IPS Style®

Scientific Documentation

ivoclar vivadent
passion vision innovation

Contents

1 Introduction

IPS Style® is an innovative ceramic system for the fabrication of metal-ceramic restorations, ranging from anterior crowns to multi-unit bridges.

IPS Style is based on newly developed glass-ceramic materials that contain leucite, fluorapatite and oxyapatite crystals. They enable the fabrication of restorations featuring high firing stability, minimal shrinkage and true-to-nature esthetics.

For the **conventional veneering technique** there is **IPS Style Ceram**, a highly esthetic metal-ceramic with leucite, fluorapatite and oxyapatite crystals for individualized design possibilities and virtually limitless creativity.

As a **single-layer ceramic**, **IPS Style Ceram One** offers a suitable option for quick and uncomplicated layering, depending on the respective patient requirements.

IPS Ivocolor is a **universal range of Stain and Glaze materials** for the individualized staining and characterization of ceramic materials. The range of products is coordinated with the layering, press and CAD ceramics from Ivoclar Vivadent and the zirconium oxides from Wieland Dental. The materials can be used regardless of the coefficient of thermal expansion of the ceramic.

Overview of the system:

Indication:

IPS Style

- Conventional multi-layer veneering ceramic for the most popular dental alloys (including electroplating) in the CTE range of 13.8–15.2 x 10^{-6}/K (25–500°C)
- One-layer veneering ceramic for the most popular dental alloys (including electroplating) in the CTE range of 13.8–15.2 x 10^{-6}/K (25–500°C) (IPS Style Ceram One)
- Veneers on refractory die material (only IPS Style Ceram)
- Characterization with IPS Ivocolor Shade and Essence stains
- Glazing with IPS Ivocolor Glaze

IPS Ivocolor

- Staining and/or characterization and glazing of ceramic materials from Ivoclar Vivadent and Wieland Dental:
    - ✓ veneering ceramics
    - ✓ press ceramics
    - ✓ milling ceramics
    - ✓ zirconium oxide (frameworks and full-contour restorations)

Contraindication:

IPS Style

- Bruxism
- Veneering of titanium and zirconium oxide frameworks
- Any other use not explicitly listed in the indications

IPS Ivocolor

- IPS Ivocolor is contraindicated for all applications not explicitly listed as indications.
- IPS Ivocolor is not suitable for staining, characterizing and glazing of partially crystallized IPS e.max® CAD restorations (in their blue state) and IPS e.max® CAD Veneering Solutions.

2 Material description

Since time immemorial, human kind has been aware of the medical and esthetic benefits of tooth replacements. More than 3500 years ago, the ancient Egyptians attempted to close gaps in dentition by carving false teeth out of mulberry wood and tying them to adjacent teeth with gold wire. Guided by this principle, the Etruscans produced more esthetic tooth replacements using bovine teeth. Until the beginning of the 19th century, ivory and natural human teeth taken from fallen soldiers at the battlefields of the time ("Waterloo teeth") were used to fabricate dentures. The first porcelain teeth were developed in 1709. However, it would not be until 1837 before industrial production of porcelain teeth began in England. With the fabrication of the first sets of dentures based on rubber and porcelain teeth (1846), a new era in denture prosthetics was ushered in [1; 2].

The suitability of ceramic materials for durable tooth replacements is limited because of their brittleness and susceptibility to fracture. Attempts to overcome these limitations by using metal frameworks were undertaken as early as 1733. However, it would not be until the nineteen-sixties before metal-ceramics would become available in dentistry with the patented use of gold alloys for the porcelain fused to metal technique. To date, the range of dental alloys has grown considerably and base metal alloys have also become available [3].

2.1 Basic materials science of dental ceramic materials

2.1.1 Basic raw materials

Materials must demonstrate several properties to be suitable for the production of highly esthetic and functional tooth replacements. They must not only offer optical properties that are similar to those of natural teeth (shade and translucency) but they must also be biocompatible. For use in the oral cavity, special attention must be devoted to hardness, elasticity, stability and wear. Glass-ceramic materials meet this versatile range of specifications.

