МИКРОСТРУКТУРА НА IPS-EMPRESS КЕРАМИКАТА: литературен преглед

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СТОМАТОЛОШКИ ФАКУЛТЕТ - Загреб (Хрватска), Отесек за фиксна стоматолошка протетика

Според структурата и својствата, емпрес керамиката припаѓа на групата глас-керамика. Глас-керамиката е поликристален материјал во цврста состојба добиен по пат на кристализација на стакло во контролирани услови. За време на таа контролирана кристализација на стаклото се добива двофазен фин кристален материјал, со поголемо или помало учество на аморфна стаклена матрица. Добивањето микрокристали коишто се големи неколку микрометри се случува за време на процесот на мешање на целиот волумен на стаклото, при создавањето на јадрата и градењето на кристалите. Јадрата се или латентно присутни во стаклото или се додаваат како нерастворлив материјал, со фино дисперзирана распределеност на микстурата (глас аморфна маса). Поради хетерогената структура и/или провоцирањето компресивен стрес во граничните фази, можно е постигнување значително зголемена сила, а на тој начин и намалување на тенденцијата за фрактура што пак е од особено значење за клиничката практика. Појавата на микронивоата, особено од несоодветното термичко ракување, доведува до пукнатини, до нивна експанзија за време на цвакањето и на крајот до фрактура на фикснатa керамичка протетичка изработка.

Клучни зборови: емпрес-керамика; глас-керамика

Glass ceramic is a polycrystalline material obtained by controlled glass crystallization. During controiied, guided giass crystallization, a biphasic, finely crystallized material with a higher or lower proportion of amorphous giass matrix is produced. The microcrystals of several millimeters in size are formed by the procedure of totai giass volume heating, nucleus formation, and crystal growth. The nuclei may be latently present in the glass or added as an insoluble materiai to the finely dispersed mixture (amorphous giass mass), in case of homogeneous basic giass, certain causal relationships may result in an excessive critical size of the nuclei, which may then undergo an uncontrollable growth. The guided, directed crystallization differs from the sporadic crystal growth by the formation of a large number of nuclei of uniform, small dimensions of a few micrometers, evenly distributed all over the total glass volume. The specimens of glass and of heated glass can be readily differentiated. The plates made of glass are transparent, whereas those made of heated glass are slightly turbid.

The heterogeneous structure and/or induction of pressure strain at the phase borders allows the hardness to be considerably increased in these systems, thus reducing the possibility of rupture: Compared to the conventional dental ceramic, the glass-based systems offer a wide array of applications in dentistry because of their chemical inertness, high mechanical strength, relatively simple working technique, low grade of shrivelling during manufacture, and other thermal and physical properties. For instance, glass ceramic with large β-quartz microcrystais, recommended for use in dental medicine, contains Li_2O, Al_2O_3, SiO_2, ZnO, MgO, TiO_2, ZrO_2, P_2O_5 and CeO_2, whereby Al_2O_3 increases the strength by incorporating into the grate. In 1956, Cahoon and Christensen (1) demonstrated a small admixture of MgO in Al2O3 to prevent nucleus growth discontinuity and to allow the synthesis of a material of an almost theoretical density.

Attempts have also been made to use some other types of glass ceramic in dental medicine, e.g., the one containing beta-spodumene crystals (a mineral containing lithium aluminum silicate in the form of monoclinic crystals, LiAISi206). Internal mica crystals are randomly directed and are responsible for the strength, reinforcing the final product, which results in a two-fold shear strength found in conventional ceramic. The inner strength of glass ceramic is ensured by its monolithic structure. Variations and improvement of the material characteristics to emphasize the favorable features of each individual component are obtained by target modifications of the process parameters. The affluence of its atomic structure and a myriad of possible mutual exchanges of chemical elements or combinations offer an almost indefinite number of ceramic materials with most diverse characteristics. Its atomic structure ensures chemical stability of the ceramic, making it insensitive to multiple impacts of the environment. Great hardness and strength of the ceramic material is ensured by its atomic bonds, whereby the bond strength prevents the sliding of atomic facets over one another, which does occur with metals. On strain, ceramic retains its shape until trespassing a certain threshold of strain, when the bonds slacken and the material breaks down. Ceramic materials are less resistant to the tensile strain and torsion than to pressure strain, because the tensile strain and torsion stretch the edges of the fissures and distend them, whereas the air pressure results in their closure (2).

