

Abstract

Recently, many improvements have been done in the field of the production and the mechanical properties of the ceramics, particularly in the field of dentistry. In this review at first, we will make a brief introduction to dental ceramics. Special emphasis will be given to lithium disilicate ($\text{Li}_2\text{O}\cdot 2\text{SiO}_2$) based ceramics. $\text{Li}_2\text{O}\text{-SiO}_2$ binary system will be examined and then will be evaluated in terms of controlled crystallization of $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-K}_2\text{O}$ system. Also, P_2O_5 has an important affect for lithium disilicate crystallization. Studies have concluded that fine-grained interlocking microstructures are observed in dental lithium disilicate ($\text{Li}_2\text{O}\cdot 2\text{SiO}_2$) ceramics with the addition of P_2O_5 nucleating agent. In the present review, we summarize the mechanical strength of dental lithium disilicate ($\text{Li}_2\text{O}\cdot 2\text{SiO}_2$) ceramics depending on the microstructural evolution.

Key words: Ceramics, Dental, Lithium disilicate, Restoration.

Introduction

Natural tooth is a complex hard tissue made up of special cells called ameloblasts, odontoblasts and cementoblasts. The hard-outer coating (tooth crown) called enamel is formed by the ameloblasts. Ameloblast cells are present in a layer on the outside of the tooth bud whereas the enamel is present on the inside of the ameloblasts. Enamel wear or loss due to decay is unable to be repaired by the body. The odontoblasts form the dentin and are located on the inside of the tooth bud between the enamel and the dental pulp. The dentin grows inwardly by the odontoblasts. The dentin supports the tooth enamel and it gives protection to the pulp and covers under the gums. It provides an attachment by a ligament to the bone surrounding the tooth. The enamel is relatively hard and brittle ($E \sim 65 - 70$ GPa), the dentin is softer and more compliant ($E \sim 15 - 19$ GPa) and bone is much more compliant ($E \sim 12$ GPa).¹

The medical biocompatible and bioactive glass-ceramics can be grouped according to environment of application and desired properties. These are called implantology (medical protheses) and restorative dentistry (dental protheses).²

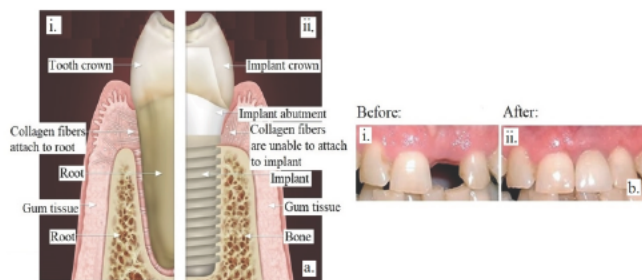


Figure 1 a.i. Natural Tooth; ii. Dental Implant;³ b.i. Before single tooth implant; ii. After single tooth implant.⁴

Figure 1.a shows natural tooth and dental implant.³ Dental restoration glass-ceramics must fit the biomaterial application standards, i.e compatibility with the oral environment. Dental restoration surface bioactivity is not a desired property. Further, shade, translucency, and toughness properties of the surface must fit those of the natural teeth. The chemical durability of dental restorations has to be even higher than that of the natural teeth due to

undesired cavity formation in the glass-ceramics.² Figure 1.b indicates before and after single tooth implant⁴

Dental ceramics do not exhibit good resistance to tensile and shear stresses. However, under compressive stresses, they show very good resistance. This gives the ceramics brittle behaviour and a fracture occurs under tensile stresses. There are several kinds of ceramic structure fractures in the oral environment. First, The crack can initiate from the contact region between the teeth, from dental cement surface under the contact region or from crown and connector margins in fixed partial dentures. Failure occurs due to structural defects in dental ceramic protheses. Defects in dental ceramic protheses can appear as sub-millimeter micro-cracks. These can be due to ceramic protheses production or due to the chewing forces in the oral environment.^{5,6}

The addition of P_2O_5 results in the formation of elongated rod-like $\text{Li}_2\text{Si}_2\text{O}_5$ crystals with an interlocking microstructure. Thus, a high flexural strength is obtained in the highly translucent glass-ceramic. Other mechanical properties, particularly the microhardness and fracture toughness are also high and the elasticity modulus value is moderate.