Conventional dental ceramics are derived from porcelain, but they have a different composition. Although both dental ceramics and porcelain are based on the same three main components, namely feldspar, quartz and kaolin (also referred to as clay), Figure 1 clearly shows that they differ drastically when it comes to the concentration of these three components. While kaolin is the central component of porcelain formulations, making up approx. 40 - 70 % of the content by weight, this clay mineral is nowadays used only in very low concentrations in dental ceramics, if at all. The central component of the latter is feldspar. It is therefore misleading to use the term "porcelain" when referring to artificial teeth or veneering materials. The difference to porcelain increases the higher the proportion of feldspar in relation to kaolin is. The character of the ceramic becomes increasingly glasslike as these proportions are altered [3].

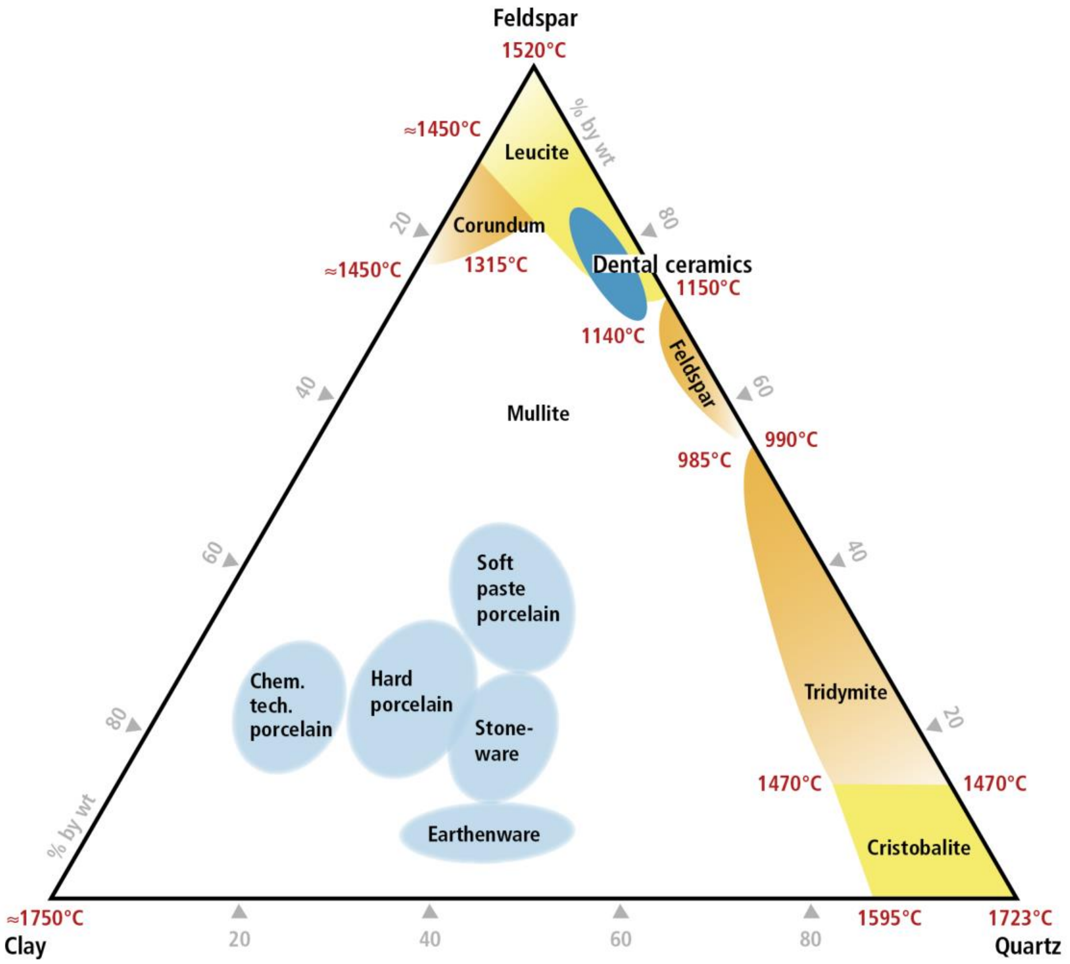
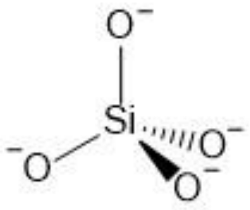


Figure 1: Ternary materials system of clay-feldspar-quartz and typical composition of certain ceramic products [adapted from [3]].

