

Research and Development Ivoclar Vivadent AG FL-9494 Schaan / Liechtenstein

airceranic ...airou need

# TABLE OF CONTENTS





Frank Rothbrust IPS e.max ZirCAD Zirconium oxide – "White gold"



Marcel Schweiger IPS e.max Ceram The universal veneering ceramic – One for 4



Marcel Schweiger IPS e.max ZirPress The pressable veneering ceramic



Harald Kerschbaumer IPS e.max The development in terms of dental technology and shading



Dr Alexander Stiefenhofer | Harald Kerschbaumer IPS e.max Two clinical cases

## EDITORIAL



Dr Thomas Völkel

With the launch of IPS Empress in 1991, Ivoclar Vivadent achieved a breakthrough in the development of all-ceramic dental restorations. IPS Empress is a glass-ceramic, which is composed of a glass matrix and reinforcing crystalline contents. The crystals in IPS Empress are made of leucite. The ceramic exhibits sufficient strength for the fabrication of crowns, inlays, onlays and veneers. Within these indications, IPS Empress has been replacing metals and alloys. Consequently, the biocompatibility and aesthetics of dental restorations has been considerably improved. IPS Empress restorations are fabricated by means of an efficient heat-pressing technique, which endows the restorations with a high accuracy of fit. This processing technique has proven itself for all-ceramic restorations. Although IPS Empress continues to be the market leader in this segment, 25 competitive systems have been developed in the meantime.

In the further development of all-ceramics, particular attention has been paid to increasing the strength and perfecting processing methods for the fabrication of restorations. A stronger material has been achieved in the form of the lithium disilicate glass-ceramic IPS Empress 2. The strength of this ceramic enables the fabrication of three-unit anterior bridges if the stipulated layering thicknesses and connector dimensions are observed.

Even higher strength values are achieved with oxide ceramics. The strength of yttrium-stabilized zirconium oxide comes close to that of metals. It is already recognized as a fully-fledged metal replacement. Clinical data covering longer observation periods are still rare but look promising.

In recent years, CAD/CAM procedures have continuously gained in popularity. New systems that allow the shape of teeth to be scanned accurately and modified are gaining in importance on the market. ProCAD from Ivoclar Vivadent is a leucite ceramic which is milled in CEREC units. The advantage of this method is that the restorations can be fabricated chair-side. Thus, the treatment time is considerably reduced. The use of oxide ceramics, such as zirconium oxide, is only advisable if they are machined.

The new **IPS e.max** all-ceramic system allows the user to cover the entire range of indications with only one system. The frameworks are fabricated using either materials for the press or the CAM techniques.



# EDITORIAL

- IPS e.max Press is a lithium disilicate glass-ceramic for the press technique. Due to its fracture strength of 400 MPa, the material is suitable for the fabrication of 3-unit bridge frameworks up to the second premolar as a distal abutment.
- IPS e.max CAD is also based on lithium disilicate glass-ceramics. The blocks are in a pre-crystallized metasilicate phase. The blocks appear blue in colour, are soft and can thus be quickly milled to crown copings. In addition, they prolong the service life of the tools used. The subsequent crystallization firing endows the blocks with the final lithium disilicate form. In this crystallization process, the blocks acquire their strength of 360 MPa.
- IPS e.max ZirCAD is a pre-sintered zirconium oxide block that contains small quantities of yttrium oxide to stabilize the meta-stable tetragonal phase. In the pre-sintered state, the block features a porous, chalk-like morphology. After milling the framework, the zirconium oxide is densely sintered in a process with temperatures up to 1500 °C. The resulting material has now acquired its final strength of more than 900 MPa. During the sintering process, the volume shrinks by approx. 20%. This reduction in volume is already taken into account in the milling program.
- Zirconium oxide is only used for the framework. The white-opaque surface has to be covered with a more translucent ceramic to achieve aesthetic restorations. Such a build-up is possible with IPS e.max ZirPress. The IPS e.max ZirPress ingots are pressed on the zirconium oxide frameworks in the proven IPS Empress technique.
- IPS e.max Ceram is the layering material that is suitable for sintering on all the IPS e.max materials.

The present Report No 17 is an addition to a series of issues about allceramics. Number 6 described "IPS Empress – A new ceramic technology". Number 10 covered "IPS Empress: Material and clinical science". "IPS Empress 2: The all-ceramic bridge and more..." was the title of No 12. While a collection of general articles on all-ceramics was published in the previous issue No 16, Report No 17 is devoted to the material properties and processing of the entire IPS e.max system.

The Head of Ceramic Development, Marcel Schweiger, and his coworkers Frank Rothbrust and Dr Harald Bürke describe the properties of ceramics, the theoretical background and the procedures during the fabrication and preparation of restorations. Harald Kerschbaumer, Master Dental Technician, focuses on dental technical aspects, particularly in the aesthetic perfection of restorations. Finally, Dr med dent Alexander Stiefenhofer presents two clinical cases which he has documented with photos.

The articles, however, can only convey a few aspects of the materials and their compatibility with each other. For further information, please refer to the cited literature. It goes without saying that issue No 17 will not be the last report about all-ceramics.

Dr Thomas Völkel Scientific Services





# IPS e.max Press and IPS e.max CAD

Harald Bürke

D



Two state-of-the-art glass ceramics

## 1 Introduction

IPS e.max Press and IPS e.max CAD are the two framework materials from the IPS e.max range that offer medium strength and maximum aesthetics.

IPS e.max Press ingots are particularly suitable for single tooth restorations in the anterior and posterior region as well as anterior bridges with a maximum of 3 units up to the second premolar as the abutment tooth. The current range of indications for IPS e.max CAD includes the fabrication of anterior crown copings. The results of currently conducted clinical studies will show whether the range of indications can be further extended. Both materials belong to the same group of materials. Depending on the facilities available in the dental laboratory or the preferences of the dental technician, either the well-known IPS Empress press technique or the CAD/CAM technique can be chosen.

The results after veneering are very similar, so that it is even possible to combine both techniques in one patient.

After a section with general information, the present article will provide a more detailed description of the two materials. Special emphasis will not only be placed on the possibilities of glass ceramics, but also on their limitations. Their potential can be fully tapped only when the preparation guidelines are strictly observed.

# The building blocks of allceramics: Ceramic – glass – glass ceramic

Apart from acrylics, composites and metal alloys, all-ceramic materials are increasingly used in dental prosthetics today. By definition, ceramic is an inorganic, non-metallic material which is solidified by means of a firing process and has a crystalline content of at least 30 % [1]. In the English language, the term has an even broader meaning as there is no limitation with regard to the crystalline content.

## Table 1:

Possible classification of ceramic materials according to different characteristics (examples)

Manufacturing technique:	Sintering
	Reaction sintering
	Hot isostatic pressing
	Infiltration
	Melting
	Crystallization
Structure:	Crystalline
	Glassy-amorphous
	Multiple-phase partially crystalline
Composition:	Oxide ceramic
	Non-oxide ceramic
	Nitride ceramic
	Silicate glass
	Phosphate glass
	Borosilicate glass
	Alumosilicate glass ceramic
	Lithium silicate glass ceramic
Function:	Consumer good
	Heat exchanger
	Heating conductor
	Electroceramic
	Optoceramic
	Dental ceramic
Property:	Strength
	Resistance to temperature changes
	Electrical conductivity
	Chemical resistance
	Thermal conductivity
	Shade
	Translucency



Various criteria are applied to differentiate between the different materials in this large group. The criteria may be eg production method, composition or function (Table 1).

## 2.1 Ceramics

Ceramics in the narrower sense are predominantly crystalline products which are produced with the help of a sintering procedure. For this purpose, the ceramic powders are mixed with a bonding agent and the required, slightly larger moulds are produced. Various moulding procedures are available, eg uniaxial pressing, extrusion moulding, slip casting, injection casting or cold/hot isostatic pressing. The pre-formed materials are fired at high temperatures. During this "sintering" process, diffusional mass transport occurs in the course of which porosity is eliminated. The driving force for this process is a reduction in surface energy. Each system strives to reach the lowest possible energy level. The finer the initial powder, the smaller the gaps that have to be closed, the larger the surface area that needs to be decreased and the higher the driving force for the sintering process. Finer powders show more sintering activity. The grain boundaries also show a higher energy level than the crystal, eventually leading to crystal growth in the final stage of sintering. Large crystals grow at the cost of small ones.

#### 2.1.1 Solid-state sintering

In pure solid-state sintering, the sintering temperatures are lower than the lowest melting temperature of the components involved. Diffusion occurs at the surface of the powder particles and is brought about by rearrangement within the crystal lattice as well as through the gas phase by evaporation and condensation.

### 2.1.2 Liquid-phase sintering

A liquid phase is often used to accelerate the sintering process. A component, of which the mixture usually only contains a small amount, melts at the sintering temperature and wets the solid particles. This enables grain rearrangement in the initial stage. As sintering proceeds, mass transport occurs through solution and precipitation, which considerably accelerates the kinetics of sintering.

### 2.1.3 Reaction sintering

We speak of reaction sintering if new components are formed during the sintering process. This is useful if, for example, the desired ceramic shows little sintering activity. Some-times, the changes in volume that occur as a result of the reaction are used in a targeted fashion to counteract the shrinkage.

#### 2.1.4 Porcelain

The porcelain firing process represents a special case. The crystal phases (quartz, mullite, feldspar, aluminium oxide) are surrounded by a large amount of glass phase. Apart from solution and precipitation, different chemical reactions occur. The high viscosity of the liquid phase prevents deformation of the fired material.

#### 2.2 Glass

In the traditional sense, glass is an inorganic non-metallic, non-crystalline melt product. In this context, the picture of the "frozen super-cooled liquid" is helpful. Condensed material has a higher chemical order than glass. Crystals are characterised by a particularly high order. There is a fixed spatial relationship between all of their atoms, be it the nearest neighbours or more remote atoms. A chemical short-range and long-range order exists. In the case of glasses and liquids, the long-range order is completely annihilated. However, a short-range order exists, since the distance from an atom to its nearest neighbour is predetermined. The glass formation is linked to the kinetics of crystallization. Even though the crystals strive to reach the higher order if the temperature falls below the melting point, crystallization is prevented due to the limited mobility of the structural units at lower temperatures.

#### 2.2.1 Glass structure

The prevailing models relating to the structure of glass are based on the microcrystallite hypothesis and the network hypothesis. According to the microcrystallite hypothesis, glass is composed of small, ordered structural units of the size of a unit cell, which are separated from each other by un-ordered areas. The network hypothesis is based on the assumption that the structural elements are randomly connected (Fig 1). Depending on the glass composition, both hypotheses are justified. [4].





Fig 1: Amorphous silica structure (b) compared to crystal structure (a) [4]



Fig 2: Left: Glass network with sodium (Na) as network modifier Right: Schematic representation of the replacement of SiO<sub>2</sub> by Al<sub>2</sub>O<sub>3</sub> in a sodium silicate glass [2]

#### 2.2.2 Composition of glasses

Standard glass is composed of network formers and at least one network modifier.

Usually, silicon dioxide is used as the network former. SiO<sub>4</sub> tetrahedrons which are connected via their corners thus form the glass network. Further commonly used network formers are  $B_2O_3$  or  $P_2O_5$ , either alone or in combination with SiO<sub>2</sub>.

The network modifiers serve as fluxing agents. By breaking the Si-O-Si bonds, they ensure that the glass melts more easily. Alkali as well as alkaline-earth oxides are used for this purpose (Fig 2).

Depending on the composition of the glass, some bivalent and trivalent oxides can either be incorporated into the network or produce gaps. These oxides are called intermediate oxides. By means of the composition, the properties of glasses can be varied within broad limits.

### 2.2.3 Bulk glass technology

As raw materials, individual oxides, hydroxides or carbonates are used. They are thoroughly mixed and jointly melted at high temperatures. Initially, the chemicals react with each other, hydroxide groups are segregated and carbon dioxide is released. Eutectic mixtures help to decrease the melting temperature. After initial melting, the melt must be mixed and homogenized. Trapped air or gas should be able to escape. This procedure is called purification. For this purpose, the melt must have a very fluid consistency. Once the glass melt is homogeneous and free of air bubbles, the temperature is reduced, so that the glass becomes more viscous. Depending on the moulding process used, a clearly defined viscosity must be achieved. The glass, which demonstrates a viscosity similar to that of honey, is poured into eg a separable steel mould. The material is left to cool in this mould until it has reached a temperature which ensures that no deformation occurs during removal. The corresponding temperature range is called the transformation temperature range.

The glass is then frozen to a degree that macroscopic deformation cannot take place. However, the elements of the glass structure are still sufficiently flexible to relax the thermal stress. In a cooling furnace which, depending on the glass type, is operated at a temperature of between approx 400–600 °C, this stress is relieved before the glass is finally cooled down to room temperature.

#### 2.2.4 Sinter glass technology

When coating steel (enamel) or ceramic (glaze), glass powder is sintered onto a substrate. When producing the glass powder, the raw materials are melted at high temperatures as usual. However, the glass melt is not poured into a mould but quenched in water. Due to the thermal shock, the glass fractures. The resulting granulate is milled into a fine powder. In the case of glass, the sintering process is particularly simple. A reduction of the surface area is achieved by viscous flow processes. The open porosity found initially is thus quickly eliminated. Further densification is dependent on whether or not gas is contained in the remaining closed pores. The rate of diffusion of the gases determines the speed of the final densification.



### 2.2.5 The characteristics of glass

A typical characteristic of glass is a largescale homogeneity which is characterized by the absence of grain or phase boundaries. Furthermore, glass is spatially isotropic, which means that its properties are the same in all directions of space.

The absence of grain boundaries is the reason for the disastrous fracture behaviour of glass. As long as the surface is intact, glass can exhibit extremely high strength. Virgin glass fibres, for example, have a tensile strength under vacuum of 12,000 to 16,000 MPa. However, as soon as a crack is initiated, it may spread unhindered. The typical shell-shaped fracture pattern results. In order to increase the strength of glass, it is thus particularly important to improve its scratch resistance. This prevents the surface of glass products from becoming damaged prematurely and the products from being weakened.

