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Microscopic studies of the degradation processes of dental composite materials

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Abstract

The article addresses the strength of dental composite materials, with particular emphasis on their resistance to compression. This type of loading is predominant in the anterior and posterior chewing teeth of humans. Understanding the mechanisms of initiation and development of internal stresses in materials used for restorative procedures is essential for the informed selection of materials and the further improvement of their properties.

The dental composite materials Estelite Asteria and Estelite Posterior, manufactured by Tokuyama Dental, were used in the experiments. A specimen preparation technique for compression loading was considered, and experimental studies were conducted with the recording of ultimate forces and stresses. The fractured specimens were examined to determine the type of failure. It was established that the plastic component during the deformation process is practically absent, while the load–deformation relationship is quasi-linear.

For further investigation, fragments of the materials were embedded in epoxy resin, after which cubic specimens were prepared. Based on these specimens, metallographic sections were produced for a detailed examination of the mechanisms of damage accumulation and the formation and propagation of microcracks within the material structure.

It was found that the fracture mechanisms and microcrack configuration depend on the size of the inorganic fillers in the composites. It was demonstrated that the avalanche-like loss of structural integrity of a composite material occurs when a certain microcrack density is reached, defined as the total crack length per unit area. At the same time, the sizes of the fragments bounded by cracks vary within a relatively narrow range. It was also found that the process of filling cracks with fracture products and substances from the service environment requires further investigation.

Keywords: composite materials, Estelite Asteria, Estelite Posterior, compression loading, strength, structure, microcracks, microscopic examination.

Introduction

Modern restorative dentistry is characterized by the widespread use of composite materials for the restoration of defects in the hard tissues of teeth. The use of such materials makes it possible to accurately restore the anatomical shape of a tooth while ensuring the required aesthetic and functional characteristics of the restoration [1–6].

One of the most important properties of dental composite materials is their ability to withstand mechanical loading [1, 2]. During the functioning of the dentoalveolar system, the dentition and the restorative materials used in it are subjected to compression, tension, bending, and shear forces [1, 9]. Among these, compressive loading is one of the major mechanical factors affecting restorations, particularly in the posterior regions of the dentition [1–3].

The mechanical behavior of a composite material is determined by its structure, the composition of the polymer matrix, and the characteristics of the inorganic filler [4, 7–9, 23]. The filler content, particle size and

shape, their distribution within the polymer matrix, and the strength of interfacial bonding are of particular importance [7–9, 23].

One of the modern composite materials is Estelite Asteria, manufactured by Tokuyama Dental. According to the manufacturer's information, the material contains 82% filler by weight and 72% by volume; its filler includes, among other components, silicon dioxide and zirconium dioxide, while the average size of the spherical particles is approximately 200 nm. Estelite Posterior is recommended for restorations in the posterior regions of the dentition, including occlusal surfaces. The manufacturer declares high mechanical properties of the material, which are important for its functional application. According to the manufacturer, the material contains 84% filler by weight and 70% by volume; the average particle size of the silicon–zirconium filler is approximately 2 μm [20–22, 24].

For composite materials, the composition and morphology of fillers play a critical role in the mechanical load-bearing and stress-transfer mechanisms, as well as in the distribution of internal mechanical stresses within the material structure, thereby influencing the kinetics of microcrack initiation and propagation under loading. [14–17]. One of the important indicators among the set of physicomechanical properties specified by the international standard ISO 4049:2019 is resistance to compressive failure. Ultimate compressive forces and stresses should be complemented by an investigation of the mechanisms of microcrack initiation and propagation, particularly in the region preceding failure [9–13]. Microscopic analysis of microcracks makes it possible to identify structural defects and assess their geometric characteristics [1, 9, 16, 25].

Therefore, the combination of mechanical testing and microscopic examination is appropriate for a comprehensive characterization of modern dental composite materials [20].

The aim of this study is to compare the mechanical strength of light-cured dental composite materials under compressive loading, based on their ultimate load-bearing capacity and microstructural changes, for Estelite Asteria and Estelite Posterior.