Lithium Disilicate Based Ceramics

The thermal, chemical and dielectric properties of the glass-ceramics are superior to many materials. In the production of glass-ceramic materials, the chemical composition of the parent glass is very important. The crystalline phases formed in various microstructures is the most important advantage of the glass-ceramic production. Based on the mode of growth of the crystalline phases, different appearances exist.⁷

 $\text{Li}_2\text{O}\text{-SiO}_2$ Binary System

Regarding the $\text{Li}_2\text{O}\text{-SiO}_2$ binary system, many theoretical studies focused on the phase separation, nucleation and crystallization in the glasses. Because of the phase separation, opalescent or opaque glasses form during cooling of $\text{Li}_2\text{O}\text{-SiO}_2$ liquids containing less than 30 mol.% Li_2O . In SiO_2 -rich glass matrix, there are Li-rich phase droplet zones and also segregations inside the droplets, which was observed by TEM. In 14-16 mol.% Li_2O range in the entire glass, inside the droplet phases, there is

stoichiometric $\text{Li}_2\text{Si}_2\text{O}_5$ composition. On entrance of Li_2O into the SiO_2 -rich phase around the droplets when the Li_2O amount of the entire glass increases, the surface tension of the two phases is reduced and the droplet sizes continuously decrease. When 33.3 mol.% of Li_2O corresponds to $\text{Li}_2\text{Si}_2\text{O}_5$ in the entire glass, the droplet phase and the phase around the droplets has the same composition of stoichiometric glass composition.⁸

In the Li_2O - SiO_2 binary system, there is an immiscible region between the Li_2O - 2SiO_2 and SiO_2 . The controlled nucleation and crystallization of lithium metasilicate and/or lithium disilicate phases dominate the final product characteristics after the production of the glass-ceramic materials. When in the glasses with Li_2O amount is <30 mol. %, because of the phase separation, upon cooling opalescent or opaque appearance is obtained. However, when SiO_2 amount is higher than the stoichiometric $\text{Li}_2\text{O} \cdot 2\text{SiO}_2$ (33.33 mol. % Li_2O -66.66 mol. % SiO_2) in the glasses, the phase separation occurs where there is a matrix phase, which is similar to lithium disilicate, and an isolated droplet SiO_2 rich phase. The glass-ceramics produced from binary system, show some disadvantageous mechanical and chemical properties, which prevents various candidate applications, although the examination of the nucleation of parent glass, which has lithium disilicate stoichiometric composition, is done widely. The dental restorations, metal-glass seals etc are the candidate applications of lithium disilicate glass-ceramics, which belong to non-stoichiometric compositions.⁹

By a suitable heat treatment, molten glass is converted into a ceramic with a fine grain thus glass-ceramics are produced. Changing the composition and applying a suitable heat treatment provide well known, wide-range properties of glass-ceramics. The parent glass composition, type of nucleating agent and the thermal treatment determine the final crystalline phases, which are precipitated from the glasses and the microstructures and the properties of glass-ceramics. In order to develop a new kind of glass-ceramics, the crystallization of the glass must be controlled.¹⁰

From the aspect of controlled crystallization of glasses, the best and the most detailed examination was done in the Li_2O - SiO_2 system. Stookey developed lithium disilicate ($\text{Li}_2\text{Si}_2\text{O}_5$) as the first glass-ceramic. The controlled nucleation and crystallization mechanism were discovered and the mechanical properties of the glass-ceramic, which is the final product is higher than those of the parent glass.¹¹ According to Höland and Beall, following this main development on lithium disilicate glass-ceramics, the research on two topics is done. First is the glass-ceramics derived from the binary Li_2O - SiO_2 system and the second is the multi-component glass-ceramics. The nucleation mechanisms and phases formed before lithium disilicate precipitates in the binary Li_2O - SiO_2 glass system, of which the composition is close to $\text{Li}_2\text{O} \cdot 2\text{SiO}_2$, are the main focus of research activities.¹²

