IPS e.max® ZirCAD

Scientific Documentation

ivoclar vivadent

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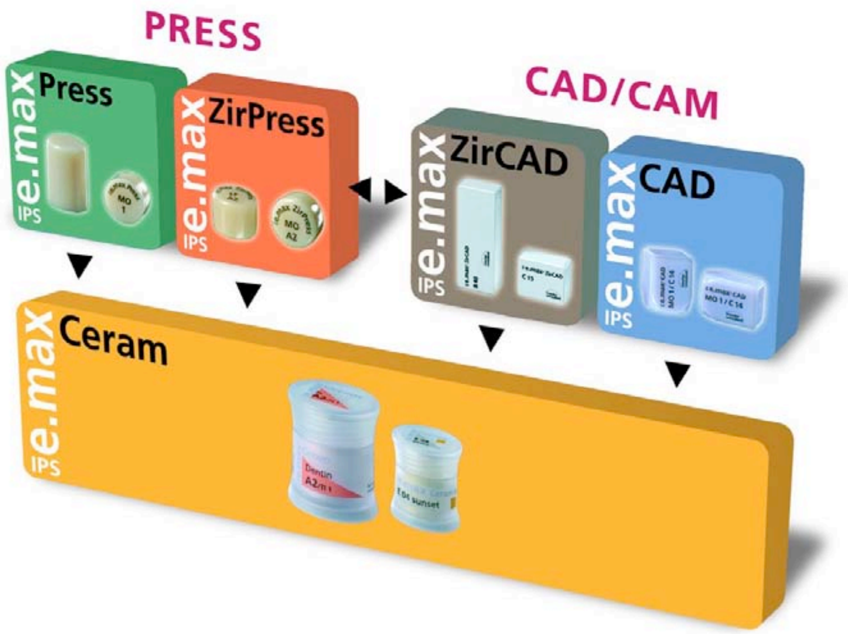
1. Introduction

1.1 Overview of IPS e.max range of products



IPS e.max is an all-ceramic system that consists of the following five components:

- IPS e.max Press (lithium disilicate glass-ceramic ingot for the press technique)
- IPS e.max ZirPress (fluorapatite glass-ceramic ingot for the press-on technique)
- IPS e.max CAD (lithium disilicate glass-ceramic block for the CAD/CAM technique)
- IPS e.max ZirCAD (zirconium oxide block for the CAD/CAM technique)
- IPS e.max Ceram (fluorapatite veneering ceramic)

1.2 Materials and technical terms [1,2,3]

1.2.1 Technical terms

- *Zircon* is the term for the $ZrSiO_4$ mineral (zirconium silicate). It is a component of the earth's crust and is used as a natural resource for the fabrication of zirconium oxide structural ceramics.
- *Zirconium* (Zr) is a shiny silvery metal. It is relatively soft and flexible when in a highly pure form; its most important compound is zirconium oxide ZrO_2.
- *Zirconium oxide*, *zirconium dioxide or zirconia* (ZrO_2) is used to refer to technical oxide ceramics. It is also the name for the natural deposit ZrO_2 (Baddeleyite). Baddeleyite is occurs in monoclinic form at room temperature. It is severely contaminated and not suitable for zirconium oxide structural ceramics.
- *Y-TZP*: **T**etragonal **Z**irconia **P**olycrystals: Partially stabilized ZrO_2 is achieved by adding yttrium oxide (Y_2O_3). The structure occurs as microcrystalline tetragonal zirconium oxide at room temperature.
- *PSZ*: **P**artially **S**tabilized **Z**irconia. By doping with e.g. MgO, at room temperature a ceramic is formed containing a tetragonal phase which is embedded in a matrix consisting of cubic ZrO_2.
- *Green body or green compact*: pressable ingot without heat treatment, i.e. an object which was pressed out of ceramic powder and bonding material. As the material has not been presintered, it is soft as chalk. Thus, processing is facilitated; however, due to the low inherent strength of the ingots, there are great problems concerning transport and handling. The green body features an open porosity, a linear loss of approx 25% has to be considered during the sintering process.
- *White body*: is a presintered ingot. By thermally pre-treating the material, the organic pressing additives have vanished and the ingot acquires sufficient inherent strength. During the presintering process, the white body undergoes a shrinkage of approx. 5%. When CAD/CAM technology is used to fabricate objects from white bodies, an additional shrinkage of 20% (linear) has to be considered during the final sintering process.
- *Martensitic transformation:* diffusion-free transformation of the lattice structure without thermal activation.