Other properties, such as chemical resistance, light refraction and thermal expansion are significantly influenced by the chemical composition.

#### 2.3 Glass ceramics

In general, crystallization phenomena represent defects in glassy products. However, in the production of glass ceramics, one or more crystalline phases are deliberately precipitated. Glass ceramics are polycrystalline solids produced by means of a controlled crystallization of glass [3]. A typical characteristic of glass ceramics is that the crystals do not grow sporadically, randomly or accidentally within the glass matrix. This would lead to uncontrolled crystal growth and result in an inhomogeneous structure with inferior properties.

Initially, a glass of suitable composition is melted and moulded into a still glassy ingot using the conventional moulding procedure. For the subsequent two-stage temperature treatment, the temperatures are chosen in such a way that first the temperature range of maximum nucleation is passed and then that of maximum crystallization. The crystal size and number can best be controlled by clearly separating the temperature ranges of maximum nucleation and maximum crystal growth (Fig 3). The end product is a ceramic whose properties are determined by the type, morphology, size, content and degree of cross-linking of the precipitated crystal phase(s) as well as the remaining glass phase. The objective of this crystallization process is eg an increase in mechanical strength, resistance to temperature changes or the targeted adjustment of thermal expansion and optical or electrical characteristics.

The difference between the structure of glass ceramics and conventional ceramics is that glass ceramics are exclusively created by a crystallization of the glass phase, so that a particularly fine-grained and homogenous structure is achieved. Their high crystalline content distinguishes glass ceramics from glasses.

### 2.3.1 Volume nucleation

In order to manufacture glass ceramics, specific glass compositions are often melted in conjunction with special oxides as nucleating agents, processed like glass and moulded into a transparent glass block. The additives dramatically accelerate the nucleation that occurs within the glass in various ways. The difficult thing in developing a glass ceramic is to find a suitable nucleating agent for the crystal phase. Frequently, oxides are melted together with the glass which show only limited solubility in the corresponding glass. At a low temperature, many nanocrystals of this phase form initially. These nanocrystals then constitute the substrate on which the actual crystals are grown. This process is called heterogeneous nucleation.



Fig 3: Dependence of nucleation and crystal growth on temperature



The fact that amorphous phase separation is of particular importance for the nucleation process has been pointed out by Vogel, for example [4]. According to him, crystallization is initiated within the microdrops and initiates bulk crystallization which leads to the formation of crystallites of uniform size.

## 2.3.2 Surface nucleation

Many crystal phases are difficult to produce in a controlled way using bulk nucleation procedures. Even if nucleation-supporting additives are used, the nucleation rate of species such as leucite or anorthite is so low that the nucleation process is uneconomical because of the long processing times. In this specific case, crystal growth is initiated at the surface, so that the use of mechanically milled powder particles eventually leads to the formation of a glass ceramic. In the process, particular attention is paid to generating crystal nuclei on the surface of the powder.

In some systems, it is advantageous to densely sinter the glass powder first and then increase the temperature to a level that ensures the desired crystal growth – in particular if the crystal phase shows little sintering activity. In other systems, the sintering and crystal growth processes overlap, and in some systems crystallization of the powder particles is initiated first, while the sintering process is conducted subsequently.

## 2.3.3 Classification of glass ceramics

Glass ceramics can be classified according to their chemical composition, for example, or according to their field of application. Beall suggests classifying commercial silicate glass ceramics into three groups based on the mainly crystallizing mineral phase [12]:

- Silicates such as lithium metasilicate, lithium disilicate, enstatite (MgSiO<sub>3</sub>), diopside (CaMgSi<sub>2</sub>O<sub>6</sub>) or wollastonite (CaSiO<sub>3</sub>).
- Aluminium silicates with the main phases cordierite, beta-quartz, beta-spodumene, beta-eucryptite and anorthite
- Fluorosilicates; these include fluorcontaining mica glass ceramics based on fluor-phlogopite (K,Na Mg3AlSi3O10F2) and tetra silicate mica (KMg2,5Si4O10F2), as well as fluor-containing chain silicates such as canasite and fluor-richterite.

The best-known and most widely used glass ceramics are those based on the Li<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> system, which possess high thermal shock resistance due to their low thermal expansion. They are used for eg hot plates, furnace windows or telescope mirrors [5].

# The materials of the IPS e.max System

3

The IPS e.max System offers dental technicians unique freedom. With only one veneering ceramic, consistently aesthetic results can be achieved on different framework materials. The dental technician is free to choose between resistant lithium disilicate glass ceramics and high-strength zirconium oxide. While the oxide-ceramic zirconium oxide can only be shaped using a milling procedure, resistant glass ceramics can either be processed using the well-known IPS Empress hot pressing procedure or milling techniques.

## 3.1 Requirements placed upon framework materials

The requirements are based on the product profile and ISO 6872. Central issues are mechanical strength, chemical resistance as well as aesthetic properties. These desired characteristics are listed in Table 2.

#### Table 2:

## Requirements placed upon framework materials

Biaxial strength	> 360 MPa
Fracture toughness	> 2,5 MPm <sup>0,5</sup>
Low chemical solubility acc. to ISO 6872	< 100 µg/cm²
Pressability in EP 500, EP 600 and selected competitor furnaces	900 °C – 940 °C
Machinability in widely used CAD/CAM units	Edge stability, tool wear, milling times
Coefficient of thermal expansion: Compatibility with IPS e.max veneering ceramic, which can also be used for ZrO <sub>2</sub>	10.0–10.8 *10 <sup>.6</sup> K <sup>.1</sup>
Shading of the final product according to aesthetic requirements	





### 3.2 IPS e.max Press

In the production of IPS e.max Press ingots, new process technology is employed. The ingots are no longer manufactured via a powder stage as in the case of IPS Empress and IPS Empress 2, but by bulk casting. As a result, a product can be manufactured that is entirely porefree.

# 3.2.1 Ion colouring and opaquing mechanism

This new production procedure does not involve the addition of colour pigments, as these would melt at the melting temperature. The product thus shows less defects, which at the same time enables higher strength and higher translucency to be produced than with conventional sintering technology.

The colour is achieved with polyvalent ions which are atomically dissolved in the glass. The colour centres interact with each other and with the glass matrix through redox processes. In this context, the right combination and concentration of the colouring ions is essential. Also, the melting conditions must be precisely defined.

The desired opacity of the mediumopacity ingots is adjusted by means of a phase separation process which occurs within the remaining glassy phase during slow cooling in the press mould. The same effect originally led to a formation of strong, undesired opalescence in translucent glass ceramics.

## 3.2.2 Processing

Processing in the dental laboratory is done using the well-known IPS Empress technology. This is characterised by high precision of fit.

The new press ingots are offered in 3-g and 6-g sizes. In contrast to the IPS Empress and IPS Empress 2 ingots, which feature a diameter of 11.8 mm, the diameter of the new ingots has been enlarged to 12.8 mm. Correspondingly, the press system, ring base and press plunger have been enlarged to 13 mm.

However, it should be kept in mind that due the enlargement of the diameter, the press pressure has been reduced by 15 %. This may lead to longer pressing times, particularly in conjunction with furnaces of other manufacturers. However, it would be wrong to increase the temperature so that it exceeds the prescribed temperature, as this could lead to the formation of a thicker reaction layer in the area of contact with the investment material. In extreme cases, exposure to excessively high temperatures could even damage the structure of the glass ceramic which would lead to a reduction in strength. If problems occur, the calibration of the furnace should be checked first. Similarly, it should be checked whether sufficient pressing force is applied.

For restorations with thinly tapered margins, thick and short sprues should be used as a lot of press force is lost in conjunction with long sprues. This loss of force is proportional to the length of the sprue, while the diameter affects the pressing speed to the fourth power. If required, the diameter of the sprue may be slightly reduced at the point of contact with the object to reduce the time required for grinding. The following sketch illustrates how sprueing influences the press behaviour (Fig 4).

After careful divesting, investment residue needs to be removed with IPS e.max Press Invex Liquid - a procedure commonly used in conjunction with lithium disilicate materials. The indicated time should be strictly observed, as compared to IPS Empress 2, the new product features an improved chemical resistance which also affects the treatment with Invex Liquid. It is recommended to place the restorations in a small, suitable container and cover them with fresh liquid each time. Subsequently, the solution can be neutralized as indicated and discarded.

#### Flow of a fluid in a pipe (Hagen Poiseuille)

•  $dV/dt = \pi r^4 \Delta \rho / 8\eta I$ 

r = radius

 $\rho$  = pressure (press force: - 15% in 6-g ingots)  $\eta$  = dynamic viscosity

Sprues:	Diameter	Length	Pressing speed
	2.0 mm	8.0 mm	1.00
	2.0 mm	6.0 mm	1.33
	2.5 mm	4.0 mm	4.88
	3.0 mm	6.0 mm	6.75

Fig 4: Repercussions of the sprue dimensions on the pressing force, sprues with a diameter of 2.0 mm and a length of 8.0 mm correspond to standard 1.00





### 3.3 IPS e.max CAD

CAD/CAM techniques are increasingly employed in the fabrication of dental restorations. Systems such as CEREC 3, inLab, Kavo Everst and others are technically mature and enable economical, high-quality restorative results. The intention was to offer a machinable high-strength glass ceramic in addition to the high-strength glass ceramic IPS e.max Press.

### 3.3.1 The task

In the development of a suitable glassceramic version for computer-aided manufacturing, one general problem had to be solved. The higher the strength a material demonstrates, the more difficult it is to machine it. The biaxial flexural strength of the new pressable ceramic IPS e.max Press, which is manufactured using bulk casting technology, is above 400 MPa, while its fracture toughness is 2.5-3.0 MPa m<sup>0.5</sup>. Tests have shown that this material cannot be machined in most units. In general, the first set of tools is already worn after the first restoration has been milled. In addition, machining times are long, which renders the procedure uneconomical. The task was to develop a material that would enable quick, easy and precise machining, while a short, subsequent tempering process would impart the material with the required high strength [8-10].

#### 3.3.2 The approach

The mechanical properties of a glass ceramic material are closely linked to its microstructure. A low content of isolated, preferably plate-shaped crystals dispersed in the glassy matrix represent predetermined fracture points. Wear of the material occurs tangentially to the force acting on it. Uncontrolled crack propagation is halted by the crystals [11]. A high fraction of needle-shaped, close-knit solid crystals generally makes crack propagation difficult. A large amount of new surface area has to be created - a process which consumes considerable energy. Consequently, such structures show a high fracture toughness.

#### 3.3.3 The solution

By two-stage crystallization using a controlled double nucleation process and a special composition of glasses, lithium metasilicate crystals are precipitated in a first step. The resulting glass ceramic demonstrates excellent processing properties. In a second step, the metasilicate phase is completely dissolved and the lithium disilicate crystallizes. As virtually no shrinkage occurs during this conversion process, the fit of the restoration can be previously adjusted. In the course of the conversion process, the material acquires a high strength of more than 300 MPa and a fracture toughness of 2.0–2.5 MPa m<sup>0.5</sup>. The blue shade of the metasilicate glass ceramic disappears and the desired tooth shade is achieved. This colour change is attributable to a change in the coordination number of the colouring ions (Fig 7).

# 3.3.4 Complex crystallization of IPS e.max CAD

The following section will provide more details about the re-crystallization process and related solidification.

The development is based on glass ceramic material created from a lithium disilicate system. In Table 3, the composition of the material is shown. SiO2 and Li2O represent the crystal-forming components, while P2O5 is added as a nucleating agent. The remaining oxides are used to control the properties of the residual glassy phase. They serve to adjust the shade and translucency. Figure 5 shows the binary phase diagram for the SiO<sub>2</sub>-Li<sub>2</sub>O system. Within the system, three stable binary oxides occur: lithium orthosilicate (Li4SiO4), lithium metasilicate (Li2SiO3) and lithium disilicate (Li<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>). The two latter oxides play a role in the crystallization of IPS e.max CAD. When relating it to the binary system SiO<sub>2</sub>-Li<sub>2</sub>O, the composition of the material is close to that of



Fig 5: Phase diagram Li2O – SiO2

disilicate. However, it is well-known that in materials containing between 5 and 30 mol % of Li<sub>2</sub>O, a miscibility gap occurs [cfr 6, p 75; 7, p 76]. This means that when the melt is cooled, areas rich in Li<sub>2</sub>O and SiO<sub>2</sub> form. This phase separation is a prerequisite for the simultaneous nucleation of lithium metasilicate and lithium disilicate. In conjunction with certain compositions, it is possible to adjust the duration and temperature of nucleation in such a way as to achieve a defined nucleation density and a defined ratio of crystal nuclei. The subsequent crystallization process comprises two stages. During the first stage, lithium metasilicate crystallizes almost exclusively, while the lithium disilicate crystal nuclei remain in the glassy matrix in a latent state (Figs 6 and 7).

During the second crystallization stage, the lithium disilicate nuclei finally grow into crystals at higher temperatures. As the composition of the base glass corresponds to that of lithium disilicate, all metasilicate crystals are consumed in this process. The desired lithium disilicate glass ceramic forms.

Expressed in the form of a chemical reaction, lithium disilicate is created from the glassy phase of lithium metasilicate and SiO<sub>2</sub>: Li<sub>2</sub>SiO<sub>3</sub> + SiO<sub>2</sub> = Li<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>.

Expressed in terms of volume, a glass ceramic material containing 60–70 % of disilicate crystal phase and 30–40 % of glass phase is created from a glass ceramic material containing 30–40 % of metasilicate crystal phase and 60–70 % of glass phase.

