

518. Fatigue stress resistance of some composite materials for dental fillings

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Abstract. The paper investigates fatigue stress resistance of some originally made composite materials based on Bis-GMA resin with fluoridated glass and YbF₃. The material was used to fill in the cavities in teeth extracted for orthodontic reasons. For laboratory tests a mastication simulator was used. The tooth samples were placed in special holders with resin and exposed to cyclic mechanical loads (100 000 cycles) in order to assess their resistance to fatigue stress. The influence of repeatable mechanical loads on the teeth and the structure of the material were investigated. Additionally, the impact of cyclic loads on fluoride release from the composite materials was estimated.

Keywords: fatigue stress, fluoride, release, dental fillings, composite materials

Introduction

Due to their superior mechanical and aesthetic properties, dental composite materials are commonly used as dental fillings. Numerous investigations concerning physicochemical, mechanical and tribological properties of dental composite materials have been intensively conducted [1,2]. In paper [3] an *in vitro* oral wear simulator was used to compare the susceptibility of different classes of dental composites to marginal breakdown. The reported results are in general agreement with clinical studies stating that microfilled composites show greater marginal degradation than other fillings of this type. On the other hand, microfilled composites show much higher resistance to contact fatigue than other materials, like glass ionomers [4]. Fatigue behaviour of resin composites was analyzed under different environment parameters [5]. It was found that fatigue crack propagation decreased under aqueous conditions and that fatigue cracks detoured around inorganic macro-fillers, but penetrated the organic ones.

In the literature available on the subject there is not much information concerning fatigue properties of composite dental fillings. The influence of cyclic loads on forming the gap margin between dental fillings and hard tooth tissues is not specified. The mechanism of crack propagation in the composite material and hard tooth tissues directly adhering to the material is difficult to characterize [6, 7]. Dental filling undergoes mechanical and thermal loads. The process of mastication generates the complicated system of forces in the human oral cavity [8, 6]. Considerable stresses are additionally intensified by stresses resulting from the cyclical changes of a tooth thermal state [9]. Some results on the marginal fissure confirm that all kinds

of dental fillings are not perfectly leakproof on a sense of marginal fissure [10]. Very important are tests of qualitative and quantitative evaluation of both marginal fissure and crack of hard tissues and filling materials.

Krejci at. all [11, 12, 13] presented design and principles of operation of a computer controlled mastication simulator which is able to realize mechanical and thermal loads taking place in a human oral cavity. Conducted research tests which proved that the device fulfilled requirements concerning. The authors concluded that mastication simulator can be successfully used for evaluation of durability of human teeth with dental fillings. However, it should be noticed that designed mastication simulator is able to produce thermal and mechanical loads without simulation of mechanical load cycles exactly the same as in the oral cavity.

However, due to non-uniformity of the teeth structure and specific environment in the oral cavity, it is not possible to conduct research on samples different from real human teeth. Above arguments brought authors to design of test stand, which will allow for examination of dental fillings in real human teeth exposed to mechanical and thermal conditions the same as in the oral cavity.

In the paper the influence of cyclic loads on the structure of composite materials for dental fillings and the width analysis of the gap margin between the material and the tooth were investigated.

Materials and methods

In the research two ceramic-polymer microfilled composites containing powder fluoride sources were tested. The materials were made of light-cured organic matrix (40 vol. %), fluoridated powder filler glass (J-20 symbol) and an additional fluoride source – YbF₃. The organic matrix was composed of a resin Bis-GMA {2,2-bis[p-(2'-hydroxy-3'-methacryloxypropoxy)-phenyl] propane}, TEGDMA {tri(ethylene glycol) dimethacrylate}, DEA-EMA {2-(diethylamino)ethyl methacrylate}, BHT, CQ {D,L-Camphorquinone} and a photoinitiator. All the components were provided by the Sigma-Aldrich Company.