The three basic components fulfil specific functions in the ceramic material. For instance, the function of *kaolin* is to provide sculptability and plasticity to the material. Because of its low mechanical stability, it is only present in insignificant amounts in dental ceramic materials. The proportion of the glassy phase can be increased by the addition of *quartz*, one of the most widespread minerals. In nature, crystalline quartz (SiO_2) can be found in diverse modifications. The rose quartz used in dental ceramic materials has to be of very high purity as any potential impurities (metal oxides) may affect the quality of the product. Quartz of this quality is predominantly mined in Sweden and Norway. The central component of dental ceramics, *feldspar*, is also found in the required quality in deposits in Norway. Feldspar belongs to the group of framework silicates. Spatially, they are built from networks consisting of SiO_4 tetrahedra (Figure 2 and Figure 3). Some of the silicon ions (Si^{4+}) are replaced by aluminium ions (Al^{3+}) in the tetrahedral layers. The charge balance of the resulting negative charge of the oxygen ions is restored by various cations (e.g. sodium, potassium). This process results in the formation of mixed crystals of varying composition:



Description	Mineral	Composition
Sodium feldspar	Albite	$NaAlSi_3O_8$
Potassium feldspar	Orthoclase	$KAlSi_3O_8$
Lime feldspar	Anorthite	$CaAl_2Si_2O_8$

Figure 2: Left: $[SiO_4]^{4-}$ tetrahedron, basic unit of framework silicate networks. Right: Table of typical feldspar minerals

The properties of feldspar minerals are to a large extent determined by their composition. When potassium feldspar is melted (~1150°C), leucite crystals are precipitated ($KAlSi_2O_6$). Leucite has a higher melting temperature (1520°C), which enhances the stability of dental ceramics so that contoured objects keep their shape even when fired at high firing temperatures. In addition, leucite formation leads to an increase in the coefficient of thermal expansion (CTE) and improvement in mechanical stability.

As it is difficult to source raw materials of consistent quality and in adequate quantities for industrial production, base materials can now be produced synthetically. The chief advantages of these feldspar-free dental ceramics are the consistency in quality and the prevention of errors caused by impurities [3],[4].

2.1.2 Glass and ceramics – Glass ceramics

We often use the term "ceramics" when talking about dental ceramics, even if, strictly speaking, we not only mean pure ceramics but also glasses and glass-ceramics.

Glass is an amorphous material in which the $[SiO_4]^{4-}$ tetrahedra (see Figure 2) are spatially arranged in a random network. Their atoms only have a short range order and no long range order (Figure 3 a).

Ceramics, by contrast, have near and far configurations (Figure 3 b); they have an ordered crystalline structure [5].

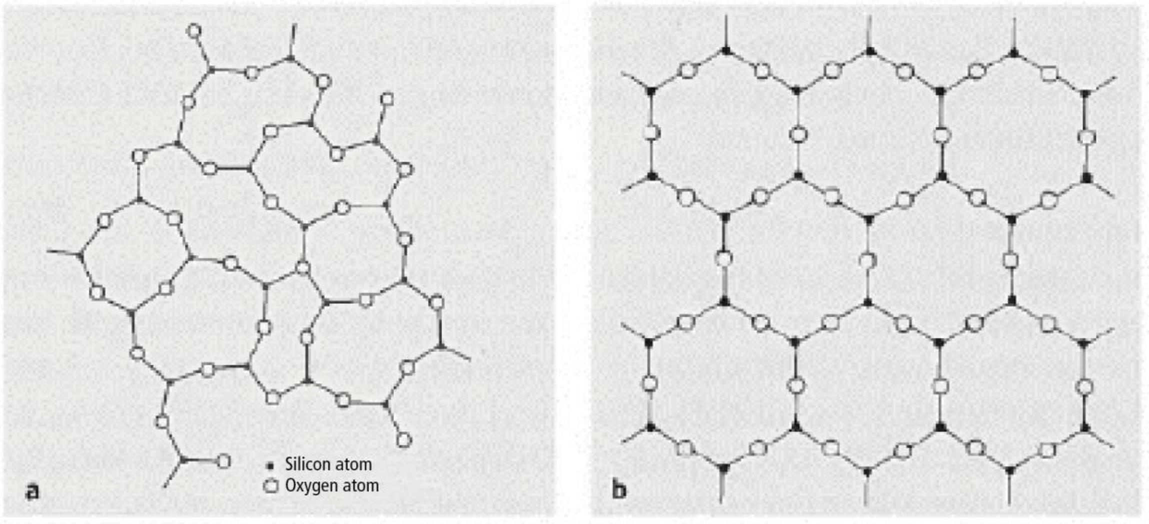


Figure 3: Amorphous glass structure (a) and crystalline structure of ceramic (b) [adapted from [5]].