### Table 3:

Chemical composition of the IPS e.max CAD glass ceramic in % mass

SiO <sub>2</sub>	57 – 80	
Li <sub>2</sub> O	11 – 19	
K2O	0 – 13.5	
ZnO	0 - 8	
P2O5	0 – 11	
ZrO <sub>2</sub>	0 - 8	
Additional components Colouring oxides	0 – 12	



Fig 6: Crystallization pattern of IPS e.max CAD; the first temperature treatment is carried out industrially, while the final crystallization takes place in the dental laboratory (LS = lithium metasilicate, LS2 = lithium disilicate)



Isolated LS nuclei after the first crystallization stage enable simple, precise machining



Cross-linked LS2 crystals result in high fracture strength and toughness



In the Ostwald ripening stage, a further temperature increase produces crystal growth



Nuclei from various crystal species

develop independently

Transparent glass block after nucleation of LS and LS2





Glass ceramic block after the first crystallization stage



CAD/CAM-manufactured restoration made of LS glass ceramic



Restoration after conversion into LS2 glass ceramic at 850 °C

Fig 7: Complex crystallization of IPS e.max CAD (LS = lithium metasilicate, LS2 = lithium disilicate)



### 3.3.5 Processing

The nucleation and initial crytallization process establishes the basis for the final lithium disilicate glass ceramic product. Nevertheless, the temperature and crystallization regime must be strictly observed. Excessively high temperatures may lead to a plastic deformation of the restoration. То counteract this, supporting paste or zirconium oxide spheres are used. Rounded edges and severe vitrification of the surface are an indication of too high firing temperatures. If the firing times are too short or the furnace temperature is too low, the shades may not be precisely matched. In contrast, the firing parameters have hardly any impact on the strength.

The special lvoclar Vivadent firing tray made of SiN ceramics is capable of balancing the temperature profile due to its good thermal conductivity and relatively high thermal mass, independent of the number of objects placed in the furnace. The use of this firing tray in conjunction with a calibrated ceramic furnace is both essential and sufficient to successfully conduct the final crystallization of IPS e.max CAD.

#### 3.3.6 Sandblasting

A whole section will be dedicated to this issue, as very different views exist. Many dental technicians are used to sandblasting their work patterns to create clean and rough surfaces for the subsequent working steps.

However, if glass and glass ceramic surfaces are sandblasted, so-called median cracks are produced. These cracks are perpendicular to the surface and weaken the restoration due to the notch effect. In the case of glass, this leads to a tremendous reduction in strength, but also glass ceramics can be weakened, even though the resulting cracks are stopped by the incorporated crystals. Therefore, glass ceramic

objects should only be sandblasted in case of absolute necessity. IPS e.max CAD does not need to be sandblasted prior to further processing, layering or glazing. It is sufficient to clean the surface with eq a steam blaster. After immersion in Invex Liquid, restorations fabricated with IPS e.max Press should be blasted with glass beads at maximum 2 bar pressure to completely remove the reaction layer. To minimize the impact of the abrasive medium, the surface should not be positioned vertically, but at an angle. After sandblasting, the surface needs to be cleaned with steam again or in the ultrasonic unit.

After finishing, the restoration should not be sandblasted (Figs 9 and 10). The retentive surfaces required for cementation or adhesive luting are created by etching with hydrofluoric acid (Fig 8). Investigations have revealed that hydrofluoric acid etching hardly affects the strength of glass-ceramics (Table 4). It is interesting to note that (careful) grinding with highquality fine-grain diamond burs is even less critical than sandblasting. However, any generation of heat during the grinding procedure should be strictly avoided.



Fig 8: Etched IPS e.max CAD surface - a retentive pattern has been created by etching with hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent)



Fig 9: IPS e.max CAD surface after sandblasting – deep damage of the glass ceramic is noticeable



Fig 10: IPS e.max CAD surface after sandblasting and etching – the damaged areas cannot be eliminated by etching - this is reflected in the biaxial strength values (Table 4)

### Table 4:

Biaxial strength of IPS e.max CAD after different surface treatments						
	Surface polished with SiC paper grade 1000	Etched surface	Sandblasted surface	Sandblasted and etched surface		
Biaxial strength [MPa]	554 +/- 78	520 +/- 98	232+/-18	235 +/-25		



## 3.3.7 Microstructure

Figures 11 to 13 show SEM pictures of glass-ceramics in different processing stages.



Fig 11: SEM picture of the glass ceramic after completion of the first crystallization stage at 650 °C; approx 30 % of LS crystals in the residual glassy phase (crystals etched with HF)



Fig 12: SEM picture of the glass ceramic after completion of the second crystallization stage at 850 °C; approx 70 % of LS2 crystals in the residual glassy phase (glass phase etched with HF)



Fig 13: SEM picture of the glass ceramic pressed using the IPS Empress technique at 920 °C; approx 70 % of LS2 crystals in the residual glassy phase (glass phase etched with HF)

## 3.4 Properties of framework materials

The properties and respective values achieved by IPS e.max CAD and IPS e.max Press are indicated in the table below. The requirements stipulated in the product profile as well as ISO 6872 could be met.

## Table 5:

## Properties of framework materials

	IPS e.max CAD machinable	IPS e.max CAD crystallized	IPS e.max Press
Biaxial strength [MPa]	100–160	300-420	400-480
Fracture toughness [MPam <sup>0,5</sup> ]	0.9–1.1	2.0–2.5	2.5-3.0
Elastic modulus [GPa]	-	90–100	90–100
Vickers hardness [MPa]	5300-5500	5700-5900	5700-5900
Solubility acc to ISO 6872 [µg/cm <sup>2</sup> ]	-	30–50	30–50
Crystallization temperature / Press temperature	-	850 °C	920–925 °C
Manual processing	with rotary diamond instruments	with rotary diamond instruments	with rotary diamond instruments
CTE 100–400 ° [10·6K·1] CTE 100–500 °C [10·6K·1]		9.75–10.55 10.20–10.70	9.75–10.55 10.20–10.90
Thermal shock test		> 160 °C	> 160 °C
Shading of the final product according to aesthetic requirements	Blue-purple shade of shaded versions	A-D and Chromascop shades in various degrees of translucency	A-D and Chromascop shades in various degrees of translucency



## 4. Literature

- Hennicke, H.W.; Zum Begriff Keramik und zur Einteilung keramischer Werkstoffe. Ber. Dtsch. Keram. Ges. 44 209-201(1967)
- [2] Scholze H. Glas; Springer Verlag Berlin, 1988
- [3] P. W. McMillan; Glass-Ceramics; Academic Press Inc. London 1979
- [4] W. Vogel; Glaschemie; Springer-Verlag Berlin 1992
- [5] Hans Bach; Low Thermal Expansion Glass Ceramics; Scott Series on Glass and Glass Ceramics, Springer Verlag Berlin (1995)
- [6] Wolfram Höland and George Beall; Glass-Ceramic Technology; The Am. Ceram. Soc. Ohio (2002)
- [7] Z. Strnad; Glass-Ceramic Materials, Glass Science and Technology 8; Elsevier Amsterdam – Oxford – New York – Tokyo (1986)
- [8] H. Unuma, K. Miura, K. Kodaira; Improvement of Mechanical Properties of Machinable Glass-Ceramics through Postmachining Heat treatments; J. Am. Ceram. Soc. 75 [8] 2300-2301 (1992)
- [9] M. Reise; Untersuchungen zur Verfestigung und Aushärtung maschinell bearbeitbarer Glaskeramiken; Dissertation; Technische Universität Darmstadt (1999)
- [10] H. Bürke; TEM-Grundlagenuntersuchungen zur Gefügeentwicklung in aushärtbaren Glimmerglaskeramiken; Dissertation, Universität Würzburg (2004)
- [11] D. G. Grossmann; The Formation of Chips in a Machineable Glass Ceramic; Glass Technology Vol. 24 No. 1 11-13 (1983)
- [12] G. H. Beall; Design and Properties of Glass-Ceramics; Annu. Rev. Mater. Sci. 22 (1992) 91-119







# IPS e.max ZirCAD Zirconium oxide – "White gold"

## 1 Introduction

Zirconium oxide has been used for more than 20 years as a high-performance material in industries such as engineering, sensor technology and medical engineering due to its excellent properties. The reason why it is used in a variety of application fields is due to the crystal-chemical characteristics of zirconium oxide. Based on these properties, Garvie et al. has gone so far as to describe zirconium oxide as "ceramic steel" [1]. In the past few years, in which zirconium oxide has been successfully employed in the dental field as brackets, abutments, framework material and implants, the term "white steel" has often appeared in publications. However, can the term be generally applied to all types of zirconium oxide? And can IPS e.max ZirCAD be described as "ceramic steel"?

## 2 Mineralogical properties of ZrO<sub>2</sub>

Zirconium oxide is a chemical compound of  $Zr^{4+}$  metal ions and the O<sup>2</sup>-oxide ion. The result is an oxidic compound of an ionic character, which – despite the  $Zr^{4+}$  metal ion – does not belong to metals but to the group of non-metallic inorganic substances. It is basically wrong to talk about metal or steel in this context because zirconium oxide is clearly an oxide ceramic with a fine-grain, poly-crystalline structure. The characteristics of  $ZrO_2$  can best be described on the basis of the mineral baddeleyite (ZrO<sub>2</sub>). They are based on the mineralogical structure and the different crystal lattices this material can adopt. In nature, the monoclinic phase is found, which is present in a stable form at room temperature up to 1170 °C. If the temperature increases above this limit, the tetragonal phase is formed and as of 2370 °C, the cubic modification takes place. When the material cools down, the transformation is reversible at slightly different temperatures [2–6] (Fig 1).

It is particularly noteworthy that the volume increases by 3-5% during transformation from the tetragonal modification into the monoclinic phase, only because of the differences in density in both unit cells. The transformation takes place spontaneously in the form of a martensitic transformation as is known from steel ( $\alpha$ -martensite) [2–6].

This specific characteristic of ZrO<sub>2</sub> has been used in technology to develop special materials with selectively controlled properties. For this purpose, however, the mineral baddeleyite could not be used. Instead, synthetic base materials had to be employed. This synthetic ZrO<sub>2</sub> is characterized by the fact that it contains stabilizers, such as Y<sub>2</sub>O<sub>3</sub>, CaO, MgO, CeO<sub>2</sub>, Sc<sub>2</sub>O<sub>3</sub> etc. These doping agents fulfil different tasks depending on the added quantity [7–8]:

 To avoid the transformation of the tetragonal phase into the monoclinic (t/m transformation) up to room temperature and to keep the tetragonal phase metastable at room temperature.



Fig 1: Modification of zirconia at different temperatures





Fig 2: Phase diagram in the ZrO<sub>2</sub>-Y<sub>2</sub>O<sub>3</sub> system according to Yashima et al. [9]



Flg 3: Different types of microstructure for high-performance zirconium oxide ceramics



Fig 4: Crack propagation and stress-induced transformation reinforcement within Y-TZP

- 2. To shift the transformation of the tetragonal phase to lower temperatures
- 3. To keep the cubic phase metastable up to room temperature

Based on the example of the doping additive  $Y_2O_3$  one can see which phases are maintained stable in which areas depending on the added quantities [9, 10]. In the formation of a ceramic microstructure, the structural conditions and the thermal process have to be taken into account to enable the cubic or tetragonal stabilization of the modification up to room temperature (see Fig 2) [11, 12].

## 3 ZrO<sub>2</sub> materials

In the course of the past 30 years, a number of different high-performance materials have been developed. Their functional, biocompatible and mechanical properties have been optimized over time [5, 7, 8]. Four of the main types of these materials are shown below (Fig 3).

- 1. FSZ,CSZ: Fully Stabilized Zirconia
- 2. PSZ I: Partially Stabilized Zirconia I
- 3. PSZ II: Partially Stabilized Zirconia II
- 4. TZP: Tetragonal Zirconia Polycrystals

The transformation-reinforced materials PSZ I, PSZ II and TZP are particularly noteworthy due to their mechanical properties. In these materials, the above described t/m transformation was employed in such a manner that the typical brittleness of ceramics was reduced. A "pseudo-ductile" behaviour of the ceramic increases the fracture toughness in the event of a fracture, similar to steel. Two reinforcement mechanisms can be distinguished here [5, 7, 8]:

- microcrack-induced transformation reinforcement (MR)
- 2. stress-induced transformation reinforcement (TR)

The MR in PSZ I is produced when the stabilization of the tetragonal particles in the cubic matrix is not sufficient and the t/m transformation takes place. The resulting microcracks can impair the propagation of cracks if a macrocrack occurs in the structure, as the energy is distributed among many small microcracks along the process zone. In PSZ II, however, TR takes place: The tetragonal grains are not able to transform in the strained matrix due to their crystallite size and the stabilization. They remain metastable in the matrix. If a



crack penetrates the structure, the tension in the matrix is released and the tetragonal particles can transform. Due to the ensuing volume change, the crack cannot propagate. The fracture toughness of the material increases. Because of this "pseudo-plastic" deformation, the material is also called "ceramic steel" [1].

The following diagram (Fig 4) shows the transformation mechanism of Tetragonal Zirconia Polycrystals TZP. In this type of ZrO2 partially stabilized with 3 mol-% Y2O3, all individual crystals in the structure are maintained metastable up to room temperature to the tetragonal phase. However, the crystallites exhibit the chemical driving power to martensitically transform into the monoclinic phase [5]. Given the surrounding matrix, the crystallites impair each others growth, ie a microstructure that consists of virtually 100% tetragonal crystallites with a mean size of about 0.4 - 0.5 µm is the result. Clearly, the stress of the microstructure can be compensated by the transformation of tetragonal crystallites into the monoclinic phase at the edges of the crack, the "wake zone". Crack propagation is inhibited. The fracture toughness is considerably increased.

In addition to excellent mechanical properties, this material also exhibits biocompatible and optically acceptable characteristics. Therefore, it is used in the dental industry as a framework material in the anterior and posterior region. Hence, it is possible to cover the indication range of metal-ceramics with this material. In this context, the material can be called "white steel". However, it has to be noted that with regard to its hydrothermal durability, the material features poorer properties than metals to some extent [13, 14]. Various publications have shown that pure 3Y-TZP is increasingly prone to t/m transformation during ageing in a temperature range of 70–250 °C in moist surroundings. The result is a loss of strength. A type of corrosion becomes apparent, which is similar to that in metals. Consequently, ZrO2 also features metal-like properties in this respect.