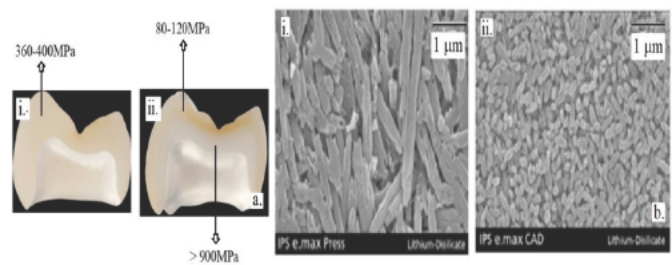


Figure 2: a. Mechanical properties of i. Lithium disilicate and ii. Zirconium oxide tooth; b. Scanning Electron Microscope (SEM) images of Lithium disilicate i. IPS e.max Press and ii. IPS e.max CAD

Figure 2, indicates the mechanical properties of Lithium disilicate and Zirconium oxide tooth¹³ and the microstructures of Lithium disilicate IPS e.max Press and IPS e.max CAD.¹⁴ A non-stoichiometric metastable precursor phase was discovered by Deubener, Bruckner, Sternitzke *et al.*¹⁵

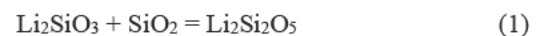
2.2 Controlled Crystallization Of SiO_2 - Al_2O_3 - K_2O System

Lithium disilicate based dental ceramics can be produced by the controlled crystallization of SiO_2 - Al_2O_3 - K_2O system. The synthetic lithium disilicate can be added into powdered glass to obtain them. Another method is to mix a low expansion glass frit with a high expansion frit containing lithium disilicate.¹⁶

Lithium disilicate glass-ceramic based dental prostheses can be manufactured by sintering, heat pressing, and computer aided design and machining (CAD/CAM) – hot-pressed IPS Empress1, which fits the dental restoration aesthetic standards.^{17,18}

The stoichiometric lithium disilicate compound has gained importance after the work of Stookey.¹³ Ivoclar-Vivadent developed IPS Empress1 and Empress12 products using a composition range of %wt. 57-80 SiO_2 , 11-19 Li_2O , 0-13 K_2O , 0-5 Al_2O_3 , 0-8 ZnO , 0.1-6 La_2O_3 , and 0.1-11 P_2O_5 . These two products were manufactured by hot pressing.¹⁹

In the glass-ceramics, which contain Al_2O_3 , the crystallization of lithium disilicate ($\text{Li}_2\text{Si}_2\text{O}_5$) is done by lithium metasilicate (Li_2SiO_3). Lithium disilicate and lithium metasilicate crystallized primarily at 600°C in the glass composition which does not contain Al_2O_3 . The solid-state reaction of lithium metasilicate and silica yields lithium disilicate as in Eq.1:



Lithium metasilicate has high machineability and thus the product can be machined using CAD/CAM at this stage. When the crystallization of lithium disilicate occurs as in Eq.1, high strength can be obtained at the final stage.^{20,21}

Ivoclar-Vivadent company examined in a multi-component system, the glass-ceramic production with a composition of (in wt.%) 57-80 SiO_2 , 11-19 Li_2O , 0-13 K_2O , 0-5 Al_2O_3 , 0-8 ZnO , 0.1-6 La_2O_3 , and 0.1-11 P_2O_5 . As a result, the material IPS Empress12 was developed. With the same hot-

pressing route, they produced IPS Empress12 and IPS Empress1 glass-ceramics similarly and the properties were investigated.²⁰ Lithium disilicate glass-ceramic contains 1-2 μ m crystals in a glass matrix and also has high density. For dental crown and three-unit bridge (for anterior teeth) applications, a metal-free framework can be produced from high strength, translucent IPS Empress12 glass-ceramic.¹⁶