1.2.2 *Material science: Zirconium oxide*

Pure zirconium oxide (ZrO_2) occurs in different crystal structures, depending on the temperature (Fig. 1:).

When the zirconium oxide grains cool down from the molten state they go through the following crystal phases: cubic (k), tetragonal (t) and monoclinic (m) (see Fig. 2:).

Phase transformation t → m is a diffusion-free transformation (martensitic transformation). This transformation is associated with an increase in volume of 3 to 5%. This means that components made of pure ZrO_2 would burst due to the volume increase of the grains and the tension and microcracks affiliated with this increase. By adding various materials such as e.g. Y_2O_3, MgO or CeO_2, this phase transformation can be relocated towards lower temperatures, which enables the t phase to be stabilized at room temperature. This is achieved e.g by doping ZrO_2 with 3 mol-% (corresponds to 5.1 % by weight) Y_2O_3, called 3Y-TZP.

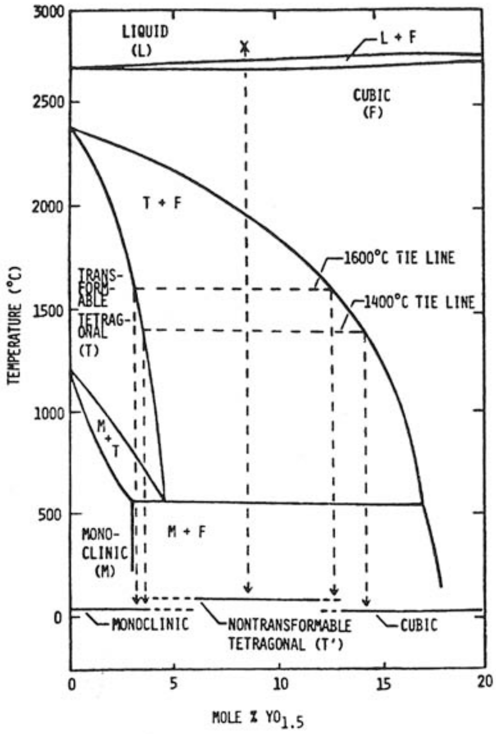


Fig. 1: Diagram of the phase that contains a high content of yttrium oxide in the Y_2O_3-ZrO_2 system [4]

The tetragonal grains of 3Y-TZP are in a metastable state at room temperature. The state is metastable because the transformation t → m can be induced by external influences like tensions, temperature and environment. This phase transformation and the volume increase associated with it can have highly advantageous effects. This is called *tension induced transformation strengthening*. The dreaded crack propagation in ceramic materials and the subsequently devastating fracture of the component can be delayed. The stress field at a crack tip causes the phase transformation t → m. The resultant volume increase of the transformed grains leads, on the one hand, to a widening of the crack tip and thus takes the pressure off the tip and, on the other hand, compresses the crack flanks. This provides the material Y-TZP with exceptionally high strength and high fracture toughness.