By adding further doping agents, such as Al<sub>2</sub>O<sub>3</sub> or CeO<sub>2</sub> in small quantities, these negative properties of 3Y-TZP have been considerably improved. Consequently, the hydrothermal instability has become irrelevant for the application [15]. IPS e.max ZirCAD (Fig 5) has been doped with a content of about 0.25 wt% Al<sub>2</sub>O<sub>3</sub>. The excellent mechanical properties are maintained despite the additive. At the

4

same time, the ageing behaviour is improved in such a way that there is no risk of strength loss after hydrothermal exposure.

## ZrO<sub>2</sub> in dental applications – processing strategies

Of all the different ZrO<sub>2</sub> materials, 3Y-TZP has established itself for dental applications. The difference in the products is less between the different material parameters of the individual manufacturers than between the type of processing method used and the technology for fabricating a dental restoration with a TZP framework. Basically, two different strategies are applied:

- 1. CAD/CAM processing of ZrO<sub>2</sub> in a densely sintered state or
- 2. CAD/CAM processing of the ZrO<sub>2</sub> white body.

The major distinguishing features are shown in Table 1.

# 5 Properties, also compared to other dental ceramics

Due to the above mentioned reinforcing mechanisms, 3Y-TZP features improved mechanical properties compared to conventional dental ceramics. Both the fracture strength and fracture toughness are significantly higher than in other dental ceramics as is shown in the following diagram (Fig 6). Therefore, the material can be used as a framework material in the anterior and posterior region for single crowns as well as multi-unit bridge frameworks. In addition, 3Y-TZP is biocompatible. Consequently, it can even be used for orthopaedic applications. Moreover, the white frameworks can be layered with, for example, IPS e.max Ceram for the fabrication of highly aesthetic restorations.



Fig 5 IPS e.max ZirCAD B40 and C15



### Table 1:

Comparison of the advantages and disadvantages of the individual processing strategies for  $ZrO_2$  in dental applications

Processing of white body		Processing of HIP ZrO <sub>2</sub>		
Advantages	Disadvantages	Advantages	Disadvantages	
Easy processing	Detailed calculation of shrinkage required	Accuracy of fit, precise milling, no distortion	High processing costs (tools and machine)	
Short processing time, particularly with CAD/CAM	Risk of fracture in white state	Excellent aesthetics, high translucency	Complex reworking procedure with effects on the framework structure	
No complex HIP technology	No control of model fit	Direct model fitting after CAM	Long processing times	
Favourable price	Low translucency	Impeccable structure	Stained base material required	
Post-staining	Low strength	High strength		

## 6 IPS e.max ZirCAD – the procedure

MANUFACTURER

Below is a description of the process steps for the manufacture of a dental restoration made of IPS e.max ZirCAD (Fig 7). Apart from the fabrication itself, the further processing by the user is highlighted in particular. The dental technician is responsible for the computer-aided design of the framework structure and the veneer. He uses precise computeraided machining in a milling unit to create an accurately fitting framework.

#### 10 9 Fracture toughness, Kic/ MPa<sup>1/2</sup> 8 7 3Y-TZP, IPS e.max ZirCAD 6 5 Inceram Zirconia Procera Allceram Inceram Alumina 4 3 IPS e.max Press ŀ IPS e.max CAD 2 IPS Empress 0 0 200 400 600 800 1000 1200 Strength, o/MPa



Fig 6: Comparison of mechanical properties of different dental ceramics

Fig 7: Process diagram for fabricating an IPS e.max ZirCAD restoration

## REPORT

The procedure for processing the white body shows the following characteristics: A block, which has not yet attained its final properties, such as strength, fracture toughness, density etc, but is predensified, is machined. In this presintered state, the block can very easily be machined with suitable grinding instruments. Only after shaping (CAM process) is the block finally densified. Densification means that the material shrinks by a certain degree. This shrinkage has to be taken into account in the CAD/CAM process. Therefore, an enlarged framework is milled from the block. The more detailed the degree of shrinkage in a block is determined, the more precise is the accuracy of fit after final hardening of the framework in the firing procedure.

#### 6.1 Material properties

Below is a description of the most important phases that IPS e.max runs through in the manufacturing process on the basis of the material properties.

# 6.1.1 IPS e.max ZirCAD block – presintered

The base material of IPS e.max ZirCAD is a block in a presintered state, i.e. the material has not been finally densified yet but the individual crystallites - which later form the dense structure - are only connected to each other by sinter necks. The result is a loose, porous structure with 50-% densification (Fig 8). The strength of the material has been optimized in such a way that machining is still easy to conduct on the one hand, while on the other hand, the block does not break in the CAD/CAM process. However, careful handling is required for the finishing process.

After shaping, the material is finally densified in a high-temperature furnace (Fig 9), which has been especially developed for oxide ceramics. The process of final hardening in which the individual crystallites move closer together by diffusion processes due to thermal activation until virtually no pores are left in the structure is called sintering. This process takes place at 1500 °C. What is important in this process is that both heating and cooling rates are exactly coordinated with the actual sintering procedure. Consequently, the thermal process of the equipment has to be accurately adjusted to the processes in the material. Hence, the entire firing process in the Sintramat furnace takes about 8 hours. The material shrinks in every direction by about 20% in relation to the original size.

The following table (Table 2) shows a summary of the most important material properties of IPS e.max ZirCAD in a presintered state.

# 6.1.2 IPS e.max ZirCAD block – sintered to full density

In the following image (Fig 10) a threeunit bridge and a crown coping are shown before and after firing in the sintering furnace. The densification process changes the colour of the framework and endows it with a slight translucency compared to the original material. The fully densified structure shows how the individual tetragonal crystallites are sintered to a dense compound and form a homogeneous microstructure (Fig 11). The grain boundaries can only be made visible by etching, in this case with thermal etching at 1420 °C. Residual porosity is very low. Thus, the number of errors in the structure is minimized. The material is densified to more than 99% of the theoretical



Fig 8: SEM image of a fracture surface of an IPS e.max ZirCAD block, 30,000x magnification



Fig 9: High-temperature furnace Sintramat from Ivoclar Vivadent AG for firing IPS e.max ZirCAD



## Table 2:

Properties of IPS e.max ZirCAD in the presintered state with chemical composition

Density	/g cm-3	3.09 – 3.21
Porosity	/%	47.3 - 49.3
Strength of presintered body	/MPa	50 – 90
Crystallite size	/µm	0.25 – 0.35
ZrO <sub>2</sub>	/wt.%	87.0 – 95.0
Y2O3	/wt.%	4.0-6.0
HfO <sub>2</sub>	/wt.%	1.0 – 5.0
Al <sub>2</sub> O <sub>3</sub>	/wt.%	0.1-1.0

## Table 3:

Material properties of IPS e.max ZirCAD after the firing process in the densely sintered state

Density	/g/cm <sup>-3</sup>	>6.0
Porosity	/%	<0.5
Flexural strength [ISO 6872]	/MPa	>900
Fracture toughness	/MPa m <sup>1/2</sup>	5.5
Hardness HV10	/MPA	13000
Thermal expansion $\alpha$ (100–400°C)	/10 <sup>-6</sup> K <sup>-1</sup>	10.75
Thermal expansion $\alpha$ (100–500°C)	/10 <sup>-6</sup> K <sup>-1</sup>	10.80
Mineralogical phase	_	tetragonal
Crystallite size	/µm	0.50–0.65



Fig 10: Comparison of IPS e.max ZirCAD frameworks before and after the firing process



Fig 11: SEM image of the IPS e.max ZirCAD microstructure sintered to full density (thermally etched at 1420 °C, 20 min)

density. The combination of high densification, low error rate in the structure and the small, mean crystallite size of approx. 0.5  $\mu m$  results in the high fracture strength of the material. In addition, the above mentioned possibility of transforming the tetragonal particles leads to a reinforcing mechanism and thus to a fracture toughness of more than 5.5 MPa m^{1/2}. Other properties are summarized in Table 3.

# 6.2 Sources of error: reasons and effects

Some rules have to be observed in the fabrication of high-quality IPS e.max ZirCAD restorations.

As already described above, the firing process is decisive for the result. It is important that parameters, such as strength, density etc. are controlled by the temperature-time regime. Furthermore, the thermal process in the furnace also has a considerable effect on the accuracy of fit. Consequently, a component with a complex geometry, as is the case with a dental framework, can distort during the shrinkage process. This deformation has to be controlled in such a way that neither distortion nor torque takes place. Therefore, the framework should have free possibility of movement during the firing procedure so that it can move equally into all directions. For this purpose, ZrO<sub>2</sub> beads are used (Fig 13). The beads provide an even support for the framework and allow it to move freely during the sintering process (Fig 12). It is only in this way that the ZrO2 framework sintered to full density acquires its excellent accuracy of fit (Fig 14).

## 6.2.1 Surface finishing

CAM processing with diamond tools produces a surface structure on the ZrO<sub>2</sub> framework, which is also maintained after the sintering process. The image of the surface after milling and sintering shows even milling traces that are produced by finishing the



**Fig 12**: 3-unit bridge framework on IPS e.max ZirCAD on ZrO<sub>2</sub> beads after the sintering process



**Fig 13**: Firing aid for the sintering process: Sintramat ZrO<sub>2</sub> beads



Fig 14: Accuracy of fit of a 3-unit bridge framework after the firing process on the model



surface with diamond tools. Although one might assume that this surface roughness implies great defective areas or even cracks, the magnification (Fig 15) clearly shows that no sharp-edged scrapes are visible within the milling pattern.

In contrast, the surface is severely stressed if it is sandblasted with aluminium oxide particles (Fig 16), producing precipitous structures up to deep cracks (Fig 17). The sharp-edged cracks and defects have a disastrous effect on the fracture behaviour in brittle ceramics and thus reduce the strength of a material.

Garvie et al. [1] and Kosmač et al. [16], however, have been reporting for some time that sandblasting does not have a negative effect on the strength of TZP materials. To the contrary, they argue that the strength is even increased. This reinforcement is achieved due to the phase transformation in the surface from the tetragonal to the monoclinic phase, whereby a superficial compressive stress strengthens the ceramic [16]. The XRD pattern (Fig 18) shows the superficial t/m transformation. Moreover, examinations have proven that apart from the phase transformation Al<sub>2</sub>O<sub>3</sub> particles penetrate into the surface.

What has to be noted here is that a reinforcement by sandblasting is only possible for the framework. Reinforcing the entire restoration with veneering material is not possible. To the contrary, sandblasting the surface prior to veneering weakens the entire framework. The framework ceramic must not be sandblasted before layering, since the bond between the framework ceramics and layering ceramics is weakened. In principle, only the recommended tools must be used for finishing after the sintering process.





Fig 15: Surface of the ZrO<sub>2</sub> framework milled in a CAD/CAM process a) with 200x and b) 5000x magnification, both in a sintered state



Fig 16: Aluminium oxide particles 100 µm for sandblasting



Fig 17: Surface sandblasted with 100-µm aluminium oxide particles at 4 bar



#### 6.2.2 Influence of thermal treatment

The thermal process is decisive for the formation of the microstructure and thus the properties of the material. If the firing process is not accurately controlled, undesirable firing results will be the outcome, which often cannot be visually differentiated. The difference only becomes apparent upon inspection of the microstructure and the comprehensive characterization regarding the dental application. Grain growth processes and the formation of different phases are often not visible to the user.

## 6.3 Applications / indications of IPS e.max ZirCAD

Due to the excellent mechanical properties, IPS e.max ZirCAD is ideal for multiple-unit bridge frameworks. In combination with the IPS e.max Ceram layering ceramic, highly aesthetic dental restorations with a lifelike appearance can be fabricated (Fig 19). Apart from the conventional design of a bridge framework, inlay-retained bridges can be fabricated with the 3Y-TZP material.

Although there are no high demands on the material regarding mechanical properties for crown copings, particularly in the anterior region, zirconium oxide exhibits a big advantage over conventional materials in certain situations. Given the high opacity compared to glass-ceramics, severely discoloured and dark preparations can be covered with zirconium oxide. Hence, the shade of the restoration can be matched to the surrounding teeth. Consequently, the influence of the dark preparation can be compensated.

# 6.3.1 Press-on technique in conjunction with frameworks

In addition to layering the IPS e.max ZirCAD framework with the IPS e.max Ceram veneering material, IPS e.max ZirPress can be used to press onto the framework. The presson technique unites the advantages of the CAD/CAM and press techniques. Layering a ceramic shoulder can be facilitated by pressing a suitable material on a reduced framework. A time-consuming and technically complex layering process is no longer required with the press-on technique. At the same time, highly aesthetic results can be achieved. The different materials, such as framework, layering and pressed ceramic are compatible at all times (see article on IPS e.max ZirPress).