The character of *glass-ceramics*, the material out of which many dental ceramics are made, arises from the definitions of glass and ceramic [5]. Glass-ceramics (Figure 4) are composed of both glass phases (amorphous areas) and crystal phases (crystalline areas). They must contain at least one of each type of phase; sometimes several crystalline and amorphous phases coexist in the same material. By using different types of crystals, defined properties can be obtained. For instance, controlled precipitation of leucite is used to increase the coefficient of thermal expansion while fluorapatite assists in regulating the light transmission of glass ceramics. Grain boundaries form between adjoining crystals. These boundaries may hinder the propagation of cracks and in this way they heighten the mechanical stability of the glass ceramic.

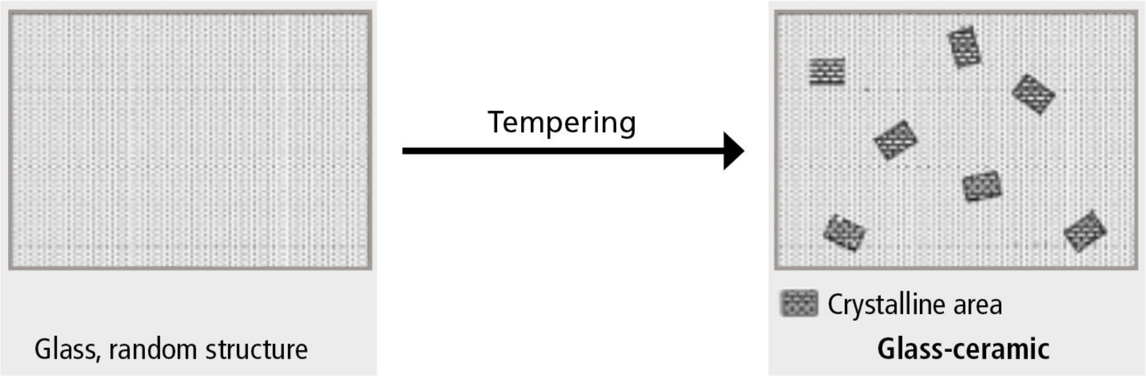


Figure 4: Glass-ceramic formation by controlled crystallization of amorphous glass [adapted from [5]].

Most glass ceramics are produced from ground base glasses by means of controlled crystallization in a complex tempering process. Two different mechanisms can be used to obtain glass ceramics. Controlled nucleation and crystallization within the body of the material (*volume crystallization*) or by tribochemical surface activation (*surface crystallization*). Which mechanism takes place depends on the base materials and the type of crystal being formed. Both mechanisms will be described in more detail below [6; 7].

2.1.3 Fabrication of glass-ceramics - volume crystallization and surface crystallization

Crystal formation in glass was an issue in glass manufacturing long before the advent of controlled crystallization. Crystallization in a glassy matrix may be uncontrolled. Uncontrolled crystallization is referred to as devitrification (or devit). This process is in most cases unwanted. The resulting products appear as a milky haze and uncontrolled crystal growth (inhomogeneous distribution of crystals of different sizes) results in stresses in the glass. These stresses lead to a high susceptibility to fracture. Increasing knowledge about the mechanisms involved in crystallization eventually made it possible to control this process to produce glass-ceramics with defined properties.

The crystallization process in glass ceramics is separated into two processes – nucleation and crystal growth, which occur either successively or partly overlapping. Rapid formation of nano-scale nuclei of an identical size is required to ensure an ideal progression of the controlled crystallization process. These nuclei will grow to uniform crystals that are homogeneously distributed.

Volume crystallization can be initiated and controlled by using a suitable chemical composition of special base glasses and additives and appropriate heat treatment. Nano-scale amorphous and crystalline phases form in a mechanism known as phase separation. Concurrently, drop and matrix phases of various compositions develop. All the constituents required for the formation of stable chemical bonds (nuclei/crystals) are concentrated in the droplets (blue spheres in Figure 5 a and c). During the growth of the microphases, various network formations (Figure 5 b) are passed through and this process allows the initially formed droplets to increase in size until they have attained their equilibrium size. Within the droplets, the individual components of the nuclei have to travel only very short distances to get to each other and build a nucleus. Nucleation is therefore rapid and results in nuclei of a uniform size. A shell forms around the droplets and this shell creates a boundary to the surrounding glassy matrix. The shell encapsulates the nucleus and inhibits further growth of the crystals [8].