Li₂O-SiO₂-Al₂O₃-K₂O-P₂O₅ system was investigated to produce lithium disilicate glass-ceramic and the effect of ZrO₂ addition was also examined. Fine and strong microstructure was observed in the glass-ceramics without ZrO₂ addition. Zirconia prevents crystal growth and thus it affects the crystallization. As the amount of zirconia increases, small crystals take part. As the crystallization temperature increases and thus the crystallization of Li₂SiO₃ is developed, Li₂Si₂O₅ crystallizes vigorously and the grain size becomes coarse. As a result, the strength increases and a strength up to 200MPa is obtained. The strength of the glass-ceramic, which contain 4 wt% ZrO₂, has a higher strength than glass-ceramics containing 2.0 wt% or 2.91 wt% ZrO₂. ZrO₂ addition adjusted the translucency of the glass-ceramic. A high strength microstructure was produced along with a highly translucent glass-ceramic.¹⁸



Figure 3: a. IPS e.max CAD block and tooth; b. Dental technician is working with Lithium disilicate; c. IPS e.max, Lithium disilicate restoration

Figure 3 shows Lithium disilicate based IPS e max CAD dental restoration and technician work on Lithium disilicate.²²⁻²⁴ SiO₂-Li₂O-K₂O-Al₂O₃-ZrO₂-P₂O₅ system was also investigated to develop lithium disilicate glass-ceramics. From 0.5 mol.% to 2.0 mol.% P₂O₅ (increasing by 0.5 mol.%), lithium disilicate glass-ceramics were produced to make dental restorations. Flexural strength and translucency were determined for the developed lithium disilicate glass-ceramics. The effect of the amount of P₂O₅ on the microstructure and properties of the glass-ceramics was evaluated. An interlocking microstructure formation was observed in the glass-ceramic with a P₂O₅ amount of 1.0 mol.%. In the microstructure elongated rod-like Li₂Si₂O₅ crystals were present. As a result, the highest flexural strength was determined for this particular composition.²⁵

In a study, SiO₂-Li₂O-Al₂O₃-K₂O-ZrO₂-P₂O₅ system was analyzed to produce lithium disilicate glass-ceramics and the nucleation, primary phase formation and solid state reaction were examined. The amount of nucleating agent, P₂O₅ was 3.2 wt% for a maximum. The microstructure evolution, the nucleation mechanism and the solid state reaction of the crystal phase precipitation were investigated by using thermal analysis, SEM (Scanning Electron Microscopy) and XRD (X-ray diffraction analysis). The bulk crystallization was controlled by adding P₂O₅. Nano-scale Li₃PO₄ phase catalyzed the nucleation process. This was visualized by using HR-SEM. Both Li₂SiO₃ and Li₂Si₂O₅ phases were catalyzed heterogeneously most probably by Li₃PO₄. Following the nucleation of lithium metasilicate and lithium disilicate, lithium disilicate crystals are in the form of agglomerated nanocrystals in a relatively small amount. On the contrary, there is a rapid growth of lithium metasilicate and also there is a decomposition of lithium metasilicate at 780-820°C. As a result, a drastic increase of lithium disilicate phase occurs. Lithium disilicate phase formation is a result of a solid state reaction with the SiO₂-rich glassy phase. As a preliminary phase, there is a cristobalite phase formation. Finally, interlocking microstructure occurs with a high crystallinity when a glass-ceramic with 3.2 wt% P₂O₅ is produced and this glass-ceramic exhibits a high-strength of 726 ± 63 MPa and translucency.²⁶

SiO₂-Li₂O-Al₂O₃-K₂O-ZrO₂-P₂O₅ system was investigated to produce lithium disilicate glass-ceramics. The maximum P₂O₅ amount used in this study is 3.2 wt%. P₂O₅ addition controlled the bulk crystallization. Nano-scaled Li₃PO₄ phases catalyzed the nucleation heterogeneously by epitaxy action, of Li₂SiO₃ and Li₂Si₂O₅ phases. Lithium metasilicate and lithium disilicate were nucleated at first. Lithium disilicate turns into a form of agglomerated nanocrystals with a small amount. However, there is a rapid growth of lithium metasilicate and later decomposition occurs at 780-820°C, which results in an increase of lithium disilicate phase. The reason for this is a solid-state reaction with the SiO₂-rich glassy phase. Also, the formation of cristobalite occurs accompanying this reaction. The addition of 3.2 wt% P₂O₅ resulted in a high-strength of 726 ± 63 MPa and translucency. At first, between 500-560°C, Li₃PO₄ nanophases form then Li₂SiO₃ and Li₂Si₂O₅ were nucleated. In the range of 530-590°C, Li₂SiO₃ grows and Li₂Si₂O₅ nanophases agglomerate but crystal growth does not occur. Between 590-750°C, there is a fast growth of Li₂SiO₃ and Li₂Si₂O₅ does not grow. Later, in the range of 750-820°C, cristobalite forms and decomposes. Finally, between 780-820°C, Li₂SiO₃ decomposes and a fast growth of Li₂Si₂O₅ occurs.²²