Fig 18: XRD patterns of the surface of finished IPS e.max ZirCAD samples: the monoclinic phase in the surface becomes detectable after sandblasting



Fig 19: Three-unit IPS e.max ZirCAD bridge veneered with IPS e.max Ceram



## 7 Literature

- Garvie R.C., Hannink R.H., Pascoe R.T.; Ceramic Steel?, Nature (London); 258, 703-704 (1975)
- Subbarao E.C., Mati H.S., Srivastava K.K.; Phase Transformation in Zirconia; Phys. Status Solidi A 21 [1], 9-40 (1974)
- Wolten G.M.; Diffusionless Phase Transformations in Zirconia and Hafnia; J. Am. Ceram. Soc., 46 [9], 418–422 (1963)
- [4] Bansal G.K., Heuer A.H.; On a Martensitic Phase Transformation in Zirconia (ZrO<sub>2</sub>): I, Metallographic Evidence; Acta Metall. **20** [11], 1281-1289 (1972); II, Crystallographic Aspects, Acta Metall., 22 [4], 409–417 (1974)
- [5] Heuer A.H., Claussen N., Kriven W.H. and Rühle R.; Stability of Tetragonal ZrO<sub>2</sub>-Particles in Ceramic Matrices; J. Am. Ceram. Soc. **65**, 642-650 (1982)
- [6] Deville S., Guénin G., Chevalier J.; Martensitic transformation in zirconia Part I. Nanometer scale predicition and measurement of transformation induced relief.; Acta Materialica 52, 5697–5707 (2004)
- [7] Lange F.F.; Transformation Toughening; J. Mat. Sci, 17, 247–254 (1982)
- [8] Lange F.F.; Transformation-Toughened ZrO2: Correlations between Grain Size Control and Composition in the System ZrO2-Y2O3; J. Am. Ceram. Soc. 69 [3], 240-242 (1986)
- [9] Yashima M., Kakihana M., Yoshimura M.; Metastable-Stable Phase Diagrams in the Zirconia-Containing Systems Utilized in Solid-Oxide Fuel Cell Application; Solid States Ionics; 86–88 [2], 1131–1149 (1996)
- [10] Scott, H.G.; Phase relationships in the zirconia-yttria system; J. Mat. Sci. 10, 1527–1535 (1975)
- [11] Becher P.F., Swain M.V.; Grain-Size-Dependent Transformation Behavior in Polycrystalline Tetragonal Zirconia; J. Am. Ceram. Soc., 75 [3], 493–502 (1992)
- [12] Ruiz L, Readey M.J.; Effect of Heat Treatment on Grain Size, Phase Assemblage, and Mechanical Properties of 3 mol% Y-TZP; J. Am. Ceram. Soc., 79 [9], 2331–2340 (1996)
- [13] Lange F.F., Dunlop G.L., Davis B.I.; Degradation During Aging of Transformation-Toughened ZrO<sub>2</sub>-Y<sub>2</sub>O<sub>3</sub> Materials at 250°C; J. Am. Ceram. Soc. 69, 237–240 (1986)
- [14] Tsugio Sato, Masahiko Shimada; Transformation of Yttria-Doped Tetragonal ZrO<sub>2</sub>-Polycrystals by Annealing in Water; J. Am. Ceram. Soc. **68** [6], 356–359 (1985)
- [15] Tsugio Sato, Shiro Ohtaki, Tadashi Endo, Masahiko Shimada: Improvement of Thermal Stability of Yttria-Doped Tetragonal Zirconia Polycrystals by Alloying with Various Oxides; Int. J. High Technology Ceramics 2, 167–177 (1986)
- [16] Kosmač T., Oblak C., Jevnikar P., Funduk N., Marion L.; The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic; Dental Materials 15, 426–433 (1999)
- [17] Schweiger M.; Zirkoniumoxid Hochfeste und bruchzähe Strukturkeramik; Ästhetische Zahnmedizin 5, 248–257 (2004)





Marcel Schweiger



The universal veneering ceramic – One for 4

## 1 Introduction

IPS e.max Ceram

In the development of the IPS e.max Ceram veneering ceramic, the main emphasis was placed on characteristics such as aesthetics and universal application. In regard to aesthetics, excellent results had previously been achieved in conjunction with veneering materials such as IPS d.SIGN and IPS Eris for E2 [1–3] using fluorapatite glass ceramics as opaquing agents – a successful strategy that has been continued in IPS e.max Ceram.

IPS e.max Ceram is truly universal because it is compatible with the IPS e.max framework materials ZirCAD (zirconium oxide ceramics), CAD and Press (lithium disilicate glass ceramics) as well as the pressable dentin component ZirPress (fluorapatite glass ceramics). Optimal adjustment of the thermal expansion and firing temperature was an essential pre-requisite to meeting these demands.

## 2 Composition

IPS e.max Ceram is composed of silicate glasses and fluorapatite glass ceramics. The only natural ingredient used in the fabrication of silicate glasses is quartz sand. No feldspar components are required to manufacture IPS e.max Ceram. Additionally, it cannot form

any leucite crystals because of its composition. The components of the mixture include glasses from the SiO<sub>2</sub>-Li<sub>2</sub>O-Na<sub>2</sub>O-K<sub>2</sub>O-ZnO-Al<sub>2</sub>O<sub>3</sub> multicomponent system. The base glasses of fluorapatite glass ceramics additionally contain CaO, P2O5 and F components. These three basic components are a pre-requisite for fluorapatite crystal formation Ca5(PO4)3F. Silicate glasses are homogeneously melted in special glass melting furnaces at temperatures between 1300 and 1500 °C and subsequently converted into the final IPS e.max Ceram product through controlled and reproducible tempering, milling and mixing procedures. Except for the Glazes, Shades and Essence materials, all IPS e.max Ceram products contain varying concentrations of fluorapatite crystals. Table 1 shows the composition of the main products.

The main component in all product groups is SiO<sub>2</sub> with a content of more than 50 %wt. The sintering behaviour and wetting of the framework surface during ceramic firing in the dental laboratory is influenced by additives such as Li<sub>2</sub>O, B<sub>2</sub>O<sub>3</sub> and F. Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub> improve the chemical resistance, temperature stability and mechanical strength of the ceramic material. The alkali metal oxides K<sub>2</sub>O and Na<sub>2</sub>O are extremely important when it comes to adjusting the thermal expansion coefficient and the processing temperature. Depending on the SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> content, the amount of alkali metal oxide must be modified. CaO, P<sub>2</sub>O<sub>5</sub> and F are mandatory in order

## Table 1:

chemical composition of the unreference's elinax cerain products (in 2000)					
	Layering materials	Add-On	ZirLiner	Shade, Essence, Glaze	
SiO <sub>2</sub>	60.0-65.0	61.0-68.0	50.0-60.0	61.0-68.0	
Li <sub>2</sub> O	1.0-2.0	1.0-2.5		1.0-2.5	
Na <sub>2</sub> O	6.0-9.0	5.0-8.0	6.0-11.0	5.0-8.0	
K <sub>2</sub> O	6.0-8.0	5.0-8.0	4.0-8.0	5.0-8.0	
CaO	1.0-3.0	1.0-3.0	1.5-4.0	1.0-3.0	
ZnO	2.0-3.0	2.0-4.0		2.0-4.0	
Al <sub>2</sub> O <sub>3</sub>	8.0-12.0	5.0-8.0	16.0-22.0	5.0-8.0	
ZrO <sub>2</sub>	1.0-1.5	0.5-1.5	1.5-3.0	0.5-1.5	
P2O5	0.1-1.0		0.8-2.0		
F	1.0-2.0	1.0-2.0	0.2-1.5	1.0-2.0	
Addition	al components: SrO BoO	$7rO_2$ TiO_2			

Additional components: SrO, B2O3, ZrO2, TiO2



to precipitate the fluorapatite. Thus indirectly, the content of apatite-forming components has an impact on the opacity and aesthetics of the material.

The milling technique used when processing glass granulates into glass powder plays a decisive role. By means of the grain size distribution and final grain size of the powder, the handling properties can be selectively adjusted. While the Dentin and Incisal materials feature a coarse grain structure with average particles sizes ranging from 30 to  $36 \,\mu$ m, very fine powders with average particle sizes of 5 to 10  $\mu$ m are used for the Essence materials can be admixed, just like colour pigments, in order to individually and homogeneously enhance the chroma of the existing coarse-grain materials.

## 3 Microstructure

IPS e.max Ceram is a homogeneous mixture of fluorapatite-containing glass ceramic and sintered glass powders. The sintered glasses are single-phase glasses and do not contain any crystalline phases. As a result, these glasses remain completely stable during the ceramic firing process and do not form any crystals. The glass ceramics contain fluorapatite crystals Ca<sub>5</sub>(PO<sub>4</sub>)<sub>3</sub>F. Evidence of crystalline phases is provided by means of X-ray diffraction (XRD) and scanning electron microscopy (SEM). Figures 1 and 2 show the fluorapatite crystal shapes that typically occur in IPS e.max Ceram.

Fluorapatite forms long prismatic crystals with a hexagonal base. Longitudinal cuts show an oblong rectangular surface and lateral cuts the corresponding hexagonal surfaces. The crystalline symmetry is thus hexagonal. The main components are PO<sub>4<sup>3+</sup></sub> tetrahedrons laterally cross-linked with Ca2+ ions. Fions are embedded in the intermediate spaces. Fluorapatite has a refractive index ranging from 1.629 to 1.667.

By using different concentrations of CaO, P<sub>2</sub>O<sub>5</sub> and F and by varying the crystallization temperature and time, different shapes of fluorapatite crystals can be produced in a targeted fashion. These processes are conducted in a controlled, reproducible way during production. The quantity and shape of the crystals will not change any more during the ceramic firing procedure in the dental laboratory, which is conducted at 750 °C. This is ensured by the fact that during the production procedure, a crystallization temperature between 850 °C and 1150 °C is maintained during a tempering period of more than one hour. All subsequent firing cycles in the dental laboratory are conducted at processing parameters that are clearly below these temperatures. Consequently, no crystal growth can ensue. In the case of IPS e.max Ceram, nanoscale as well as oblong fluorapatite crystals are present. The long prismatic crystals have a length of 1-2 µm and a cross-sectional diameter of less than 200 nm. With less than 200 nm, the nano-scale crystals are very short in length. Their cross-sectional diameter is less than 100 nm. For sizes below 100 nm, the term "nano" is justified. Evidence of the crystalline content can be provided by means of XRD. In Figure 3, the X-ray diffraction diagram of a fluorapatite glass ceramic from the IPS e.max Ceram range is shown and contrasted with a reference pattern from pure fluorapatite.

Apart from the fact that the structure of fluorapatite crystals is very similar to that of natural tooth enamel, the crystals are systematically employed to adjust the optical properties of the different materials. More details on this subject can be found in chapter 4.



Fig 1: Longish fluorapatite crystals (etched; 3 % HF, 10 s)



Fig 2: Nano-fluorapatite crystals (etched; 3 % HF, 10 s)





Fig 3: X-ray diffractogram of a fluorapatite glass ceramic which is used in IPS e.max Ceram



Fig 4: Schematic illustration of the opalescence effect: Transmission of long-wave light and scattering of the short-wave light

## 4 **Properties**

#### 4.1 **Optical properties**

Fluorapatite crystals have a significant impact on the light dynamics of IPS e.max Ceram. The light is mainly refracted along the crystals. In this refraction process, the light wave range, particle size (cross-sectional diameter) as well as the refractive index of the filler particles and matrix play a crucial role. In IPS e.max Ceram, the opacity is established with the help of fluorapatite crystals which are embedded in the glass matrix as particles. The refractive index of the crystals (particles) is between 1.63 and 1.67, which is higher than that of the glass matrix which features 1.50 to 1.55. In contrast to conventional opaquing techniques using ceramic particles such as ZrO<sub>2</sub> (refractive index 2.1 to 2.2) or TiO<sub>2</sub> (refractive index 2.5 to 2.6), the differences between the refractive index of the particles and that of the matrix is significantly smaller in IPS e.max Ceram. Consequently, light scattering conditions which are ideal for dental applications are created. Optical properties such as translucency and brightness can be ideally adjusted. If the difference between the two refractive indices is too high, for example, high brightness may be achieved, but hardly any translucent properties can be created because of the strong scattering effect.

Opalescent properties are also caused by light scattering. The short-wave (blue) portion of the incident light is scattered and reflected by the small crystals and phase separation areas whose size is in the range of 100 to 500 nm. The long-wave (reddish) portion of the light is not affected by these small particles and may pass practically unhindered. In incident light, the material appears bluish, in transmitted light reddish-orange. This creates the typical opalescent effect which imparts a natural aesthetic appearance to restorations veneered with IPS e.max Ceram in the incisal region (Fig 4).

In addition, phase separation effects, which play a crucial role in the production of glass ceramics, are selectively employed in the Effect materials, apart from the nano-scale fluorapatite crystals. Phase separation processes can be controlled and reproduced just like crystallization processes. In the case of IPS e.max Ceram, these are conducted at temperatures between 900 and 1150 °C. As a result, the particle size remains completely stable during the ceramic firing cycles conducted at 750 °C in



the dental laboratory. The opalescent effect is thus maintained even during multiple firings. The drop-shaped phase separation areas have a diameter of approx 300 nm (Fig 5). Consequently, the basic requirements for wavelength related light scattering and thus opalescent effects are met.



**Fig 5**: Drop-phase in the nano-scale range and fluorapatite crystals to achieve a natural opalescent effect (etched with 3 % HF; 10 s)

#### 4.2 Mechanical properties

In clinical use, veneering ceramics are exposed to high chemical, thermal and mechanical stress. The individual types of stress overlap and may even be mutually reinforcing. The initial measurable strength of a material cannot be considered a characteristic value, as the preparation technique and homogeneity of the manufacturing process greatly influence the data obtained. Glasses and ceramics are brittle-rigid materials to which the principles of linear-elastic fracture mechanics apply. They do not undergo any plastic deformation until failure occurs and thus demonstrate purely elastic behaviour. In brittle-rigid materials, common fracture points are material defects such as porosities, entrapments or voids at the surface. SiO<sub>2</sub> fibres with perfect surface and homogeneous volume may reach tensile strength values between 12,000 and 16,000 MPa under vacuum. A prepared sample rod made of the same glass, however, achieves only 45 MPa. If the surface of such a rod is sandblasted, tensile strength values drop to a mere 14 MPa [4]. The larger the material defect, the higher the intensity of the tension under similar stress or tension. Thus minimizing the number and size of defective areas is an essential objective in the fabrication of component parts with brittle-rigid materials. IPS e.max Ceram achieves biaxial strength values of between 80 and 100 MPa and thus easily fulfils the standard requirement

(ISO 6872) of >50 MPa. The fluorapatite crystals do not measurably strengthen the glass ceramic as, for example, leucite and lithium disilicate crystals do. The precipitation density is too low and the difference in the thermal expansion coefficient compared to the glass matrix is too small to reinforce dispersion. It should be pointed out that all the currently available veneering ceramics for ZrO<sub>2</sub> frameworks exhibit similar strength values of between 70 and 100 MPa (measurements lvoclar Vivadent AG, 2003–2005). Table 2 shows a comparison of the biaxial strength value of different veneering ceramics for ZrO<sub>2</sub> frameworks.