Materials and Methods. Two dental composite materials manufactured by Tokuyama Dental were investigated in this study: Estelite Asteria and Estelite Posterior. The materials were examined separately, without mixing them with each other. Five cylindrical specimens were prepared for each material (Fig. 1).

a b

Fig. 1. Specimens of composite materials for mechanical compression testing: a **– geometric dimensions;** b **– examples of actual specimens**

After the specimens had been formed and polymerized, their mass and geometric parameters were determined. Profield electronic scales with a weighing range of 0.1–500 g (YT02820), with an accuracy of at least 0.01 g, were used for mass measurements, while MK25 micrometers were used to measure the specimen dimensions.

The width of the microcracks was measured using a Euromex iScope IS.1152-EPL microscope. The area of the structural components on the surface of the metallographic sections and the lengths of the microcracks bounding them were measured using SOLIDWORKS software based on images of the metallographic sections obtained with Euromex iScope IS.1152-EPL microscopes.

To reveal microcracks and improve the accuracy of their measurements, the specimens were immersed in a 1% aqueous–ethanolic solution of methylene blue.

Table 1

Geometric characteristics of the investigated specimens

Parameter	Estelite Asteria	Estelite Posterior
Number of specimens	5	5
Mass , г	1,14 ±0,01	1,36±0,01
Diameter , мм	8,9±0,01	9,0±0,01
Height , мм	9,75±0,01	10,15±0,01
Cross-sectional area , м²	$6{,}218 \times 10^{-5}$	$6{,}3585 \times 10^{-5}$

The cylindrical specimens were placed between the compression platens of a PMM-125 hydraulic press, and the load was gradually increased until specimen failure occurred. Observation of the compression loading

process showed that the force–deformation relationship remained linear, while failure occurred almost instantaneously, which is consistent with the findings of other authors for materials of similar composition [10, 11, 17, 18].

The maximum load, expressed in newtons (N), recorded immediately before specimen failure was taken as the ultimate load. Following compression loading, a microscopic examination of the fracture surface was performed. The crack network identified on the surface of the metallographic section, as well as the width and length of the cracks, was evaluated.

Results

The results of the conducted tests demonstrated differences in the ability of the investigated materials to resist compressive loading. The Estelite Posterior specimens exhibited a higher load-bearing capacity and withstood a maximum load that was 3 kN higher than the corresponding value for the Estelite Asteria specimens. In relative terms, the maximum load for Estelite Posterior was approximately 17.7% higher, while its compressive strength exceeded that of Estelite Asteria by approximately 15%.

Microscopic examination of the fracture surfaces of the failed specimens was performed at ×100 magnification using a Euromex iScope IS.1152-EPL microscope. Fragments of the fractured materials were immersed in a 1% aqueous–ethanolic solution of methylene blue, dried, and embedded in epoxy resin in special molds to obtain cubic specimens (Fig. 2).

Fig. 2. Fixation of material fragments in epoxy resin

From the samples described above, microsections were prepared for microscopic examination. The analysis established that compression loading results in the formation of microcracks in the structure of both investigated materials, characterized by distinct linear structural defects. As the cracks propagate, they become filled with fracture debris originating from the crack walls. Figure 3 shows an example of such a microcrack.

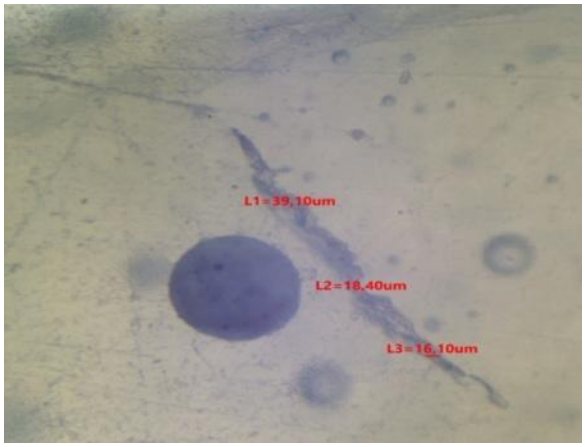

Fig. 3. Crack in Estelite Asteria material after compression loading, ×100

The identified crack propagates in the vicinity of bubble-like defects that formed in the material during its manufacturing process, but without directly interacting with them. This phenomenon may be explained based on the findings reported in [20]. It is likely that the stress fields formed around the bubble and the crack tip have the same sign, resulting in the formation of a potential barrier around the defect that prevents the crack from propagating directly toward the bubble [1, 9, 16, 25]. Under such conditions, the energetically most favorable crack propagation path is located at a certain distance from the defect.

