enamel caries in the oral cavity), during further cyclic tests the percentage of samples with local defects increased progressively. Signs of a violation of the stress-strain state in the enamel after combined treatment (samples of the comparison group) were revealed in cycles corresponding to 2–3 years of being in an artificial environment of the oral cavity. Experimental data correlated with the clinical observations both in time and frequency of complications (secondary/recurrent caries) in the long term after treatment of enamel caries by various methods.
Conclusions. In the process of equivalent cyclic tests, heterogeneous elastic-plastic deformations develop in the thickness of a section of demineralized enamel impregnated with a flowing composite and along its perifocal zones, most pronounced at the interface between intact and treated enamel, which leads to the development of local defects, delaminations, cracks, and destruction of the structurally heterogeneous environment of tooth enamel. According to the results of the comparative analysis, the most favorable results in terms of the timing of development, frequency and severity of deformation defects were obtained in samples of demineralized enamel after its treatment using a modified caries infiltration technique followed by coating with a bioactive hybrid glass ionomer.
Keywords. Composite materials, aesthetic and functional restoration, enamel caries, stress-strain state, equivalent cyclic tests.

Цель. В серии эквивалентных циклических испытаний изучить напряженно-деформированное состояние деминерализованной эмали зуба после ее импрегнации композитом низкой вязкости в сроки, эквивалентные длительному периоду функционирования в полости рта; соотнести результаты экспериментов с клиническими наблюдениями лечения кариеса эмали по модифицированной инфильтрационной методике.
Материалы и методы. Проанализированы параметры лазерной голографической интерферометрии и атомно-силовой микроскопии, отражающие сроки появления, тип и выраженность деформационных дефектов в образцах деминерализованной эмали зубов, на которых в процессе эксперимента воспроизведена классическая инфильтрация композитом по технологии ICON (основная группа) и двухэтапная методика лечения кариеса эмали, включающая проведение модифицированной по времени кондиционирования классической инфильтрации и заключительный этап (этапы) покрытия эмали биоактивным гибридным стеклоиономером (группа сравнения).
Результаты. Первые признаки генерации деформационных дефектов в проинфильтрированной по классической методике деминерализованной эмали (появление перифокальных микротрещин) зафиксированы у 11,2 % тестируемых образцов через $0{,}9 \cdot 10^6$ циклов (эквивалент 9 месяцев функционирования зуба с пролеченным кариесом эмали в полости рта), в ходе дальнейших цикловых испытаний процент образцов с локальными дефектами прогрессивно нарастал. Признаки нарушения напряженно-деформированного состояния в эмали после комбинированного лечения (образцы группы сравнения) выявлены в циклах, соответствующих 2–3 годам нахождения в условиях искусственной среды полости рта. Экспериментальные данные коррелировали с клиническими наблюдениями по времени и частоте проявления осложнений (вторичного / рецидивного кариеса) в отдаленные сроки после лечения кариеса эмали разными методами.

Выводы. В процессе эквивалентных циклических испытаний в толще участка деминерализованной эмали, проимпрегнированной текучим композитом, и по его перифокальным зонам развиваются неоднородные упругопластические деформации, наиболее выраженные на границах раздела интактной и пролеченной эмали, что приводит к развитию локальных дефектов, расслоений, трещин, разрушению структурно-неоднородной среды эмали зуба. По итогам сравнительного анализа наиболее благоприятные результаты в части сроков развития, частоты и степени выраженности деформационных дефектов получены у образцов деминерализованной эмали после её лечения по модифицированной методике кариес-инфильтрации с последующим покрытием биоактивным гибридным стеклоиономером.
Ключевые слова. Композитные материалы, эстетико-функциональная реставрация, кариес эмали, напряженно-деформированное состояние, эквивалентные циклические испытания.

INTRODUCTION

Aesthetic-functional restoration (AFR) of lost dental tissue (dentine, enamel, cement) is the most common treatment procedure in an outpatient dentistry clinic. Modern composite materials (CM) play a key role in the AFR of teeth, the requirements for which, despite their diversity in the dental market, are constantly increasing.