Thus it should be kept in mind that the firing temperatures which are used in the fabrication of test specimens are higher than those indicated by the manufacturer because the test specimens lie flat on the firing tray and are therefore not evenly exposed to the heat radiation of the furnace on all surfaces. In order to compensate for this difference in energy compared to a centrally positioned tooth restoration, the firing temperature needs to be increased. It can be easily judged from the firing result whether the specimens are completely fired. Only completely fired test specimens provide representative strength values. All the measured veneering ceramics comply with the minimum value of 50 MPa. Due to the high glass content, no significantly higher strength values have been attained in conjunction with leucite-free veneering ceramics to date. The ceramic veneer still represents the weakest part of all-ceramic restorations. Therefore, the framework dimensions should be maximized. This applies to the posterior region in particular, as function should be given priority over aesthetics in this area. A cusp-supporting design of the framework can further improve the mechanical reliability of the restoration.

#### Table 2:

Biaxial strength values (acc. to ISO 6872) of different ZrO₂ veneering ceramics (Ivoclar Vivadent AG measurements, 2003 – 2005)

IPS e.max Ceram	90 ± 10MPa
ZrO <sub>2</sub> -Keramik A (Nobel Zr)	102 ± 18 MPa
ZrO <sub>2</sub> -Keramik B (Cercon S)	84 ± 13 MPa
ZrO <sub>2</sub> -Keramik C (Vita VM9)	97 ± 8MPa



### 4.3 Thermal properties

The linear coefficient of thermal expansion (CTE) of both the framework and veneering material need to be compatibilized with each other. The CTE indicates the expansion or contraction a rod of 1 m length undergoes if the temperature is changed by 1 °C or 1 K (Kelvin). The change in absolute length is indicated in  $\mu m$  (=0.000001 m). Therefore, the unit of measurement is µm/m\*K. The measuring unit may also be abbreviated to ppm/K. However, the letters ppm stand for "parts per million" and correspond to 0.0001%. The CTE thus indicates the change in relative length of a material per K. To illustrate this relationship, a calculation example is shown below. The change in absolute length  $\Delta$ I of an IPS e.max ZirCAD bridge with a length of 30 mm (Lo) is to be determined within the temperature range  $\Delta$ T of 25 to 400 °C (CTE ZirCAD  $\alpha$  = 10.75 µm/m\*K).

### $\Delta I = \alpha \bullet \Delta T \bullet L_0 \bullet 0.000001$

<u>∆l</u> = 10.75 • (400–25) K•30mm•0.000001/K = 0.121mm=121µm

The change in absolute length  $\Delta I$  of an IPS e.max ZirCAD bridge with a length of 30 mm in the range of 25 to 400 °C is 121  $\mu m.$ 

As a rule of thumb, the veneering ceramics used for dental restorations tend to establish a stable bond to the framework if their CTE is lower than that of the framework material. The mechanically weaker surface of the veneering ceramic is thus exposed to compressive strain. Glasses and ceramics can resist compressive forces much more easily than tensile forces. This principle is applied in both metal and all-ceramics.

Due to the carefully selected CTE of IPS e.max Ceram, the material is compatible with IPS e.max CAD, Press and ZirCAD. Figure 6 shows the CTE values of the IPS e.max materials.

No matter which combination of system components is chosen, the CTE of IPS e.max Ceram is always below that of the framework ceramic. As a result, the mechanically weaker veneering ceramic is exposed to compressive stress while the surface of the stronger framework ceramic is exposed to tensile stress. The two-layer model allows the theoretically occurring surface tension to be calculated [5]. In the case of IPS e.max ZirCAD, the maximum tensile stress calculated is 15 MPa, and in the case of the glass ceramics IPS e.max CAD and Press, the maximum value is 7 MPa. These figures are clearly below the inherent strength values of the framework materials and thus do not present a problem. However, the fact that this theoretical calculation is based on the assumption of a homogeneous cooling process should be kept in mind. Quick cooling and varying thickness of the restoration may lead to an increase in tensile stress and even transfer critical tensile stresses to the veneering ceramic.

When comparing CTE values, it is important that the values relate to identical temperature intervals. The indicated or calculated CTE value is dependent on the temperature. As the transformation temperature of IPS e.max Ceram is in the range of 470–490 °C, the CTE value cannot be analysed up to 500 °C. The transformation point represents a characteristic point in the transition between the glassy solid state and the glass melt. The linear expansion changes at the transformation temperature. In the analysis, the upper temperature limit is thus set at 400 °C in order to ensure a stable linear expansion range. Correspondingly, the CTE values for framework ceramics are indicated in the temperature range between 100 and 400 °C, even though it would be perfectly possible to evaluate the CTE values of these materials up to a temperature of 500 °C.



Fig 6: Gradation of CTE values within the IPS e.max System (Ivoclar Vivadent 2005



## 5 Sintering process

IPS e.max Ceram is a low fusing glass ceramic. The sintering point of the mixture is below 800 °C. Sintering is a dynamic process. The most important factors that influence this process are grain size distribution, the specific surface texture, surface conditioning of the powder, densification of the powder, heating rate, final temperature and holding time. IPS e.max Ceram is a glass ceramic material with a high content of glassy phase. Glassy materials demonstrate a characteristic transformation as well as subsequent softening range. In the softening range, the viscosity is 108-1013.5 Pas. At the points of contact, first melting bridges form between the individual powder particles. Further densification is achieved by liquid-phase sintering [6, 7]. By selective melting of the IPS e.max Ceram glass grains, which is accomplished as from the softening point of 540 °C, the open pores are continuously closed as the temperature increases.

The sintering behaviour can be characterized by means of thermal microscopy. In the process, a cylindrical powder specimen is heated at a constant rate and the sintering shrinkage is recorded digitally by means of shadow projection. The volumetric shrinkage of the standardized, non-densified test specimens is 45.4 %. Provided that the sintering behaviour is isotropic (identical in all directions), this corresponds to a linear shrinkage of 18.3 %. To enable a comparison, several competitive ceramic materials were subjected to the same test procedure (Table 3). The linear shrinkage of all the systems was limited to the relatively narrow range between 43.2 and 46.5 (linear 17.2 – 18.8 %), while the linear shrinkage of IPS e.max ZirCAD was 20 % on average.

In order to illustrate the sintering process, a firing cycle conducted in a Programat P200 was interrupted at the temperature stages of 600, 650, 700 and 750 °C to enable SEM analysis of the sinter structure. The progress of the sintering process is shown in Figs 6 to 9.

At 600 °C, the individual glass and glass ceramic grains can still be easily distinguished. Thermal microscopy shows that the sintering process starts at 550 °C. First sinter necks form at the direct contact points between individual grains. The sinter structure exhibits low strength at this stage. In the temperature range between 600 and 650 °C, strong sintering activity is observed (Fig 8). At 650 °C, the sinter shrinkage is around 19.3 % (corresponds to a linear shrinkage of 6.9%). The individual grains are connected with each other through pronounced sinter necks. These sinter necks form through localized melting of the grains. Large grains and components with higher melting temperatures impart the material with the required resistance against deformation. Up to 650 °C, the material has an open-pored structure. Volatile organic substances need to be disintegrated and decomposed when this temperature is reached. If this is not so, isolated sinter pores in the structure remain which are filled with undesirable reaction products. Therefore, it is of utmost importance to use only IPS e.max build-up liquids. The organic components used in these materials easily decompose at these temperatures. From 700 °C, the sintering process is so far advanced that only closed porosities (isolated spaces) are present (Fig 9). In contrast to trapped gas, this type of porosity is easily eliminated as the sintering process advances. In the final stages of sintering, at 750 °C, the remaining closed porosities are densely sintered. Depending on the precompaction by the dental technician, the total volumetric shrinkage ranges between 40 and 45 %. A dense, homogenous sinter structure results as shown in Figure 10.

#### Table 3:

Comparison of ZrO<sub>2</sub> veneering ceramics with regard to characteristic temperatures and volumetric shrinkage

Product name	Ss [°C]	Se [°C]	HT [°C]	Linear shrinkage [%]	Volumetric shrinkage [%]
IPS e.max Ceram	550	749	935	18.3	45,4
Product A (Nobel Rondo Zr)	654	882	1170	17.2	43,2
Product B (Cercon CeramS)	560	803	1011	17.4	43,7
Product C (Vita VM9)	670	905	1241	18.8	46,5

 $S_S = start of sintering; S_E = end of sintering; HKT = hemisphere temperature$ 

## 6 Bonding properties

IPS e.max Ceram is sintered onto different framework materials. The properties of the veneering ceramic are optimally coordinated with corresponding framework materials as regards the CTE and firing temperature. The resulting adhesive regions are visible on the polished sections shown below (Figs 11–14).

IPS e.max CAD, Press and Ceram are of silicatic nature. Due to this chemical similarity, adhesion can also be achieved through chemical bonds within the glass structures.

IPS e.max ZirCAD is an oxide-based ceramic which does not have any bonding groups that are chemically similar to those of silicate glass. At the temperature at which ZirLiner is sintered, reactions occur that lead to a chemical bond between the oxide ceramic (IPS e.max ZirCAD) and the glass ceramic (IPS e.max Ceram ZirLiner). The subsequent firing cycles (wash and main firings) again involve bonding of silicatic materials. As a result of the chemical similarity, adhesion is established through the formation of chemical bonds within the glass structure.

Surface conditioning of IPS e.max ZirCAD is of particular importance when trying to establish a bond between IPS e.max ZirCAD / ZirLiner and Ceram. As described in the article "IPS e.max ZirCAD", the transformation of zirconia from its tetragonal to its monoclinic phase is induced by mechanical stress. In dental technology procedures, this transformation process can easily be triggered by sandblasting with aluminium oxide abrasive medium. A modified surface results which consists of a monoclinic phase and possibly trapped aluminium oxide particles. Due to the subsequent ceramic bakes, which are conducted at 750 °C, the monoclinic phase is maintained. Both the monoclinic phase and the aluminium oxide demonstrate a considerably lower CTE (in the range of 7 to 8) than the tetragonal ZrO<sub>2</sub>. The bonding layer undergoes overcritical changes of the CTE value, ranging from 10.75 (tetragonal dimensions) to 7 to 8 and finally 9.5 (ZirLiner and Ceram). Uncontrolled stress results which weakens the bond and may lead to chipping. Sandblasting the veneered surface with aluminium oxide abrasive medium is strongly forbidden.



Fig 7: Sinter microstructure at 600 °C and 64.0 % of theoretical density



Fig 8: Sinter microstructure at 650 °C and 83.3 % of theoretical density



Fig 9: Sinter microstructure at 700 °C and 93.3 % of theoretical density



Fig 10: Sinter microstructure at 750 °C and 100 % of theoretical density





Fig 11, 12: Bonding region between IPS e.max Ceram and IPS e.max CAD/Press (50x and 100x magnification, polished section, BDE Contrast)



Fig 13, 14: Bonding region between IPS e.max Ceram, ZirLiner and IPS e.max ZirCAD (100x and 1000x magnification, polished section, BDE Contrast)



## 7 Summary

IPS e.max Ceram is a veneering ceramic for universal use which is suitable for both zirconium oxide frameworks (IPS e.max ZirCAD) and lithium disilicate frameworks (IPS e.max Press, CAD). Due to the use of fluorapatite glass ceramics as opaquing agent, natural-looking aesthetics are achieved.

## 8 Literature

- Höland W., Rheinberger V., Wegner S., Frank M.; Needle-like apatite-leucite glass-ceramic as a base material for the veneering of metal restorations in dentistry; J Mater Sci Mater Med. 11 (1),11–7 (2000)
- [2] Höland W., Rheinberger V., Frank M.; Mechanism of nucleation and controlled crystallization of needlelike apatite in glass-ceramics in the SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-K<sub>2</sub>O-CaO-P<sub>2</sub>O<sub>5</sub> systems; J Non-Cryst. Solids **253**, 170–177 (1999)
- Schweiger M., Höland W., Rheinberger V.;Nanophase formation in different glass-ceramic systems; 104<sup>th</sup> ACerS Meeting, St. Louis (2002)
- [4] *Kelly J.R.*; Perspectives on strength; Dent Mater **11**, 103–110 (1995)
- [5] Lenz J.; Über die Spannungsverteilung in metallkeramischen Verbund-Biegeproben und die Ausbildung von Transversalrissen in der Keramikverblendung; Ceramic forum international/Berichte der DKG 60, 237–243 (1983)
- Kingery W.D., Bowen H.K., Uhlmann D.R.; Introduction to ceramics; 2<sup>nd</sup> edition (Wiley, New York, 1976)
- [7] Petzow G., Kaysser W.A., Amtenbrink M.; Materials science monographs 14, Sintering theory and practice" edited by Kolar D., Pejovnik S., Ristic M.M. (Elsevier, Amsterdam, Oxford, New York, 1982)







Marcel Schweiger

# **IPS e.max ZirPress**

The pressable veneering ceramic

## 1 Introduction

IPS e.max ZirPress allows a dentinshaded layer to be pressed on a CAD/CAMfabricated IPS e.max ZirCAD framework. Therefore, IPS e.max ZirPress represents a link between the IPS Empress and the CAD/CAM processing technique. The composition is similar to that of IPS e.max Ceram Dentin materials. The opacity concept is also realized by using fluorapatite glass-ceramics. Due to the IPS Empress technique, a very good cervical fit with high aesthetics in the anterior region is achieved. The fully anatomical presson procedure with subsequent stains and glaze firing in the posterior region is a fast and sound method to fabricate accurately fitting homogeneous restorations. In addition, the material is ideally suitable for the fabrication of ZrO2supported inlay-retained bridges in conjunction with the fully anatomical press-on technique.

## 2 Composition

IPS e.max ZirPress is composed of silicate glasses and apatite glass-ceramics. The only natural raw material used is quartz sand. IPS e.max ZirPress does not contain any feldspar components or leucite crystals. Glasses from the SiO<sub>2</sub>-Li<sub>2</sub>O-Na<sub>2</sub>O-K<sub>2</sub>O-ZnO-Al<sub>2</sub>O<sub>3</sub> system are used for the mixing components. For the glass-ceramics which contain fluorapatite, the base glasses additionally include CaO, P<sub>2</sub>O<sub>5</sub> and F. These three basic components are the prerequisite for the formation of the Ca<sub>5</sub>(PO4)<sub>3</sub>F fluorapatite crystals. The base

## Table 1:

Composition of IPS e.max ZirPress ingots (wt.%)		
SiO <sub>2</sub>	57.0 – 62.0	
Na <sub>2</sub> O	6.0 - 8.0	
K <sub>2</sub> O	2.0 - 4.0	
CaO	2.0 - 4.0	
Al <sub>2</sub> O <sub>3</sub>	12.0 – 16.0	
ZrO <sub>2</sub>	1.5 – 2.5	
P2O5	1.0 – 2.0	
F	0.5 – 1.0	
Additional components: SrO, B2O3, ZnO		

glasses and glass-ceramics are identical to those of IPS e.max Ceram. The glasses are homogeneously melted in special glass melting furnaces at temperatures of 1300–1500 °C. Subsequently, they are transformed into the IPS e.max ZirPress ingots by means of complex tempering, grinding, mixing and sintering processes. Table 1 shows the composition range of the main components.