Fig. 4 presents the results of measurements of crack opening width performed using a Euromex iScope IS.1152-EPL optical microscope. Statistical analysis of the results obtained for the Estelite Asteria specimens showed that the mean crack opening width was 24.50 μm.

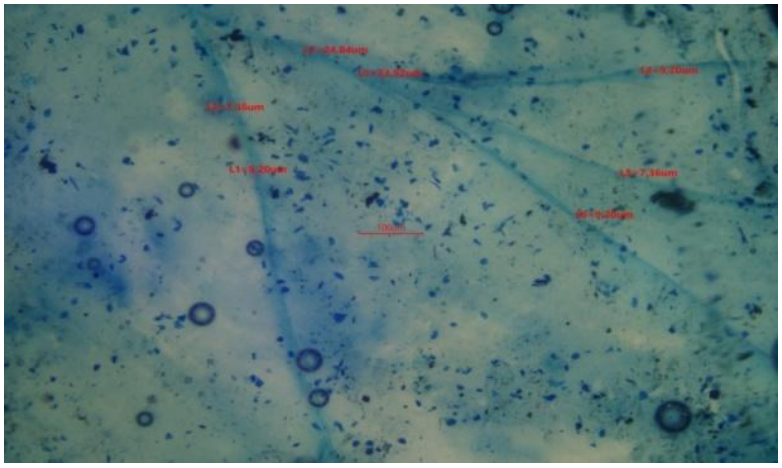

Fig. 4. Defects in Estelite Posterior material after compression loading, ×100

Fig. 4 shows a characteristic pattern of the initiation and subsequent development of a microcrack system. Initial crack formation is observed in the upper left corner of the micrograph, followed by crack branching and a subsequent increase in the number, length, and opening width of the cracks. As the magnitude of the applied load increases, the intensity of crack formation increases, resulting in the development of an extensive microcrack network (Fig. 5), the majority of which propagates along the boundaries of the structural blocks of the material.

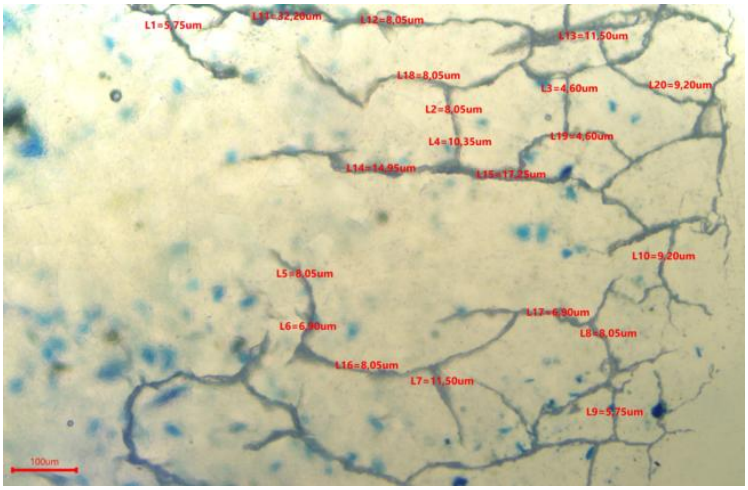

Fig. 5. Surface of a metallographic section of a fractured composite material specimen containing 50% Estelite Posterior and 50% Estelite Asteria, ×100

At this stage, the material is in a state of quasi-equilibrium and retains temporary structural integrity due to the presence of regions not affected by the microcracking process. Relaxation of internal stresses is accompanied by energy consumption for the initiation of new cracks, as well as for their subsequent propagation, which manifests itself as an increase in crack length and opening width [19, 24].

The results of measurements of crack opening width in the investigated materials are presented in Table 2.