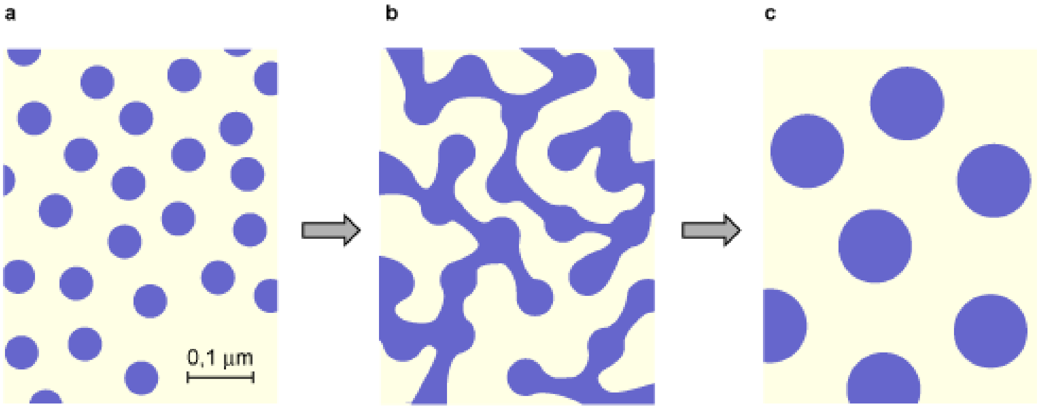


Figure 5: Schematic representation of the growth process of the phase-separated droplets (blue) in the glassy matrix (yellow) [9]

Droplet encapsulation results in the formation of crystals of a uniform size. Mass transfer of crystal components from the matrix phase to the encapsulated droplets begins to occur again only when the firing temperatures are increased. In the process, the crystals grow beyond the size of the former droplets and quickly join up due to the high number of initial nuclei, which keeps the dimensions of the crystals small. This normally leads to products that exhibit a considerably increased mechanical strength [8]. Fluorapatite crystals, for instance, are formed by a volume crystallization process.

The second mechanism that can be used for controlled crystallization is surface crystallization, which takes place at a faster rate. Nucleation and crystallization are induced on the surface of the glass grains. This mechanism results in e.g. the precipitation of leucite and oxyapatite. Glass ceramics are manufactured by powder technology. The glass is mixed from ground raw materials in a suitable blend. The mixture is then subjected to a melting and quenching process. The resulting glass frits are again milled to obtain a glass powder with a mean particle size of 20-40 µm. In the process of fine grinding (tribochemical activation), the chemical bonds are broken and reactive OH$^-$ groups form on the surface of the glass grains. The resulting reactive grain surfaces act as initial nucleation sites to induce surface nucleation. The crystallization process, which occurs simultaneously with the sintering process, can be explained by Figure 6 [6; 7].

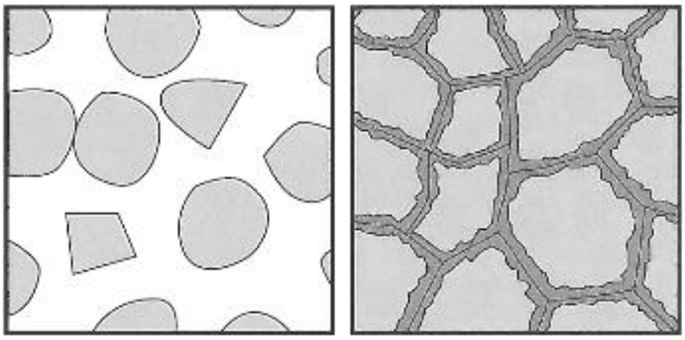


Figure 6: Schematic representation of the surface crystallization process. Left: individual glass grains. Right: crystallization on the surface during tempering [6]

The crystals grow from the surface of the initial glass grain into the inside of the glass during the sintering process. This results in the formation of dense microstructure. The newly formed crystals are located along the boundaries of the initial grains in the completely sintered glass ceramic. This leads to the phenomenon shown in Figure 7. The leucite crystals appear at growth fronts that look like a string of pearls.