Molla, Chakradhar, Kesavulu *et al.* produced transparent MnO₂ doped lithium disilicate glass-ceramics by melt quenching and controlled crystallization and the microstructure, mechanical and optical properties were characterized. Elongated, highly interlocked and dense (~80 vol.%) nanocrystals were observed in the

microstructures. The average sizes of the crystals were measured to be ~100 nm. Following the heat-treatment at 700°C for 1h., lithium disilicate was the only crystalline phase as determined by XRD. The average microhardness and high fracture toughness were determined to be ~5.6 GPa and ~2.8 MPa.m^{1/2}, respectively. 3-point flexural strength value was measured to be ~250 MPa and the elastic modulus value was moderate.²²

2.3 Addition of P₂O₅ As Nucleating Agent

Fernandes, Tulyaganov, Goel *et al.* investigated the structure, properties and devitrification of glasses in the Li₂O–SiO₂ system by Al₂O₃ and K₂O addition and stated that in general, when the glass composition of lithium silicate changes slightly, the phase morphology is significantly affected. The amorphous phase separation and the crystal nucleation rate were observed to be enhanced simultaneously when P₂O₅ is added into lithium disilicate. The phase evolution, morphology and thus thermo-physical properties of low alumina Li₂O–SiO₂ glasses were affected by co-addition of TiO₂ and P₂O₅. In Li₂O–SiO₂ glass, the traditional nucleating agent is ZrO₂ and this agent was observed to enhance the polymerization of the silicate network and it also causes amorphous phase separation prior to the crystallization. Low amount of MnO₂ and V₂O₅ can cause the critical cooling rate of the melt to decrease thus the glass forming tendency increases.⁹

As a nucleating agent, P₂O₅ is traditionally into lithium silicate glass systems to enhance phase separation thus heterogeneous nucleation occurs. Finally, following the heat treatment interlocking morphology is observed with a fine-grain.²²

Except one study, most articles are focused on the theory of nucleation, regarding to P₂O₅ and the crystallization of lithium disilicate glass-ceramics. Following one-step heat treatment at 880°C, interlocking microstructures with fine grains were obtained when 1.5-2.5 mol% P₂O₅ was added into the system.²¹

a.		b.	
CTE (100–400°C) [10 ⁻⁶ /K]	10.2	CTE (100–400°C) [10 ⁻⁶ /K]	10.2
CTE (100–500°C) [10 ⁻⁶ /K]	10.5	CTE (100–500°C) [10 ⁻⁶ /K]	10.5
Flexural strength (biaxial) [MPa]*	360	Flexural strength (biaxial) [MPa]*	400
Fracture toughness [MPa m ^{1/2}]	2.25	Fracture toughness [MPa m ^{1/2}]	2.75
Modulus of elasticity [GPa]	95	Modulus of elasticity [GPa]	95
Vickers Hardness [MPa]	5800	Vickers Hardness [MPa]	5800
Chemical solubility [µg/cm ³]*	40	Chemical solubility [µg/cm ³]*	40
Crystallization temperature [°C]	840-850	Crystallization temperature [°C]	915-920