Ceramic materials would be much more resistant to rupture, if damages that occur during their manufacture or due to mechanical load exerted by the construction could be avoided. Any strain is initially concentrated at such weak sites. As critical strain cannot be exclusively localized to the site of damage, it is transferred through the material, leading to the rupture of the ceramic material. Therefore, attempts have been made in practice to prevent or neutralize macroscopic damage to the surface, in order to increase the strength of ceramic objects. In homogeneous monolithic materials, the mpture occurs spontaneously, because the fissures are being freely distended, whereas in heterogeneous materials the spread of rupture is prevented by the phases, resulting in a higher resistance to rupture.

Upon cooling down, anisotropic strain occurs in the heterogeneous polyphasic ceramic. During the procedure of glass meltage cooling, the two phases are bound (leucite crystals and glass matrix). The glass matrix is exposed to tangential pressure strain, whereas leucites are exposed to radial pulling strain. These strains are in mutual balance. Leucite crystals show stronger contraction during the process of cooling than the glass matrix, based on their higher thermal elasticity. On the other hand, this higher contraction is followed by transformation of cubic, high leucites into tetragonal leucites, causing a 1.2% volume reduction. In the last stage of contraction, fissures occur in the leucite, because the tensile strain exceeds the tensile strength of the leucite. This is accompanied by concurrent, partial "separation" of the glass matrix and leucite crystals. The initial balance between the tensile and pressure strain changes on the account of the former. Pressure strength of the glass matrix is adequate for the pressure strain to be "frozen" on the phase sides. According to this presentation, the spontaneous spread of the fissures is diminished due to the latently present pressure strain. Furthermore, fissures on the particle sides are diminished and fracture energy is decreased due to the dispersed structure. Eventually, the latently present microfissures with rounded, "blunt" tips can prevent or stop the spread of fissures.

Another possibility of increasing the glass strength is seen in the concept of the macroscopic pressure strain transfer to the microscopic area. If crystals characterized by thermic elasticity exceeding the elasticity of the glass surrounding are formed during the process of cooling down, the crystals from the surrounding will be exposed to the effect of pressure. The greater the difference between the coefficients of glass elasticity and crystal phase elasticity, the greater the action of pressure strain and simultanenous hardening. This principle of material hardening has been achieved in the Empress ceramic (3-5).

Empress ceramics

Empress ceramics are the result of many years of effort to discover a ceramic material which would not only be sufficiently hard to withstand masticatory forces, but which would also be sufficiently aesthetically acceptable. Two materials are applied for two different techniques of work: pressed ceramics for the technique of tinting and pressed ceramics for the technique of layering, which both have their specific indications. Attainment of the best aesthetics is helped by colour, corrective material and material for glazing. The material mentioned can consist of a basic glass mass, from a mixture of glass materials and/or additives such as: colour particles, means for fluorescence and additives for increasing the points of softening (6). A precondition for

the attainment of necessary resistance to breakage Empress ceramics is harmonious adaptation of the fired ceramics on the layer of pressed ceramics. Because of the difference in coefficients of thermal ductility of the material for the technique of layering, i.e. technique of tinting, in relation to the coefficient of ductility of the material used in the subsequent process of ceramizing, during the cooling on the marginal surface, towards basic ceramics the aimed compressive stress neutralizes the tensile stress. With this technique the genial conception of antique vaults is transferred to the micro area and structural strengthening of this ceramics is achieved.

According to Ludwig (7) 1991, strength measured by loading at an angle of 30o amounts to 335 N for Empress ceramics. Strength under axis loading for Empress ceramic amounts to 2180 N. The flexural strength of Empress ceramics, obtained by loading at three points, during an investigation by Seghia & Sorensen (8) amounted to 127 MP, and value of the same test in an investigation carried out by Lehner & Scharer (cit. according to Ludvig (7)) amounted to 182 MP.

In 1991, Unterbrink (9) measured the shear strength of the Empress ceramic and obtained values of about 200 MP, pointing to the possible use of this material in the oral cavity, for the manufacture of crowns, inlays, facets, and onlays (10-14).