The chemical composition of IPS e.max ZirPress and IPS e.max Ceram is very similar. Identical basic materials are used in the mixing processes. By suitably adjusting the mixing ratios, the pressing temperature was established in such a way that IPS e.max ZirPress remains resistant to firing in subsequent firing cycles at 750 °C.

## 3 Structure

IPS e.max ZirPress is a homogeneous mixture of fluorapatite-containing glass-ceramics and sinter glasses. The sinter glasses are single-phased and do not contain any crystalline phases. The glass-ceramics used contain fluorapatite crystals Ca<sub>5</sub>(PO<sub>4</sub>)<sub>3</sub>F. The

presence of a crystalline phase is confirmed by XRD measurement and scanning electron microscopy (SEM). Figure 1 and 2 show the crystal forms typical of IPS e.max ZirPress.

By using different concentration ratios of CaO-P2O5-F and coordinated crystallization temperatures and times, various fluorapatite crystal forms can be selectively produced. These processes take place in a controlled fashion on a production scale. The crystal quantity and form no longer changes during the firing process in the dental laboratory. IPS e.max ZirPress contains needle-like and nanoscale fluorapatite crystals. The needles measure 1–2  $\mu$ m in length and less than 200 nm in diameter. The nano-scale crystals have grown along a



Fig 1: Longish fluorapatite crystals



Fig 2: Nano-fluorapatite crystals

slight longitudinal axis and have a length of less than 200 nm. Their diameter measures less than 100 nm. The term nano-crystals is applicable to sizes below 100 nm.

Apart from the fact that fluorapatite crystals are employed because their structure is very similar to that of natural dental enamel, the crystals are selectively used to establish the optical properties as regards translucency and brightness of the different ingots. This topic is explained in the chapter IPS e.max Ceram and is also valid for IPS e.max ZirPress.

## 4 **Properties**

IPS e.max ZirPress is a pressable material version of IPS e.max Ceram. Therefore, the chemical and physical properties of both are very similar (Table 2).

The industrial sintering process and the IPS Empress processing technique endows IPS e.max ZirPress with a very homogeneous, dense structure. If a veneer is built up by means of conventional layering and sintering, there is always a latent risk of unintentionally including defective areas in the structure of the veneering ceramic. This risk is averted if IPS e.max ZirPress is used. In addition, the measured biaxial strength is about 20% higher than if IPS e.max Ceram is applied according to the layering technique. The firing stability during subsequent firing cycles with IPS e.max Ceram is ensured by the temperature difference of 150 °C between pressing and layering. The accuracy of pressed ceramic shoulders or margins in inlay-retained bridges is maintained and thus they no longer have to be adjusted. Hence, possible shortcomings in the accuracy of fit of sintered and reduced  $ZrO_2$  frameworks can be improved.

The CTE is ideally coordinated to the IPS e.max ZirCAD framework ceramic and IPS e.max Ceram. The CTE is the highest in IPS e.max ZirPress and decreases continuously (see Fig 3).

The bond between IPS e.max ZirPress and the ZirLiner is very homogeneous and compact due to the similar chemical composition (fluorapatite glass-ceramic) and virtually identical processing temperatures. Given the large difference in processing temperature of 50 °C between the ZirLiner (950 °C) and IPS e.max ZirPress (900 °C), the ZirLiner which has been fired prior to the press procedure remains dimensionally stable on the ZirCAD surface during the press procedure. Before IPS e.max Ceram is layered on the pressed ZirPress surface, it is important that the surface is properly conditioned. Complete removal of the reaction layer with the Invex liquid and the subsequent wash firing with IPS e.max Ceram are the requisite for an impeccable bond. Figures 4 and 5 show the respective bond zones in the ceramo-graphic grinding pattern.

IPS e.max ZirPress	
Biaxial strength [MPa] *	110 ± 10
CTE (100-400°C) [µm/m*K]	9.75 ± 0.25
Glass point [°C]	530 ± 10
Chemical resistance [µg/cm²] *	30 ± 10
Vickers hardness HV5 [MPa]	5400 ± 200
* ISO 6872	



## Table 2:

REPORT

Fig 3: CTE gradation of IPS e.max ZirCAD - ZirPress - Ceram

IPS e.max ZirPress can be etched with hydrofluoric acid to produce a retentive pattern. The etching pattern enables a good bond to adhesive luting systems, such as Variolink II or Multilink. The retentive pattern in microscopic and macroscopic magnification is visible in Figs 6 and 7. The apatite crystals that have been produced as a result of etching the glass matrix are apparent in Fig 6. Figure 7 shows high-contrast surfaces of the individual glass and glass-ceramic grains with different etching patterns.

## Conclusion

IPS e.max ZirPress is a fluorapatitecontaining glass-ceramic, which can be processed in the IPS Empress technique. IPS e.max ZirPress is used to press onto CAD/CAMfabricated ZirCAD frameworks and thus represents a link between the IPS Empress and CAD/CAM processing technique. The chemical and physical properties are ideally coordinated with the IPS e.max ZirCAD framework ceramic and the IPS e.max Ceram veneering ceramic. By using the IPS Empress technique, reproducible results exhibiting a high accuracy of fit and good aesthetic properties can be achieved.



Fig 4: Bond between IPS e.max ZirCAD, ZirLiner and ZirPress (polished ground surface)



Fig 5: Bond between IPS e.max ZirPress and Ceram (etched, 3% HF, 10 s)



Fig 6: Etching pattern of IPS e.max ZirPress with fluorapatite crystals (4.5% ceramic etching gel (HF); 20 seconds of reaction time)



Fig 7: Etching pattern of IPS e.max ZirPress with glass and glassceramic particles (4.5% ceramic etching gel (HF); 20 seconds of reaction time)



# **IPS e.max** The development in terms of dental technology and shading



Harald Kerschbaumer

## 1 Introduction

Ivoclar Vivadent AG offers a complete all-ceramic system with IPS e.max. IPS e.max Ceram which can be fired on the different framework materials made of lithium disilicate and zirconium oxide represents the core of the system. In addition, the framework materials can be processed with different methods: IPS e.max CAD and ZirCAD in the CAD/CAM technique, IPS e.max Press and IPS e.max ZirPress in the press technique.

In order to use a veneering ceramic on the different framework materials (Fig 1), a common basis has to be established. The basis consists of the framework shades MO 0 – white, MO 1 – light yellow, MO 2 – reddish, MO 3 – yellowish, MO 4 – reddish-grey.

The shades of IPS e.max CAD and IPS e.max Press are identical. The white zirconium oxide is adjusted to the same basic shades by means of the ZirLiner.



Fig 1: Unveneered frameworks: IPS e.max CAD, IPS e.max Press and IPS e.max ZirCAD

## 2 Framework materials

- IPS e.max Press is a lithium disilicate glass-ceramic, which is available as a framework material in a medium opacity (MO). Therefore, the opacity and the resulting brightness value is of particular importance for this ingot. The ingots are coloured according to the five basic shades MO 0, MO 1, MO 2, MO 3 and MO 4.
- IPS e.max CAD is a lithium disilicate glass-ceramic that has been particularly optimized for CAD processing. During the milling process in the unit, the framework

material is in the "blue", pre-crystalline state and thus easier to process. It is only after milling that the restorations are endowed with their final strength and colour by means of heat treatment. The shade and opacity of IPS e.max CAD and IPS e.max Press frameworks are coordinated. The blocks are available in levels of medium opacity (MO).

- IPS e.max ZirCAD is a Y-TZP zirconium oxide. It can only be used for the veneering technique. In order to achieve a shade match to the IPS e.max Press and IPS e.max CAD framework materials, ZirLiner powders in special shades have been developed to apply on the zirconium oxide frameworks.
  - IPS e.max ZirPress is used to press on zirconium oxide frameworks. The ingots are available in two levels of opacity: The **MO** ingots feature a higher opacity and colour saturation. Thus, they are used for the fabrication of partially layered restorations. The LT ingot is used for pressing fully anatomical restorations. Slight shade adjustments are applied with the IPS e.max Ceram Shades. Unlike the MO ingot, the LT ingot features a higher translucency in order to achieve good adaptation to the natural tooth (inlays and partial crowns) and an incisal-like effect in fully anatomically pressed restorations. The two ingot types are shaded according to the Vita Lumin Classic shade guide. The ingots are available in the eight most popular shades.

## Table 1:

Table of opacity	
Material	CR*
e.max CAD MO 2	85%
e.max Press MO 2	85%
e.max Press HO	95%
e.max ZirPress A3 MO	85%
e.max ZirPress A3 LT	75%
e.max ZirCAD	95%



## 3 Veneering material

Basically, the development of veneering ceramics covers the entire range of desirable optical properties, such as translucency, opacity, opal effect, fluorescence etc.

## 3.1 Opacity and translucency

Since the development of IPS Empress 2 and IPS d.SIGN, the conventional opaquing agents (Fig 2), such as SnO<sub>2</sub> and ZrO<sub>2</sub>, are no longer used for achieving the opacity in highly aesthetic ceramics.

A glass component which contains a variety of nano-apatite crystals (Fig 3) is used to produce the desired opacity in IPS e.max Ceram. This procedure allows a brightness value that is similar to that of natural teeth and excellent light transmitting properties (Fig 4) to be achieved. This effect is the result of a well coordinated refraction index between apatite crystals and the residual glass matrix. The refraction index of the conventional opaquing agents stannous oxide or zirconium oxide is considerably higher than that of the residual glass matrix. Therefore, materials that have been treated with these agents appear rather opaque and dark by transmitted light (Figs 5 and 6).

### 3.2 Opalescence

The opalescence of the IPS e.max Ceram Effect materials is created by a selective precipitation of nano-apatite crystals in the size of 300 to 500 nm. The opalescent effect is maintained even after multiple firing cycles.

The IPS e.max Effect materials 1 to 4 exhibit an opalescence that comes close to that of natural teeth (Fig 7). They are mainly used to determine the individual tooth shade. For the reproduction of shades from the shade guide, however, the stipulated Incisal materials (Fig 8) have to be used.



Fig 2: Conventional opaquing with SnO2 as the opaquing agent; SEM image, 5000x magnification



Fig 3: Opacity achieved by distributed nano-apatite crystals; SEM image, 5000x magnification



Fig 4: IPS e.max Ceram basic materials for achieving the desired opacities; on the left basic material "transpa", on the right basic material "opaque"



Fig 5: Veneered zirconium oxide crowns by incident light; in the center IPS e.max ZirCAD / IPS e.max Ceram; on the left and right crowns from competitors



Fig 6: Veneered zirconium oxide crowns by transmitted light; in the center IPS e.max ZirCAD / IPS e.max Ceram; on the left and right crowns from competitors



## 3.2 Fluorescence

The fluorescence of the IPS e.max Ceram layering materials has been determined according to the proven IPS d.SIGN metalceramic. Special emphasis has been placed on the natural concentration of fluorescent agents. Like in the natural tooth, the highest concentration of fluorescent agents is in the root dentin and the lowest in the materials used for the incisal region (Fig 9).

## Table 2:

Table of opacity		
Material	CR*	
e.max Ceram Body A3	75%	
e.max Ceram Deep Dentin A3	90%	
e.max Ceram Margin A3	85%	
e.max Ceram Incisal 2	40%	
e.max Ceram Transpa Clear	10%	
e.max Ceram Transpa Neutral	20%	

## 3.3 The liner

The IPS e.max Ceram ZirLiner has two functions. It establishes a sound bond to the zirconium oxide framework. In addition, the liner endows the frameworks with the same shade as IPS e.max CAD and IPS e.max Press frameworks. In order to properly adjust the required shade intensity, a special IPS e.max Ceram ZirLiner shade guide is available (Figs 10 and 11).

The range of ZirLiners consists of four shades, which correspond to the shade groups MO 1 to 4, as well as a ZirLiner clear and four Intensive ZirLiners (yellow, orange, brown, incisal).

Unlike zirconium oxide that has been stained with colouring liquids, zirconium oxide frameworks shaded with ZirLiners can be individually characterized. In some cases, a white substructure is advantageous. For this purpose, ZirLiner clear can be used.



Fig 7: Test sample of an opal component by transmitted light



Fig 8: Test sample with ready-mixed Effect 1 material by transmitted light



Fig 9: IPS e.max Ceram crown with the respective shade tabs under UV light; Shoulder, Dentin and Enamel materials to show the course of fluorescence



Fig 10: Fired ZirLiner on IPS e.max ZirCAD crown with IPS e.max Ceram ZirLiner shade quide



Fig 11: IPS e.max Ceram ZirLiner materials shade guide



The IPS e.max Ceram ZirLiners are transparent. Thus, light is transmitted into the tooth structure and gingiva (Figs 12 and 13).

The development of the ZirLiners also fulfils the need for natural fluorescence. Zirconium oxide frameworks do not have any inherent fluorescent properties. By adding fluorescent agents to the ZirLiners, a naturallooking fluorescence is transferred to the frameworks (Fig 14).



Fig 12: ZirLiner on IPS e.max ZirCAD; light is transmitted into the framework



Fig 13: Opaque liner from a competitor; light is not transmitted



Fig 14: Zirconium oxide with ZirLiner (left) and without ZirLiner (right) under UV light







Kerschbauer

Stiefenhofer

The following two clinical cases were treated with all-ceramic crown and bridge restorations. The first case involved a single tooth restoration with IPS e.max CAD/IPS e.max Ceram crowns. The second case was restored with an inlay/crown-retained bridge made of IPS e.max ZirCAD/IPS e.max ZirPress.