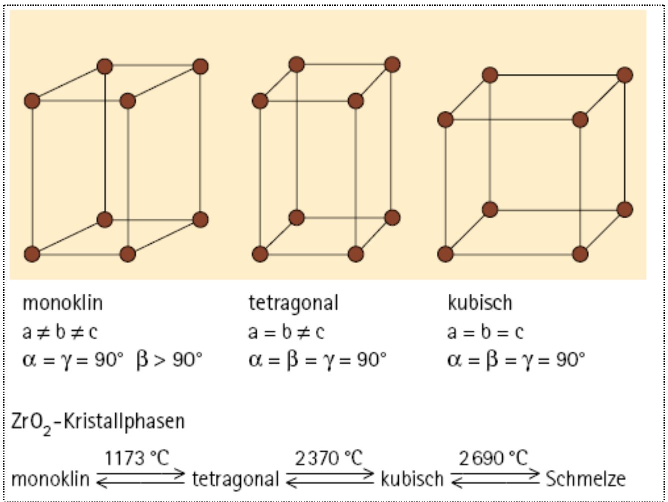


Fig. 2: Crystal phases and transition temperatures of pure zirconium oxide

1.3 IPS e.max ZirCAD

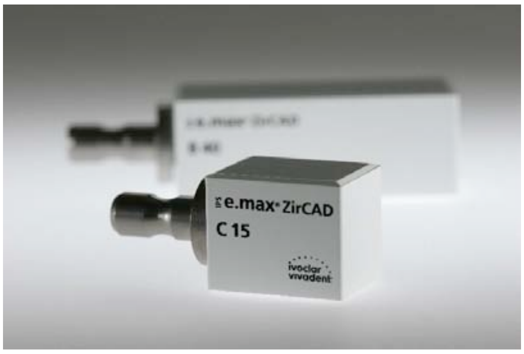


Fig.3: IPS e.max ZirCAD

IPS e.max ZirCAD is a presintered yttrium-stabilized zirconium oxide block (Y - TZP) for the CAD/CAM technology (Fig.3)

The microstructure of the block is very porous. The grains are only slightly connected with each other by weak sintering necks, which formed during the presintering procedure (Fig. 4).

The porosity is approx. 50%. The strength of the material is still very low, which enables easy processing.

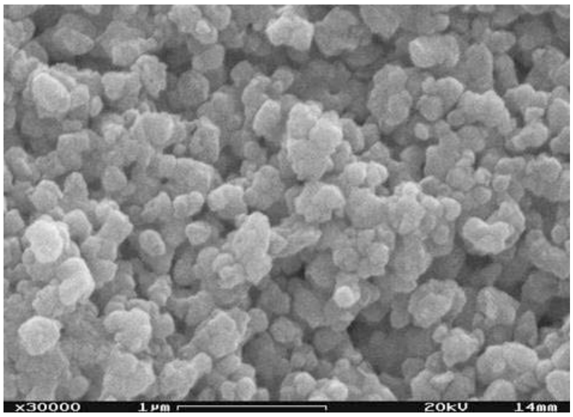


Fig. 4: Microstructure of the IPS e.max ZirCAD block (presintered); (SEM fracture surface)

After the restoration has been milled into shape with CAD/CAM technology, the material is sintered to densify the microstructure. The final restoration is densely sintered and consists of tetragonal grains (Fig. 5).

The density is approx. 99.5% of the theoretical density (TD). Strength and toughness have now reached the high values as desired.

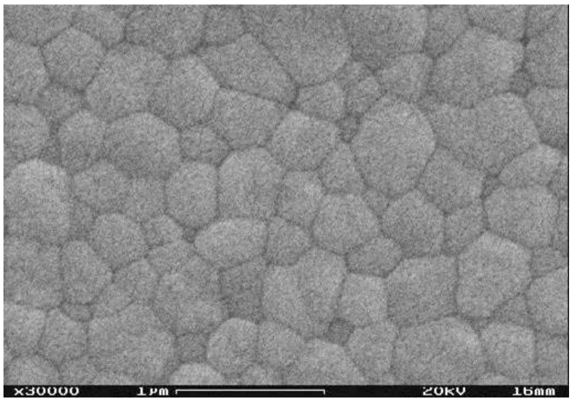


Fig. 5: Sintering structure of IPS e.max ZirCAD (SEM, thermically etched at 1420 °C for 15 min.)