Mehulić and co-workers (15) 1997, investigated the quantitative share of crystals and amorphous matrix of samples made from Empress ceramics and showed significant differences ($p<0.001$) in the share of crystals between the basic samples for the tinting technique, and for the layering technique, and also significant differences ($p<0.001$) within the two groups of samples (tinting technique and layering technique) depending on the kind of cooling (at room temperature, rapid - by quenching into water, and slow - by leaving in the furnace to room temperature. This investigation has shown a larger share of the crystalline phase in the slowly cooled samples, indicating the possible crystallization of added leucites, the sructure is more homogeneous and visually it is possible to observe a greater volume share of the white phase. Furthermore, transformation of "high" into "deep" leucites occurs during the quenching, which can be explained by the difference in the coefficients of thermal ductility of leucites and glass matrix.

In the rapidly cooled samples, by casting into water, the quantitative share of crystals is decreased in relation to the quantity of the glass matrix. On the surface of the crystals and their interconnection with the glass matrix micro-cracks can be seen and the porosity is most marked on the surface of the rapidly cooled samples, in the research carried out by Music and co-workers in 1996 and 1997 (16,17) the diffractograms of the Empres-ceramic samples, set at given temperatures, confirmed the crystalline amorphous structure. The speed of cooling clearly influences the structure of the basic material for the layering technique. The basic material for the tinting technique also indicates structural changes, depending on the speed of quenching, although not as marked. EDX analysis of samples of the basic material for the tinting technique, which were rapidly cooled, shows a preconcentration of potassium on the surface of the grain itself. The sample of the basic material for the layering technique, rapidly cool- ed by casting into water, shows a preconcentra- tion of silicon and formation-of aluminosilicate grains. Examination by electronic microscopy showed marked pores on the surface of the same sample. It is assumed that such structural changes can result fromcracking of Empress ceramic fi- xed dental prosthetics during function.

EDX analysis and examination by electronic microscopy for both techniques, slowly cooled in a furnace, showed a satisfactory appearance of surfaces with regard to the relative intensity of Na, K, Al, and Si, which corresponds to leucite-type glass-ceramics.

Fourier-transform infrared spectro-photometry (FTIR) showed a spectra with characteristic bands for leucites at 640 and 543 cm-1. The basic material for the tinting technique becomes amorphous above 1300°C, while the basic material for layering technique above 1400°C, which is demonstrated by the diffraction of X-rays, namely by the FT- spectra, which lose bands typical for leucite crystals. Raman's spectrum of leucite glass displays a wide band, with a maximum at -4 600 cm -1, due to the fluorescent/luminescent property of the material, and the two sharp luminescent peaks at 5 037 and 5 007 cm-1. Raman's spectra confirm the finding of the FT-IR spectra.

The IPS Empress ceramic belongs to a group of pressable glass ceramic. It is a biphasic crystal material in an amorphous glass matrix. If crystals characterized by a coefficient of thermic elasticity exceeding the respective value of the surrou-ding glass are formed during the process of cooling down, they will be exposed to an increased pressure strain. Thus, the synergistic effect of pressure strain and simultaneous hardening produces a combination of events that will lead to the desired hardening, achieved in the Empress ceramic.

MICROSTRUCTURE OF IPS EMPRESS CERAMICS review of literature

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Summary

According to its structure and properties Empress ceramics belongs to the group of glass-ceramics. Glass-ceramic is a polycrystalline solid produced by partial crystallization of glass under control led conditions. During that controlled crystallization of the glass a two-phase fine crystalline material is produced with more or less participation of amorphous glass matrix. The production of microcrystals of several micrometres in size occurs during the process of tempering the total volume of glass, the creation of nucleus and development of crystals. Nucleus are either present latently in the glass or are added as an insoluble material in a finely dispersed distribution of mixture (glass amorphous mass). Because of the heterogeneous structure and/or provocation of compressive stress on the phase margins, it is possible to achieve significantly increased strength and so reduce the tendency to fracture, which is extremely important in clinical practice. Occurrences at the micro level, particularly inadequate thermal treatment, leads to the occurrence of cracks, their expansion during mastication and finally fracture of the fixed ceramic prosthesis.

Key words: Empress ceramics, glass-ceramics, microstructure

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