The fluoridated glass (J-20) consisted of different oxides and fluoride ions: SiO₂-P₂O₅-Al₂O₃-BaO-SrO-Na₂O-F⁻. The surface of the inorganic filler was treated with a functional silane. The aim of the silanization process was to absorb the active silane groups on the powder surface at vacuum evaporator. After the silanisation process the reactive silane was combined with the inorganic filler so that it could co-polymerize with the polymer network. Qualitative and quantitative oxides built-up of J-20 glass ceramic are characterized by various particle sizes (1-3µm with nanoparticles). Ytterbium fluoride powder is characterized by irregular particles measuring about 1 µm.

Table 1. Composition of the composite materials

Materials' symbol	Glass ceramic [J-20, vol. %]	YbF ₃ [vol. %]
K1	60	-
K2	50	10

All the organic constituents were weighed into a porcelain mortar and mixed carefully until a uniform polymer paste was obtained. Next the filler particles were added to an organic matrix that was previously prepared. The organic-inorganic mixtures were then homogenized in the porcelain mortar for 10 minutes. The composition of the materials is shown in Table 1.

Fatigue tests

In the study, human molars and premolars extracted due to orthodontic reasons were used. The cavities with deep of 3 mm were filled with composite materials. There was a contact between the material and enamel and dentin. The composite material was applied in the layers, each of them 2 mm thick, and was polymerized by halogen lamp for 40 seconds. Then, teeth samples were placed in special holders and exposed to cyclic mechanical loads in contact with buffer solution with pH=6.8.

Degradation tests of composite materials were estimated using special mastication simulator. The tester simulate mechanical loads that taking place during natural mastication process to the highest degree. The simulator enable to design the parameters of really operating conditions and estimate the course of wear process and defects of dental fillings relate to clinical conditions.



Fig. 1. View of mastication simulator

The mastication simulator (Fig.1) consists of a pneumatic pressing mechanism and biaxial plotter driven by stepper motors. This solution provides relatively small inertia forces of moving parts and full flexibility of forces and movement path selection. The upper sample is fixed to a slider mechanism which can realize up and down movements while the lower sample is fixed to the biaxial plotter simulating submaxilla motions.

In order to provide force field independent on geometry of tested samples, the simulator is equipped with a measurement system which allows for determination of cooperating samples reaction force vector. The base of plotter mechanism is placed on a set of vibration insulators, distinguished by high stiffness in vertical direction and relatively low stiffness in horizontal direction (Fig. 2 and Fig. 3). The plotter position is fixed with the use of three force transducers. In order to measure only a shearing force without influence of appearing torques the design resembles a rocker arm suspension. Electrical signals from two parallel force transducers are summarized providing information about force in X direction while third transducer gives Y component signal.

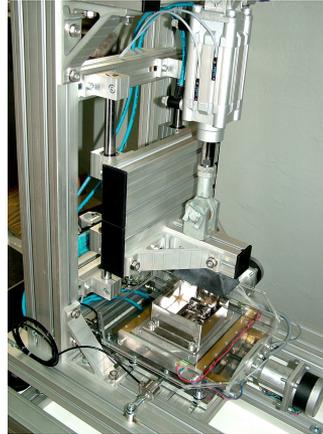
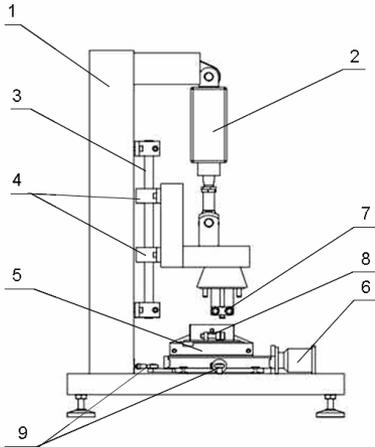


Fig. 2. The diagram of mechanical module of the simulator of mastication: 1 - body, 2 - pneumatic motor operator, 3 - guides, 4 - linear bearings, 5- plotter, 6-stepper engine, 7- upper specimen, 8 - lower specimen, 9 - set of measurement tensometers [9]

Fig. 3. View of mechanical module of simulator [6]

Measurement of forces is held constantly during operation of the simulator. During realization of the experiments the data is recorded for assessment of forces distribution. It permits introduction of some corrections of lower sample movement path. For control of simulator operation and data recording a personal computer with original software was used. The control software allows for setting of arbitrary lower sample movement path. Apart from this it is possible to introduce statistical unrepeatability of consecutive mastication cycles.