Modern dental materials science and conservative preventive dentistry highlight the following promising directions for the development of new composite materials for AFR: bioactive composites with antimicrobial/remineralizing ingredients; strengthened composites, including fiber-reinforced ones; fast-hardening composites; and CM with higher adhesive properties and increased crack resistance to ensure operational survivability of the material in the long term after AFR [1; 2]. An optimal balance of strength and viscosity properties enables improving the adhesion/marginal adaptation of CM to hard dental tissues (HDT) and its handling characteristics and ensuring the stability of the obtained AFR results over time. A modern trend in dental materials science regarding AFR are technologies for improving the physical and mechanical properties of CM by thermal, vibration, or mechanical activation of the material or their combination [3; 4]. Furthermore, modernization of AFR is progressing along the path of creating new more advanced adhesive and polymerization systems and technologies for conditioning HDT before filling [5; 6]. Notably, CMs are traditionally in demand for replacing actual dentin or cement defects. Experimental and clinical studies [7] indicate that a separate group of unfilled, high-flow composites with low viscosity (low-viscosity resin) is used for ultraconservative treatment of enamel focal demineralization, used in combination with traditional restorative treatment or teeth whitening [8; 9]. As an example of such CM with low-viscosity properties, an infiltrant is considered a classic and an improved variant of caries infiltration (C–I) using ICON technology (DMG, Germany), which in experimental and clinical conditions has demonstrated advantages compared to traditional remineralizing therapy [10].

The long-term functioning conditions of CM in the oral cavity, which determine the durability of restoration/infiltration of HDT, are largely affected by the development of the stress–strain state at the border of CM and tooth tissues (enamel, dentin, cement). The study of the patterns of multifactorial (mechanical, chemical, biomechanical, thermodynamic) biodegradation of infiltrating CM, simulating its behavior in dental tissues during the long-term functioning of the composite–enamel binary system in the oral cavity, is theoretically significant and practically justified; however, such studies are few. Testing equipment manufactured in other countries (Willytec, MTC-simulator, etc.) designed for these purposes are installed in several large world-class biomaterials science centers. Russian developments of such simulation systems and equipment are warranted, their bringing to ISO and GOST standards for qualified testing of new dental materials (including CM) and treatment and prophylactic technologies.

This study aimed to analyze, in a series of equivalent cyclic tests, the stress–strain state of demineralized tooth enamel after its impregnation with a low-viscosity composite in a long period of functioning in the oral cavity and correlate the experiment results with clinical cases of the treatment of enamel caries using a modified infiltration technique.

MATERIALS AND METHODS

Experimental and clinical studies were approved by the local ethical committee and conducted at the Department of Therapeutic Dentistry and Propaedeutics of Dental Diseases, E.A. Wagner Perm State Medical University of the Ministry of Health of Russia, and Department of General Physics, Perm State National Research University, in specialized laboratories of "ODK-STAR" (Perm). Forty premolars removed with an intact crown and root according to indications were selected, on which enamel caries was modeled using the original method[1] (ICDAS code 1, 2), confirming its compliance with the data of microcomputed tomography, atomic force microscopy (AFM), and scanning electron microscopy. The teeth were randomly distributed into two equal groups: the main group (the classical C–I technique was modeled on teeth with artificial caries) and comparison group, where artificial enamel caries on teeth was "treated" using a two-stage method, including the stages of impregnation of the demineralization focus with a composite with exposure to enamel conditioning increased to 4 minutes and of coating the impregnated enamel with a layer of bioactive hybrid glass ionomer[2].

To study the patterns of biodegradation of demineralized tooth enamel im-

[1] O.S. Gileva, M.A. Muravyova, E.S. Gileva, V.A. Valtsifer, and A.I. Nechaev, A Method for Modeling a Focus of Tooth Enamel Demineralization, Patent for invention RU 2503067 C1, 12/27/2013, application no. 2012147965/14; dated November 12, 2012.

[2] O.S. Gileva, M.A. Shakulya, A.D. Levitskaya, E.S. Syutkina, and E.V. Serebrennikova, Method for Treating Focal Demineralization of Tooth Enamel, Patent for invention RU 2571334 C1, 12/20/2015, application no. 2014146961/14 dated November 21, 2014.

pregnated with low-viscosity CM using the classical and combined C–I methods we developed, the study used a new Russian-made simulation complex of qualification equipment for conducting tribological tests of dental materials and technologies, developed jointly with engineering specialists[3].

In measuring nanodisplacements in infiltrated enamel and its microdeformations during equivalent cyclic tests of materials, the laser holographic interferometry (LHI) method with a counter-directional action scheme with double exposure was used, synchronized with the analysis of the enamel surface using AFM and calculation of its quantitative parameters to identify microcracks, ruptures, etc. in the material interface zone [11]. The hologram after each cycle of cyclic tests was compared to the original.