Table 2

Results of microscopic examination and comparison of the mean crack opening width in Estelite Asteria and Estelite Posterior materials

parameter	Estelite Asteria	Estelite Posterior
Magnification	×100	×100
Mean crack width , мкм	**24,50**	**13,01**
Absolute difference , мкм	—	**11,49**
Reduction , %	—	**46,9**

Thus, the mean crack opening width in Estelite Posterior was 11.49 μm, or 46.9%, lower than that observed in Estelite Asteria.

Figure 5 shows the results of measuring the width of individual cracks on the metallographic section after failure of the material obtained by mixing 50% Estelite Posterior and 50% Estelite Asteria. The resulting dataset makes it possible to estimate the mean microcrack width in the investigated material during the degradation process.

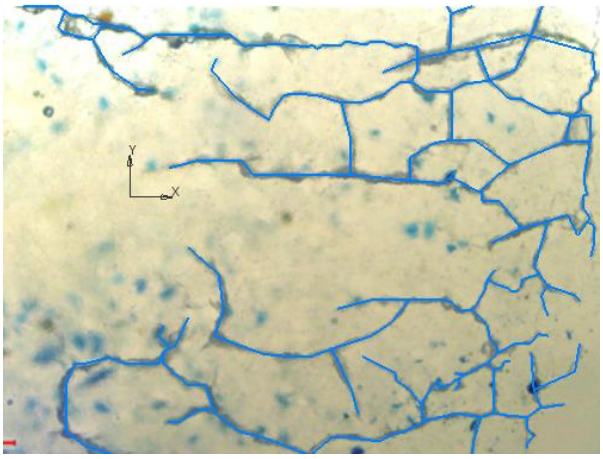

Fig. 6. Surface of a metallographic section of a composite material consisting of a mixture of 50% Estelite Posterior and 50% Estelite Asteria, with approximation lines applied to the microcracks using SOLIDWORKS software, ×100

After the approximation lines have been applied to the microcrack network, it becomes possible to measure the lengths of individual crack segments and the total length of all visible cracks intersecting the plane of the metallographic section.

Measurement of the total microcrack length within the plane of the metallographic section (Fig. 6), taking the actual scale into account, yielded approximately 5.5 mm per 1 mm². It is at approximately this defect density that failure of the material consisting of a mixture of 50% Estelite Posterior and 50% Estelite Asteria occurs.

Discussion

The results obtained demonstrated differences in the mechanical and structural behavior of the investigated dental composite materials. The data from the microscopic analysis of the fracture behavior and morphological features of crack formation provided an important complement to the results of the mechanical tests.

According to the results of the microscopic examination, the mean crack opening width in the Estelite Asteria specimens was 24.50 μm, whereas for Estelite Posterior this value was 13.01 μm, i.e., approximately 1.9 times lower. The absolute difference between the mean crack opening widths was 11.49 μm, while the relative reduction in this parameter for Estelite Posterior was 46.9%. Thus, the higher mechanical strength of Estelite Posterior under compression testing was accompanied by a smaller crack opening width after destructive loading.

The observed relationship may be associated with the specific features of structural damage initiation and development in the investigated materials. Under mechanical loading, stresses are redistributed between the polymer matrix and the inorganic filler. Localized stress concentrations may lead to the formation of microdefects, which, with a further increase in load, can transform into microcracks and propagate through the material, contributing to the formation of a fracture zone.

The nature and propagation path of cracks are determined by a combination of structural and mechanical factors, including the properties of the polymer matrix, the concentration, size, and morphology of the filler particles, as well as the strength of the interfacial interaction between the matrix and the filler. Differences in the structure and characteristics of the filler phase may therefore be one of the factors responsible for the differences in the mechanical behavior of the investigated composite materials.

At the same time, crack opening width cannot be regarded as an independent absolute criterion of material strength. Its informative value can only be assessed in conjunction with the results of mechanical tests and an analysis of fracture behavior. Within the framework of the present study, a comprehensive comparison of the obtained parameters revealed a consistent trend: the Estelite Posterior specimens exhibited a higher compressive strength and a smaller mean crack opening width compared with Estelite Asteria. This may indicate greater structural resistance of Estelite Posterior to crack development and opening under the conditions of the experiment.