The mastication simulator consists of a pneumatic pressing mechanism and biaxial plotter driven by stepper motors. One of sample is fixed to a slider mechanism which can realize up and down movements. The second sample is fixed to the biaxial plotter simulating submaxilla motions. The scheme of investigated fatigue set-up is presented in Fig.4.

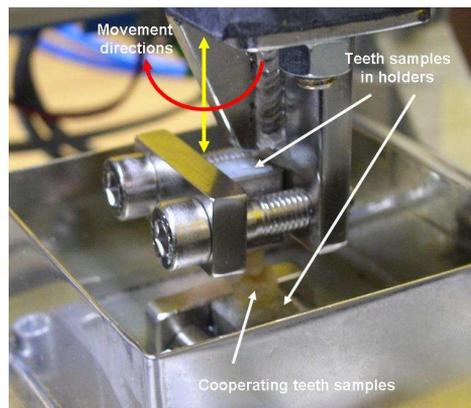


Fig. 4. Teeth samples fixed in the mastication simulator and their cooperation [6]

The simulator is equipped with a measurement system which allows to determine the cooperating samples reaction force vector in order to provide force field independent on geometry of tested samples. After fixed the samples in clamps of mastication simulator, the composite materials relative orientation is set in position typical for physiological conditions. The central point is determined. The next step encloses setting a value of vertical force and programming of lower sample motion path. The path, described by coordinates of eight points, is set to be similar to Bates cycle, which simulates physiological path of submaxilla move. The final verification of sample trajectory is made on the base of reaction forces field analysis.

Fluoride release

The investigations also involved the influence of load and the type of the filler material used on the fluoride release process. During the fatigue tests conducted on the special mastication simulator, teeth with samples of composite materials were immersed in special buffer at pH=6.8, corresponding to the saliva pH. In the process fluoride ions were released into the contact solution. Following the fatigue process, to determine the content of fluoride ions released from the examined materials, the method of direct potentiometry with combined fluoride selective electrode, manufactured by Orion, was used. The tests were performed after the complete process (100 000 cycles) of the fatigue test. Each time 3 specimens of 10 ml of each of the obtained solutions were prepared. TISAB buffer was added to the analyzed solutions before the examination in order to stabilize the pH and eliminate the influence of foreign ions during the test.

Results

The distributions of the cutting forces that were observed between the analyzed samples of K1 and K2 under vertical force of reciprocal pressure at 400 N, are presented in Fig. 5. It can be concluded from the figures that even in the central shorting point, there are significant constituents of cutting forces. It is an effect of differences in the anatomical structure of the teeth, which means that the surfaces of both tested teeth are not perpendicular to the direction of the applied pressure forces.

The distribution of forces and co-ordinates of sample motion are the effect of masticate teeth surface unevenness and the heterogeneity of the system stiffness of the investigated samples and simulator mechanisms. During the tests, 100 000 cycles under vertical load of 40 kilograms were used. The accuracy of positioning was 0.01 mm (1 step). The complete measuring trolley moves in both X and Y-axes were 30 mm. During the tests in both cases the same load conditions were used. Some differences in the operation of the shearing forces during the simulation of the mastication process and programmed trajectory of the co-operating samples of K1 (Fig. 5a) and K2 (Fig. 5b) composites were observed. The differences of results are mainly due to the specificity of the teeth used during the research, i.e. their surface structure, which has a direct impact on the distribution of forces during the fatigue tests.