2. Technical data

IPS e.max ZirCAD

Ceramic blocks for the CAD/CAM technology

<u>Standard composition:</u>	**(in wt %)**
ZrO_2	87.0 – 95.0
Y_2O_3	4.0 – 6.0
HfO_2	1.0 – 5.0
Al_2O_3	0.0 – 1.0

<u>Physical properties:</u>

According to:

ISO 6872 Dental ceramic

ISO 9693 Metal-ceramic dental restorative systems

Flexural strength (biaxial)		900 ± 50	MPa
Chemical solubility		< 10	$\mu g/cm^2$
Coefficient of thermal expansion	(100 - 400°C)	10.75 ± 0.25	$10^{-6}K^{-1}$
Coefficient of thermal expansion	(100 - 500°C)	10.80 ± 0.25	$10^{-6}K^{-1}$

3. Material science investigations

3.1 Physical properties

Chart 1: Physical properties

Property	**Value**	**Instructions for standards: ISO 13356:1997**[5]	**Instructions for standards: ISO 6872:1995/ Amd.1:1997**[6]
CTE (100°-400°C) [$10^{-6}K^{-1}$]	10.75 +/- 0.25		
Biaxial strength [MPa]	927 +/- 57	≥ 500	≥ 100
Hardness HV (F=98.1N) [MPa]	13’050		
Fracture toughness [MPa $m^{1/2}$]	5.5 +/- 0.22		
Density [g/cm^3] [%TD]	6.045 – 6.065 (99.4 – 99.7%)	≥ 6.00	
Medium size of crystallites [µm]	0.52+/-0.05	≤ 0.6	
Chemical solubility [$µg/cm^2$]	1		≤ 100

4. *In vitro* Investigations

4.1 *Incidence of chipping with/without liner*

The incidence of chipping of the veneering materials is an important clinical factor that provides a clue as to the survival chance or the need for repair of dental restorations.

For the purpose of testing the veneered crowns in the Willytec chewing simulator, the crowns were placed on standardized dies and subjected to eccentric loading with a steel antagonist. The antagonist performed a translational movement (depth of stroke = 2.0 mm, length of stroke = 5 mm, travel speed = 40 mm/sec) from the fossa up to 1 mm below the tip of the distobuccal cusp at loads from 3 and 5 to 9 kg. Each loading phase consisted of 100,000 loading cycles and 300 cycles of thermocycling (5°C/55°C).

In the Ivoclar Vivadent laboratory veneered crowns made of IPS e.max ZirCAD with and without the use of a liner were examined. IPS e.max Ceram was used as veneering material. The test measured the number of cycles that could be applied before chipping occurred.