Nevertheless, laboratory compression tests cannot fully reproduce the complex mechanical, physical, and chemical factors that determine the performance of dental restorations in the oral cavity. The service performance of composite materials may be affected by the nature and frequency of cyclic loading, temperature fluctuations,

water sorption, polymerization shrinkage, the nature of adhesive interactions with dental tissues, and the characteristics of occlusal contacts.

Therefore, the results obtained should be interpreted as a characterization of the structural and mechanical behavior of the investigated materials under the specified laboratory conditions. They confirm the appropriateness of a comprehensive approach to the evaluation of dental composite materials, combining the results of mechanical testing with microscopic examination of fracture behavior and crack morphology on the surfaces of specimens after failure.

Prospects for further research

Further research should focus on investigating the mechanical and structural behavior of the materials under repeated cyclic loading, which will make it possible to assess their fatigue strength and damage accumulation characteristics.

An important area of further research is the performance of thermocyclic tests followed by an assessment of changes in the mechanical properties, structural state, and crack formation behavior of the investigated materials.

A promising direction is to perform quantitative morphometric analysis of microcracks, including the determination of their number, length, opening width, and distribution pattern. In addition, investigation of the morphology of fracture surfaces using scanning electron microscopy would be appropriate, as this would allow the mechanisms of crack initiation and propagation to be established in greater detail.

Conclusions

1. A comparative assessment of the mechanical and microstructural characteristics of the dental composite materials Estelite Asteria and Estelite Posterior was performed based on the results of compression loading tests.
2. Microscopic examination of the fracture surfaces of the specimens at ×100 magnification established that the mean crack opening width for Estelite Asteria was 24.50 μm, whereas for Estelite Posterior it was 13.01 μm. Thus, this parameter for Estelite Posterior was 11.49 μm, or 46.9%, lower than for Estelite Asteria.
3. The higher compressive strength values of Estelite Posterior were accompanied by a smaller mean crack opening width, which may indicate less pronounced structural degradation of the material in the fracture zone under the conditions of the conducted test.
4. The combined use of mechanical testing and microscopic analysis of fracture surfaces is an appropriate approach for assessing the structural and mechanical resistance of modern dental composite materials and determining the specific features of their fracture behavior.

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Савуляк В.І., Вітавський М. О.Перлова А.В. , Король А.П. Мікроскопічні дослідження процесів руйнування стоматологічних композиційних матеріалів

У статті розглянуто проблеми міцності композиційних матеріалів стоматологічного призначення, зокрема їхньої стійкості до стискання. Цей вид навантаження є переважаючим для фронтальних та бокових жувальних зубів людини. Розуміння механізмів виникнення та розвитку внутрішніх напружень у матеріалах, що застосовуються для реставраційних робіт, має важливе значення для обґрунтованого вибору матеріалів та подальшого вдосконалення їхніх властивостей. Для експериментів використовували композиційні стоматологічні матеріали Estelite Asteria та Estelite Posterior виробництва Tokuyama Dental. Розглянуто методику виготовлення зразків для навантаження стисканням, проведено дослідження з фіксацією граничних зусиль та напружень. Зруйновані зразки розглянуті на предмет визначення виду руйнування. Встановлено, що практично відсутня пластична складова під час процесу деформування, а залежність навантаження – деформація є квазілінійною. З метою подальшого дослідження уламки матеріалів фіксували в епоксидній смолі, після чого виготовляли кубічні зразки. На їх основі готували мікрошліфи для детального дослідження механізмів накопичення пошкоджень, формування та розвитку мікротріщин у структурі матеріалу. Виявлено, що механізми руйнування та конфігурація мікротріщин залежать від розмірів неорганічних наповнювачів композитів. Показано, що лавиноподібна втрата цілісності виробу з композиційних матеріалів відбувається при досягненні певної щільності мікротріщин (сумарна довжина тріщин на одиницю площі). При цьому розміри фрагментів, обрамлених тріщинами, змінюються в достатньо вузьких межах. Виявлено, що процес заповнення тріщин продуктами руйнування та середовища функціонування вимагає подальших досліджень.

Ключові слова: композиційні матеріали, Estelite Asteria, Estelite Posterior, навантаження стисканням, міцність, структура, мікротріщини, мікроскопічне дослідження.