The extracted teeth were treated by fatigue tests in order to define changes in structure and size of the microcracks between filling and tooth tissues. These investigations enable us to evaluate the fatigue stress resistance of filling-tooth joint and/or the filling material only. From the clinical point of view, the microgaps on the occlusion surface are of the greatest interest. The evaluation of the materials' fatigue resistance at this tooth surface is difficult. Some schools of dentistry recommend putting a filling material's layer on the enamel surface in order to minimize the influence of microgaps on the filling material fatigue.

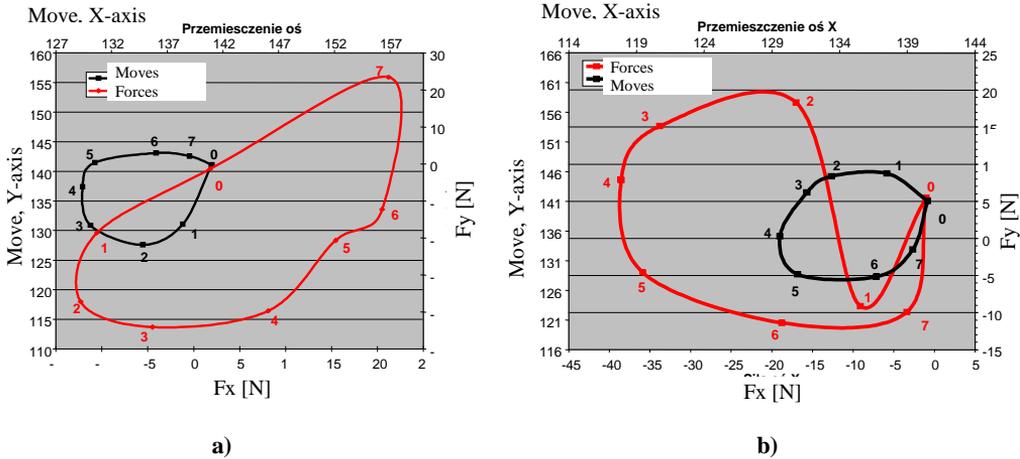


Fig. 5. Distribution of shearing forces (F) and programmed trajectory of the cooperate samples (moves) of composite K1(a) and K2 (b)

In order to observe such changes in both the tooth structure and composite materials some studies were performed using a scanning electron microscope. SEM micrographs presented in Fig. 6 and Fig. 7 show some microcracks in the composite and tooth structure. During the fatigue test phenomena similar to the friction process occurred. Apart of the structure destruction itself, the existence of some particles were observed.

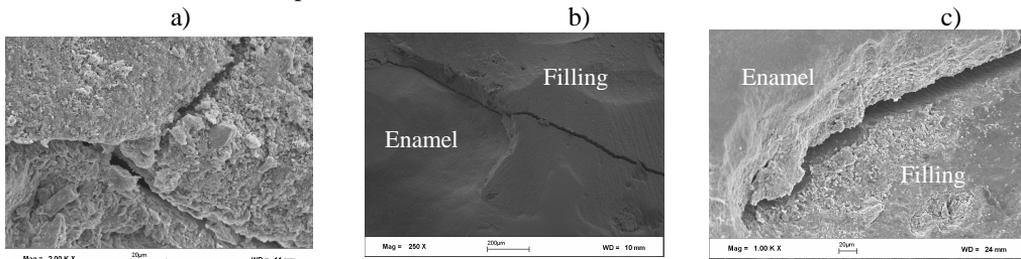


Fig. 6. SEM micrographs of the structure of composite K1 after fatigue test:
 a) material's microcracks; b, c) edge micrographs