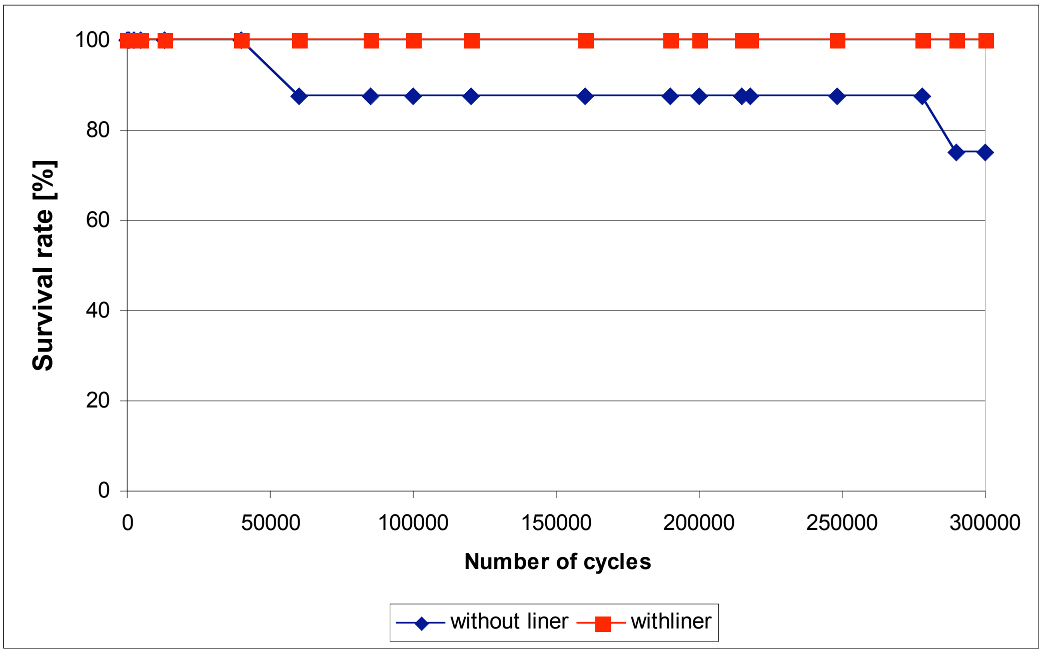


Fig. 6: Number of crowns (IPS e.max ZirCAD/IPS e.max Ceram) that passed the chewing simulation test without chipping (Ivoclar Vivadent AG Schaan, 2005)

- If a liner was applied, no chipping of the veneer occurred.

5. External clinical studies

5.1 University of Iowa

Head of studies: Prof. Stanford, Dental Clinical Research Center, University of Iowa, Iowa City

Title: Clinical performance of IPS e.max Ceram on IPS e.max ZirCAD

Objective: The clinical performance of IPS e.max ZirCAD bridges and crowns.

Experimental: Incorporation of 40 crowns and 10 bridges made of IPS e.max ZirCAD veneered with IPS e.max Ceram.

Results: Neither framework fractures nor chipping of veneering material was observed after all restorations had been incorporated.

5.2 Pacific Dental Institute

Head of studies: Prof. Sorensen, Pacific Dental Institute, Portland, Oregon

Title: Clinical performance of IPS e.max Ceram on IPS e.max ZirCAD

Objective: The clinical performance of IPS e.max ZirCAD as framework material for posterior bridges.

Experimental: Incorporation of 20 bridges made of IPS e.max ZirCAD veneered with IPS e.max Ceram.

Results: During the observation period of more than 6 months neither framework fractures nor chipping of veneering ceramic was observed.

5.3 University of Michigan

Head of studies: Prof. Fasbinder, University of Michigan, Ann Arbor

Title: Clinical performance of IPS e.max Ceram on IPS e.max ZirPress and IPS e.max ZirCAD

Objective: The clinical performance of IPS e.max ZirCAD. Half of the frameworks were veneered with IPS e.max Ceram and IPS e.max ZirPress was pressed on the other half.

Experimental: Incorporation of 30 crowns and 10 bridges made of IPS e.max ZirCAD/ IPS e.max ZirPress / IPS e.max Ceram.

Results: Neither framework fractures nor chipping of veneering material was observed after all restorations had been incorporated.

5.4 University of Munich

Head of studies: Dr. Beuer (Prof. Gernet), Clinical Center of the University of Munich

Title: Clinical study on all-ceramic restorations made of zirconium oxide ceramic veneered with a new veneering ceramic

Objective: The clinical performance of IPS e.max ZirCAD as framework material for crowns and bridges.

Experimental: Incorporation of 20 crowns and bridges (3 to 4 units) made of zirconium oxide (Y-TZP), veneered with IPS e.max Ceram.

Results: After the restorations had been observed up to one year, chipping of veneering material was recorded in one case.

5.5 University of Heidelberg

Head of studies: Prof. Rammelsberg, Clinical Center of the University of Heidelberg

Title: Clinical study on CAD/CAM fabricated, all-ceramic inlay-retained bridges based on zirconium oxide

Objective The clinical performance of IPS e.max ZirCAD with inlay-retained bridges.

Experimental: Thirty inlay-retained bridges were incorporated; each bridge included at least one inlay as bridge anchor. The frameworks were made of zirconium oxide, onto which IPS e.max ZirPress was pressed and finally veneered with IPS e.max Ceram.

Results: Neither framework fractures nor chipping of the veneering material have been reported up to this moment.