*according to ISO 6872

*according to ISO 6872

Table 1: Physical properties for a. IPS e.max Press and b. IPS e.max CAD

Table 1 indicates the physical properties of IPS e.max Press and CAD.²⁷ Zheng, Wen, Song *et al.* as the amount of P₂O₅ increased from 0 to 4.0 mol.%, at first the crystallization temperature showed an upward trend and then downward one because of the phase separation and the heterogeneous nucleation with the aid of Li₃PO₄ phase. The nucleation density increased and the growth rate decreased when P₂O₅

increased from 1.0 to 4.0 mol.%. Final microstructure exhibited interlocking rod-like and spherical morphology and prior to it, there were plate-like polycrystalline aggregations. There was one composition with out nucleation agent or other oxides, in this sample, phase transformation and microstructure were different from other glass ceramics. Because of the highest viscosity, lowest crystallinity was obtained. The precipitations of Li₂SiO₃ and Li₂Si₂O₅ were controlled by the homogeneous nucleation mechanism. Furthermore, the microstructure exhibited plate-like aggregations and there were coarser grain sizes in this sample.²⁷

Von Clausbruch, Schweiger, Höland *et al.* investigated Li₂O–SiO₂–ZnO–K₂O–P₂O₅ system to obtain lithium disilicate glass-ceramics and the crystallization and microstructure were characterized. The amount of P₂O₅ was between 0.0-2.5 mol.%. Better flexural strength values were obtained when the amount of P₂O₅ was in the range of 1.5–2.5 mol.%.²⁸

Conclusion

Material selection and the production methods are of quite significance as the new generation of ceramic materials is introduced. Regarding the restoration design and the aim of the usage, a detailed understanding of the materials is necessary for the accurate performance of these restorations.

There is not any ideal material from each aspect, which will be closer to the natural tooth structure. For the few decades, there has been an important development in the mechanical and biological properties of ceramics. From the aspect of esthetics and chemical durability, many research is present on all-ceramic crowns. Specifically, ceramic implants, which possess high strength and crowns and veneers, which have high esthetics and strength are some of these. In the area of the dental ceramics, recent research is focused on the novel ceramics and nanotechnology. Also an improvement in the biocompatibility characteristics is another topic.

Dental ceramics do not possess any adverse reactions in their dental applications along with natural aesthetics and biocompatibility and their role in the area of dentistry will likely to continue in the future. Although, the balance between aesthetics and biomechanical strength must be considered. The glassy and crystalline phase amount ratio determines the mechanical and optical characteristics in the final dental restoration. In order to obtain a dental restoration with good translucency, the glassy phase amount must be increased. On the contrary, for a dental restoration, which possess good strength, the crystalline phase amount must be increased.

There has to be a balance between these the glassy and the crystalline phases. These ceramics exhibit resistance to abrasion. However, fracture toughness and resistance to the tensile stresses are the lower properties. These disadvantages must be prevented.

In the present review, an introduction to dental ceramics was made. Lithium disilicate $\text{Li}_2\text{Si}_2\text{O}_5$ is one of the ceramic materials that are used in the dentistry. Mechanical properties of the dental restorations have also been focused in the present chapter. These mechanical properties, which are of significance in the dentistry, are hardness, strength and fracture toughness. Strength and fracture toughness are the two interrelated properties that must be considered for the structural applications. Superior thermal, chemical and dielectric properties are observed in the glass-ceramic materials. There is much research on the phase separation, nucleation and crystallization in the glasses. The most detailed investigation was done in the $\text{Li}_2\text{O}-\text{SiO}_2$ binary system in the aspect of controlled crystallization of the glass system. $\text{SiO}_2-\text{Al}_2\text{O}_3-\text{K}_2\text{O}$ system can be subjected to controlled crystallization to obtain lithium disilicate based dental ceramics. In the last sub-section, P_2O_5 as a nucleating agent, was examined. P_2O_5 has a significant role for lithium disilicate crystallization. Fine-grained interlocking microstructures are obtained with the addition of P_2O_5 nucleating agent. Thus high mechanical strength can be determined in these samples.

Each of the dental ceramics has an important role in the area of the dentistry. The advantages and the shortcomings of each of these dental ceramics are present. In the author's opinion, among all-ceramic dental restorations, lithium disilicate based glass-ceramics introduce more advantages than other dental ceramics and also metal-based restorations.

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Corresponding Author

Dr. Ertug Burcu

Department of Mechatronics Engineering,

Nisantasi University,

Istanbul, TURKEY

Email id: burcu.ertug@nisantasi.edu.tr