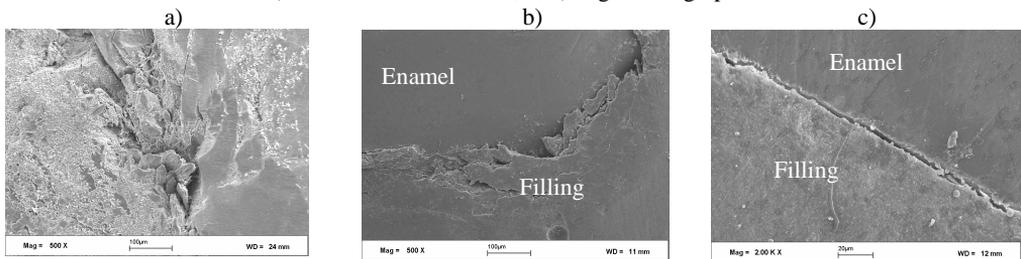


Fig. 7. SEM micrographs of the structure of composite K2 after fatigue test:
 a) material's microcracks; b, c) edge micrographs

Also microgap formation was observed following the fatigue process caused probably by polymerization shrinkage of the composite materials. The width of the microgap was about a few to several dozen micrometers. The application of an additional load increased the width.

As related in the literature, the microgap between the tooth and its filling could reach even 20-100 μm after four years of filling implementation.

There is no evident difference between the two analyzed materials: K1 and K2. One can conclude that ytterbium fluoride has no considerable influence on the structure of the analysed materials.

The first stage of the test consisted in determining the influence of two fluoride sources (fluoridated filler and ytterbium fluoride) on the amount of fluoride release to the buffer solution after 7 days of keeping the samples in agent solution. The second stage of the work concerned the estimation of fluoride release from samples that were earlier subjected to the fatigue test. The results show the amount of fluoride released from 1 mm^2 of composite material free of load and under load influence.

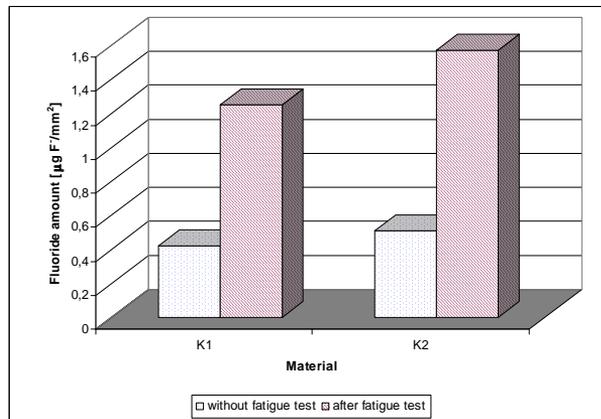


Fig. 8. Influence of loading on fluorine release from composite K1 and K2

Fig. 8 presents the emission fluoride ions from two examined composites under no load and under low impact. The results indicate that the cyclic loads increase the amount of fluoride ion release. The composites loads with no loads applied evidently release much lower fluorine amount (about $0,42\ \mu\text{gF}/\text{mm}^2$ for K1 and $0,51\ \mu\text{gF}/\text{mm}^2$ for K2) in contrast to the materials following the fatigue tests ($1,25\ \mu\text{gF}/\text{mm}^2$ for K1 and $1,57\ \mu\text{gF}/\text{mm}^2$ for K2). Most probably the fatigue changed the internal structure of the composite materials resulting in the formation of some microcracks. The observed discontinuities could be the potential channels of diffusion for fluoride release. It was also observed that the composite with additional fluoride source (material K2 with YbF_3 powder) releases more fluoride ions than the composite containing only fluoridated glass.

Conclusions

Considering the results of the research, the following conclusions may be formulated:

1. On completion of the fatigue tests the width of the microgap between the fillings and the tooth structure was about a few to several dozen micrometers.
2. It can be concluded that ytterbium fluoride had no considerable influence on the resistance of materials' structure to fatigue stress.

3. During the fatigue test, numerous microcracks were formed in the structure of the material and enamel.
4. The release of fluoride ions from the analysed composites increased during the fatigue tests.

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