RESULTS AND DISCUSSION

Based on the results of cyclic tests of teeth of the main group, where enamel caries was treated according to the classical C-I method following manufacturer recommendations, after 0.9 x 10^6 cycles, equivalent to 9 months of functioning of the treated tooth as part of the dentofacial system, microcracks with branches (thinning) were determined in 11.2 % of cases using the LHI method synchronously with AFM analysis of the enamel surface at the border of healthy and impregnated enamel. After $1.2 \cdot 10^6$ cycles (equivalent to 1 year post-treatment), the proportion of detected cracks in the enamel increased to 55.5 %. At similar stages of equivalent cyclic tests, corresponding to 9- and 12-month follow-ups, on the surface of the tested teeth samples treated under experimental conditions according to the original two-stage method (impregnation with a low-viscosity composite followed by coating with a hybrid glass ionomer), microcracks or other defects were not detected in the cases, and the marginal adhesion of the impregnation zone to the apparently intact enamel was not disturbed. Moreover, after 60 minutes of testing (equivalent to a 3-year "operation" in the oral cavity of enamel impregnated with a composite), the first signs of microdeformation of its surface layers (an increase in the curvature of interference fringes) were observed; however, even at this stage, delaminations and microcracks in border areas of enamel were not noted. Disturbances in the deformation of enamel infiltrated with a composite using the classical method, identified under experimental conditions, corresponded to the cases of development of secondary caries, visible

[3] A.D. Levitskaya, O.S. Gileva, A Device for Producing a Damping Replica of the Occlusal Surface of a Tooth *in vitro* for Conducting Cyclic Tests of the Tooth for Axial Compression. Utility model patent RU 191943 U1, 08/28/2019. Application no. 2018145620 dated December 20, 2018. A.D. Levitskaya, O.S. Gileva, A Device for Bonding a Tooth *in vitro* to Conduct Cyclic Testing of a Tooth for Axial Compression. Utility model patent RU 190383 U1, 06/28/2019, application no. 2018145617 dated December 20, 2018. A.D. Levitskaya, O.S. Gileva, *In vitro* Tooth Centering Device for Cyclic Axial Compression Testing of Teeth. Utility model patent RU 191894 U1, 08/26/2019 application no. 2018145619 dated December 20, 2018. A.D. Levitskaya, O.S. Gileva, A Device for Performing Cyclic Tests of Teeth *in vitro* for Axial Compression, Patent for invention RU 2704208 C1, 10/24/2019, application no. 2018145123 dated December 18, 2018.

linear defects, and violations of the marginal adaptation of the composite in the thickness of the enamel in the long-term follow-up in 23.9 % of 67 patients with focal demineralization of enamel (12.18 months post-treatment). The experimental assessment results of the "functional survivability" of a special structurally heterogeneous environment (demineralized enamel impregnated with a composite, laminated with a layer of bioactive glass ionomer) in the tested samples of the comparison group corresponded to the data of clinical cases (no signs of secondary/recurrent caries for 1.5 years after treatment) and isolated cases of the appearance of defects (violations of border adaptation) on the enamel surface after 18 months of monitoring.

Notably, based on the test results, the Russian-made simulation system of equivalent cyclic tests of tested samples used in this study has demonstrated high qualification characteristics, which is characterized by the originality of design solutions, methods for assessing the performance characteristics of tested samples, approaches to regulating the rate of load increase, choice of temperature conditions, and methods of maintaining liquid homeostasis with artificial saliva of the original composition, which bring *in vitro* experiments as close as possible to the real conditions of long-term functioning of teeth in the oral cavity.

The analysis of the stress–strain state in the process of tribological tests is traditionally performed by methods of strain gauging, photoelasticity, and digital dynamic spectrum photography or using mathematical models. The use of the LHI method for these purposes was demonstrated by the novelty of the approach and good results regarding objectivity and reproducibility of the results obtained and clarity of their presentation. Thus, the physical knowledge obtained from the results of equivalent clinical trials enabled for determining the critical periods, "foci," and types of deformations in demineralized enamel before and after various treatment techniques. New data on the patterns of biodegradation of composite material in the composition of demineralized enamel were obtained, which confirmed the correctness of the chosen ultraconservative approach to its treatment based on impregnation of the affected areas of enamel with a flowable composite with prolonged preliminary conditioning stage and subsequent finishing lamination of infiltrated and perifocal enamel with a bioactive glass ionomer.

CONCLUSIONS

1. Equivalent cyclic tests of samples of teeth with enamel caries treated using the classical method of caries infiltration show the clinically high (23.9 %) incidence of complications in the form of secondary caries 1–1.5 years after treatment. This is experimentally confirmed by the appearance of microcracks in 55.5 % of the tested samples at a time period corresponding to 12 months of functioning in the oral cavity.

2. The advantages of a two-stage (impregnation with a composite with a prolonged conditioning stage and subsequent coating with a bioactive glass ionomer) method of treating enamel caries are confirmed by experimental studies (the appearance of the first signs of microdeformations of the enamel surface after 18-month follow-up).

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