5.6 University of Aachen

Head of studies: Dr. Tinschert, Clinical Center of the University of Aachen

Title: Prospective clinical study on the survival rate of overpressed posterior crowns made of zirconium oxide

Objective: The clinical performance of IPS e.max ZirCAD molar crowns.

Experimental: Thirty posterior crowns featuring zirconium oxide copings made of DC-Zirkon, Lava and IPS e.max ZirCAD were incorporated. The copings were overpressed with IPS e.max ZirPress and veneered with IPS e.max Ceram.

Results: Neither framework fractures nor chipping of the veneering material have been reported up to this moment.

5.7 Summary

IPS e.max ZirCAD is a yttrium-stabilized zirconium oxide. This type of oxide ceramic has been in clinical use before and has proved its suitability for clinical applications. Featuring a flexural strength of 900 MPa, this material can be released for almost all indications that have been covered exclusively by metal up to now. The scientific documentations of IPS e.max Ceram and IPS e.max ZirPress report on the application and clinical investigations of the veneering material.

6. Biocompatibility

6.1 Introduction

IPS e.max ZirCAD is a yttrium-stabilized zirconium oxide (Y-TZP). Y-TZP is considered to be a highly biocompatible material, which has already been used in medical applications such as artificial hip joints. The endodontic post CosmoPost consists of the same material.

The biocompatibility results recorded for Y-TZP also apply to IPS e.max ZirCAD.

6.2 Chemical durability

Dental materials are exposed to a large variety of pH values and temperatures in the oral cavity. Chemical durability is therefore a highly important prerequisite for all dental materials. According to Anusavice[7] , ceramics are among the most durable dental materials.

Chemical durability according to ISO 6872:

	Chem. solubility [µg/cm²]	**Limit value according to standard [µg/cm²]**
IPS e.max ZirCAD	1	< 100

(Ivoclar Vivadent AG, Schaan, 2005)

- The chemical solubility of IPS e.max ZirCAD is far below the limit value according to the relevant standard.

6.3 Biocompatibility of yttrium-stabilized zirconium oxide[11]

The following tests were conducted on Y-TZP

Tests in vitro:	Zytotoxicity (cell cultures)
	Chromosome damage (mutagenicity test, Ames test)
	Haemolysis
Test in vivo:	(with rabbits, mice, guinea pigs and sheep)
	Haemolysis
	Acute toxicity (systemic)
	Irritation (intracutaneous)
	Sensitization

Pyrogenicity test

Short-term implantation (90 days)

Long-term implantation (24 months)

None of these tests showed a potential of Y-TZP to cause adverse health effects.

6.4 In vitro toxicity/in vitro genotoxicity

Josset et al.[8] investigated the biocompatibility of two implant materials – zirconium oxide and aluminum oxide in osteoblast cell cultures. Both materials were reported to have neither a toxic nor a genotoxic potential.

6.5 Biological reaction on implant material in vivo

Warashina et al.[9] implanted different materials (Y-TZP among others) in the skullcap of mice and traced the biological reactions (inflammation parameters, bone resorption). The biological properties confirmed the biocompatibility of Y-TZP when used for artificial hip joints.

6.6 Radioactivity

The radioactivity of CosmoPost was measured at the research center in Jülich[10]. The measured value of 0.005 Bq/g is much lower than the maximum value of 1.0 Bq/g permitted by ISO 6872.

In the examination report of Rieger[11], an activity U^{238} of 0.003 Bq/g has been recorded for zirconium oxide bio-ceramics.

6.7 Conclusion

In view of the present data and today's level of knowledge, it can be stated that IPS e.max ZirCAD does not feature a toxic potential. If the material is used according to the Instructions for Use, it does not pose any risk to the health of patients, dental technicians or dentists.

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Ivoclar Vivadent AG
Research and Development
Scientific Services
Bendererstrasse 2
FL - 9494 Schaan
Liechtenstein

Contents: Petra Bühler-Zemp / Dr. Thomas Völkel
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