Case 1: 46-year-old patient: single crowns with lithium disilicate glass-ceramic copings in the maxilla

## Preoperative situation

The endodontically treated teeth 21 and 22 which had been reconstructed with post and core build-ups had to be replaced by new prosthodontic restorations 13 years after insertion due to aesthetic shortcomings. The following aspects were particularly noteworthy: the crown margins were located in the supragingival area with the dark root surfaces

exposed, and the overall colour of the two crowns was grey compared to the contralateral natural incisors (Fig 1). The gingiva of teeth 21 and 22 was located symmetrically to the gingiva of the contralateral teeth 11 and 12. The endodontic and periodontal state did not give any cause for concern (Fig 2).

### Planning

Before abutment teeth 21 and 22 were restored, the existing metal root posts with composite build-up material had to be removed first. The prepared teeth had to be restored with tooth-shaded, metal-free post and core build-ups. For the crown, it was planned to use IPS e.max CAD MO frameworks in connection with the IPS e.max Ceram veneering ceramic. IPS e.max CAD MO is indicated for single crowns in the anterior and posterior region. Since the IPS e.max CAD framework materials feature a similar opacity to that of IPS e.max Ceram Deep Dentin, the framework can be designed more generously in order to achieve maximum strength.



Fig 1: Preoperative situation: 13-year-old crowns on teeth 21 and 22. The root surfaces were exposed, as the gums had receded. In addition, the ceramic showed a grey colour compared to the contralateral natural teeth.



Fig 2: X-ray of teeth 21, 22 in the preoperative state. Opacities according to the endodontic and prosthetic restoration of teeth 21 and 22 with composite post and core build-ups and crowns.



Fig 3: Abutment teeth 21 and 22 reconstructed and postprepared with post and core build-ups made of composite and glass fibre posts ready to accommodate all-ceramic crowns Picture A shows an overview, Picture B shows a close-up of abutments 21 and 22



#### Pretreatment

Once the crowns and composite buildups, as well as endodontic posts had been removed, new post and core build-ups were inserted after the preparation was revised and an elastomeric impression taken. The post and core build-ups were made using the lightcuring composite Tetric EvoCeram in the incremental layering technique and the glass fibre reinforced root posts FRC Postec on mounted super hard stone models with detachable segments. The restorations were inserted after retraction cords were applied in a relatively dry operating field. For the adhesive cementation technique, the chemically curing luting composite Multilink was used and the restoration conditioned with the chemically curing Multilink Primer. Subsequently, excess cement was immediately removed prior to polymerization using foam pellets and brushes. Finally, the teeth were prepared to accommodate the new crowns. The margin was prepared in the intrasulcular area (Fig 3).

Temporary restorations were provided in the form of resin crowns made of Systemp.c&b plus, which were fabricated directly on the patient. The temporary crowns were fabricated using a polyethylene vacuum



Fig 4: Temporary restoration of teeth 21 and 22 with composite materials. Situation after 4 weeks of treatment.

formed foil after the wax-up was prepared. The crowns were inserted with the eugenolfree temporary luting cement Systemp.cem (Fig 4).

After a non-inflamed gingival situation was achieved after 4 weeks, the location of the preparation margins in relation to the course of the gingival margin was checked and an impression of the abutment teeth taken. The sulcus management comprised a thorough display of the preparation margin by means of the double cord technique. An electrosurgical extension of the sulcus was not required. Iron-IIII-sulfate was used as an astringent.

## Fabrication of the restoration

The best starting point for the framework design is the fully anatomical model of the restoration, which is selectively reduced for the veneer. It is important that the veneering ceramic does not account for more than 50% of the entire restoration thickness to avoid a weakening of the overall restoration. The crown copings were fabricated of lithium disilicate glass-ceramic blocks (IPS e.max CAD MO) in the laboratory using the inLab system from Sirona (Fig 5).

After fitting and finishing, the framework is fired in a ceramic furnace. The use of the stipulated silicon nitride firing tray and the IPS Object Fix firing paste is of utmost importance (Fig 6).

Only in this way can the stipulated temperature profile be achieved and thus the accurate shade and opacity attained. Figure 7 shows the tempered crown copings. Different firing programs are available, depending on the ceramic furnace in use.



Fig 5: IPS e.max CAD MO crown copings



Fig 6: Application of the firing paste



Fig 7: Tempered crowns on silicon nitride









Fig 8: The restoration is cleaned with steam

Fig 9: Application of the wash paste

Fig 10: First firing with Deep Dentin and characterization



Fig 11: Completed crowns 21 and 22 made of IPS e.max CAD/IPS e.max Ceram. Picture A: labial view, Picture B: palatal view





Fig 12: Situation after insertion of crowns 21 and 22 using glass ionomer cement. The crowns have been in situ for 1 week. Picture A: overview, Picture B: close-up, Picture C: final X-ray examination



Before the IPS e.max Ceram materials are applied, the framework is cleaned with steam or in an ultrasonic bath (Fig 8). The IPS e.max CAD framework must not be blasted with aluminium oxide.

Before Dentin and Incisal materials are generously layered, a thin wash layer must be applied with any layering material and fired (Fig. 9). Subsequently, the restoration can be completed as usual (Figs 10 and 11).

The restoration must not be sandblasted with aluminium oxide prior to seating. The inner aspects of the restoration are treated with IPS Ceramic Etching Gel for 20 seconds. This etching procedure is conducted both with adhesive and conventional cementation.

Figure 11 shows the completed crowns after the second firing with Incisal and Transpa materials on the model from a labial (Picture A) and palatal view (Picture B).

## Placement

The strength of IPS e.max CAD/IPS e.max Ceram crowns enables conventional cementation with a retentive core preparation. The crowns were seated using the glass ionomer cement Vivaglass CEM PL. The fully veneered crowns on IPS e.max CAD frameworks that have been veneered with IPS e.max Ceram harmoniously blend into the natural surroundings (Fig 12).

Six-month recalls showed an unchanging result regarding the soft tissue and the quality of the ceramic (Fig 13).



Fig 13: Recall after 6 months (Picture A) and 12 months (Picture B) after placement. The wedge-shaped defects in the upper posterior region had been restored in the meantime.

## **REPORT**

## Case 2: 44-year-old patient: posterior bridge with zirconium oxide framework

#### **Preoperative situation**

After successful periodontal treatment, the interdental space between teeth 15 and 17 had to be closed. Both abutment teeth 15 and 17 were vital. Tooth 15 was crowned; tooth 17 showed a two-surface mesio-occlusal restoration (Fig 14).

## Planning

The interdental space between 15 and 17 was to be closed with an adhesively luted, all-ceramic inlay/crown-retained bridge with a zirconium oxide framework on which the veneering ceramic was pressed and layered in some areas. From a technical point of view, the easiest and best solution in this case was to press IPS e.max ZirPress onto the zirconium oxide framework. On the one hand, this allows the complex occlusal surface to be designed with a proven wax-up. On the other hand, the inlay in tooth 17 is much easier to fabricate by means of the press technique than with the layering method. The translucent LT ingot was used to ensure ideal adaptation of the restoration to the residual tooth structure.

# Preparation and fabrication of the restoration

Abutments 15 and 17 were prepared according to a crown preparation with a pronounced chamfer on tooth 15 and an MO inlay preparation with a proximal shoulder on tooth 17 (Fig 15). In the occlusal area, 1.5 mm were available for the bridge framework and veneer.



Fig 14: Orthopantograph after the periodontal treatment was completed. Interdental space 15-17 to be closed

After sulcus management, elastic impression taking, facebow transfer and registration of the horizontal and vertical jaw relation in the intercuspation position, the super hard stone models were mounted into a semi-adjustable articulator to fabricate the inlay/crown-retained bridge 15-17.

The zirconium oxide bridge framework was milled from an IPS e.max ZirCAD zirconium oxide block in the inLab system from Sirona. The sintered zirconium oxide was fitted to the master model. Once the framework was finished, the ZirLiner in the suitable shade was applied and fired (Fig 16).

A translucent pressed ceramic was used to press a circular shoulder to tooth 15 and the side walls of the inlay in tooth 17.

# Wax-up and preparation for the press procedure

A modelling wax that burns out without leaving residue has to be used for the wax-up. The teeth are modelled fully anatomically. A small portion of Incisal material is applied only in the buccal and lingual area (Fig 17).

If the pontics in the posterior region are voluminous, it is recommended to apply a ringshaped sprue (Fig 18) to achieve a detailed reproduction of the pontic (Fig 19).



Fig 15: Preparation of the abutments for the inlay/crown-retained bridge 15–17. Circular chamfer on 15, inlay cavity with proximal shoulder and slightly divergent cavity walls on tooth 17



### Completion

After the sprues had been removed and the restorations completely fitted on the master model, a little space was provided for the build-up in the incisal area (Fig 20).

To complete the anatomical form, the incisal area was built up according to the free layering technique with IPS e.max Ceram (Fig 21).

Finally, the restoration was stained with IPS e.max Ceram Shades and Essence materials and glazed (Fig 22).

The basal view shows the central whiteopaque IPS e.max ZirCAD zirconium oxide bridge framework which was entirely covered with the IPS e.max ZirPress veneering ceramic in the occlusal area and in the area of the preparation margins. IPS e.max ZirPress is suitable for the adhesive technique.

An inlay-retained bridge or a combined version, such as an inlay/crown-retained bridge has to be adhesively seated in order to achieve the clinically required retention and strength of the construction. As the zirconium oxide bridge framework exhibits only a very low translucency, a chemically or dual-curing adhesive and luting composite have to be used to ensure complete polymerization. In the present case, the preparations were isolated by means of electrosurgical sulcus management, iron-IIII-sulphate application and the placement of retraction cords (Ultrapak, Ultradent). It was not possible to use a rubber dam to establish a completely dry field. Therefore, the bridge was inserted under stringent moisture control. The retraction cords had to remain in place in the sulcus as far as possible during placement to avoid sulcus fluid from escaping and to protect the sulcus from penetration of adhesive and luting composite. In the present case, the chemically curing Multilink luting composite system was used for the adhesive technique. Before the bridge was seated, the restoration was conditioned with 5% hydrofluoric acid gel (IPS Ceramic Etching Gel) in the area of the etchable IPS e.max ZirPress ceramic and subsequently silanized (Monobond-S). Cement excess was removed with foam pellets, brushes and dental floss immediately after placement before the restoration was light cured. At the cementation joint a brush should be preferred over a foam pellet to prevent the luting composite from being wiped out of the cement margin. Figure 23 shows occlusal and buccal aspects of the restoration in situ. The fully veneered inlay/ crown-retained bridge seamlessly blends into the natural environment. The surrounding soft tissue looks vital.



Fig 16: Reduced zirconium oxide framework with fired

Zirl ine



Fig 17: Complete wax-up of the restoration



Fig 18: Attaching the sprues (note the ring-shaped sprue on the pontic



Fig 19: Complete and accurate reproduction of details



Fig 20: Pressed restoration completely seated on the master model



Fig 21: Build-up of the incisal areas



## Conclusion

The IPS e.max system currently offers ceramic materials for the fabrication of single tooth restorations (crowns, partial crowns, veneers) and 3 to maximum 4-unit bridges in the press and CAD/CAM techniques. Dental technicians can work with only one layering ceramic on the different framework materials and thus cover virtually all indications in allceramics. Dental technicians will appreciate the benefit of having to handle only one veneering ceramic, which will enable them to fabricate predictable restorations more efficiently.

Dental-lab work was fabricated by Franz Perkon and Andreja Mezan.



Fig 22: Completed inlay/crown-retained bridge 15-17: Picture A, B: view on the model; Picture C: basal view. The white-opaque zirconium oxide framework is visible in the center





## REPORT

## Previous issues of the Ivoclar Vivadent "Report"

Report No 1 (March 1984) \* G Beham Dentin adhesion of restorative materials

*Report No 2 (May 1985) \** Dr V Rheinberger and G Beham Adhesive bridges – new prosthetic possibilities

Report No 3 (May 1986) \* P Wollwage Veneering materials for crowns and bridges

*Report No 4 (December 1987)* \* Dr P Dorsch A review of proposed standards for metal-ceramic restorations

Report No 5 (January 1990) G Ott Composition and development of dental composites

Report No 6 (September 1990) \* G Beham IPS Empress: A new ceramic technology

*Report No 7 (November 1992)* Dr U Salz The restored tooth – a complex bonding system

Report No 8 (January 1993) G Zanghellini, D Voser Properties of resin based veneering materials

*Report No 9 (March 1993) \** R Grünenfelder Stratos 200: New possibilities in biogenic prosthetics

Report No 10 (July 1994) Prof Dr W Höland, Dipl Ing M Frank, Dr rer nat U Salz, Dr med dent G Unterbrink IPS Empress: Material and clinical science

Report No 11 (January 1997) K Hagenbuch HP Foser Artificial teeth – a symbiosis of materials, anatomy and science Report No 12 (December 1998) Prof Dr W Höland Dr med dent SD Heintze IPS Empress 2: All-ceramic bridges and more ...

Report No 13 (June 2000) A Kammann K Hagenbuch M Reis H P Foser Removable Denture Prosthetics: Materials Science, Aesthetics and Tooth Setup

Report No 14 (January 2001) Dr Dr med dent A Rathke Dr sc nat U Lendenmann Dentin adhesives: Excite in context

*Report No 15 (August 2004)* Dr G Zappini Ing HTL S Hopfauf U Spirig Focus on SR Adoro: Indirect Composites – Materials Science and Development

Report No 16 (February 2006) Dr V. Rheinberger Prof Dr H Kappert P Oehri T Specht Dr Dr A Rathke Dr T Völkel Dr SD Heintze Prof Dr JF Roulet HP Foser Dr A Stiefenhofer All-ceramic Report: All-ceramic Restorations – Materials Science and Development

\* out of print



Ivoclar Vivadent Report Published by Ivoclar Vivadent AG, Schaan, Liechtenstein Editor: T Volkel © 2006 by localar Vivadent AG, Schaan 600718/0606/e/W