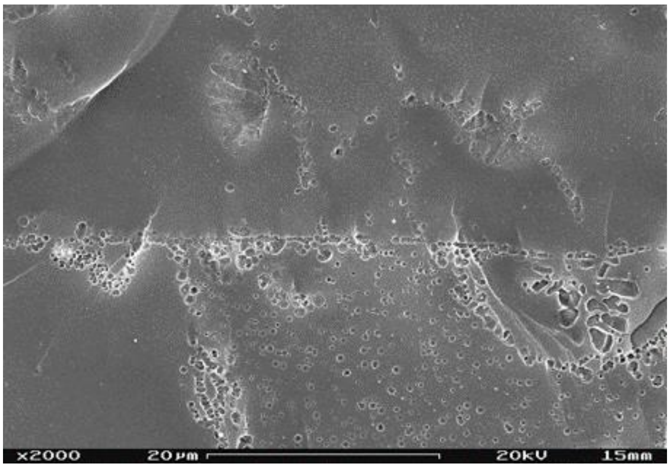


Figure 7: Pearl-string-like growth fronts of leucite crystals in glass ceramics (R&D Ivoclar Vivadent AG, Schaan, FL)

In-depth knowledge of the nucleation and crystallization mechanisms and the application of complex tempering procedures make it possible to stop crystal growth once the desired crystal size has been attained. The resulting glass ceramic is then milled and mixed with other glass-ceramic powders. The final mixture may consist of different crystalline phases and this allows us to unite the advantages of individual crystals in a tailor-made veneering ceramic. IPS Style combines the properties of the three crystal phases oxyapatite, leucite and fluorapatite in a single material.

2.1.4 Sintered structure formation by vitrification

Glass-ceramic materials are normally sintered using a viscous flow process. With increasing temperature, a liquid phase forms, which facilitates the form change and flow of the amorphous particles. Initially, sintering necks form between neighbouring particles and over time the contact surface between the necks enlarges and gaps are eliminated in the process. This results in a densification of the body. A schematic representation of this process is shown in Figure 8:

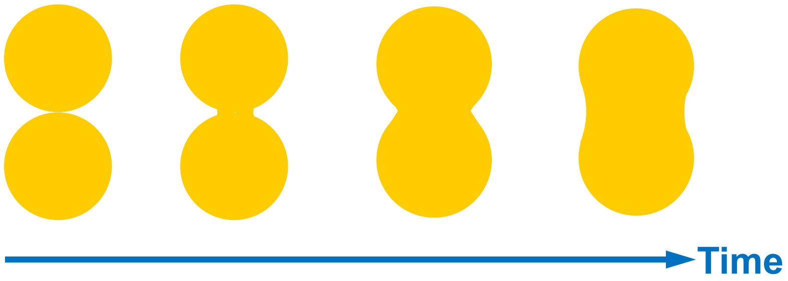


Figure 8: Schematic representation of the vitrification process. At the beginning of the process, sintering necks form between the individual particles.

Similar to all densification processes, vitrification is driven by free enthalpy minimization, coupled with volume reduction and the formation of energetically favourable grain boundaries in place of energetically unfavourable grain boundaries. The vitrification process is determined by geometrical, kinetic and thermodynamic factors and is, in other words, dependent on the grain size of the glass-ceramic particles as well as the viscosity and surface tension of the glass phase.

The real life version of the schematic process depicted in Figure 8, is shown in Figure 9 below for IPS Style. Undyed base material was used for this example. The scanning electron micrograph (SEM image) (a) shows the initial state of the sample after it had been technically compacted and dried (400x magnification; all other SEM images in Figure 9: 1000x magnification). The compacted glass-ceramic particles (sharp-edged grains) are clearly identifiable. When the material was heated to 600°C (b), hardly any changes from the initial state were noticeable. A similar sintered structure was also observed in the samples that were heated to 650°C (c), 670°C (d) and 700°C (e). Image (f) shows the sample at a temperature of 720°C, at which point sintering necks had begun to form (see red circle). The contours of the grains appear more rounded compared to the initial images (see blue arrows), indicating the beginning of flow. As the firing process progresses (g-h), the particles that have come into contact with each other begin to merge due to viscous flow. From a temperature of 800°C (i), the microstructure is densely sintered.

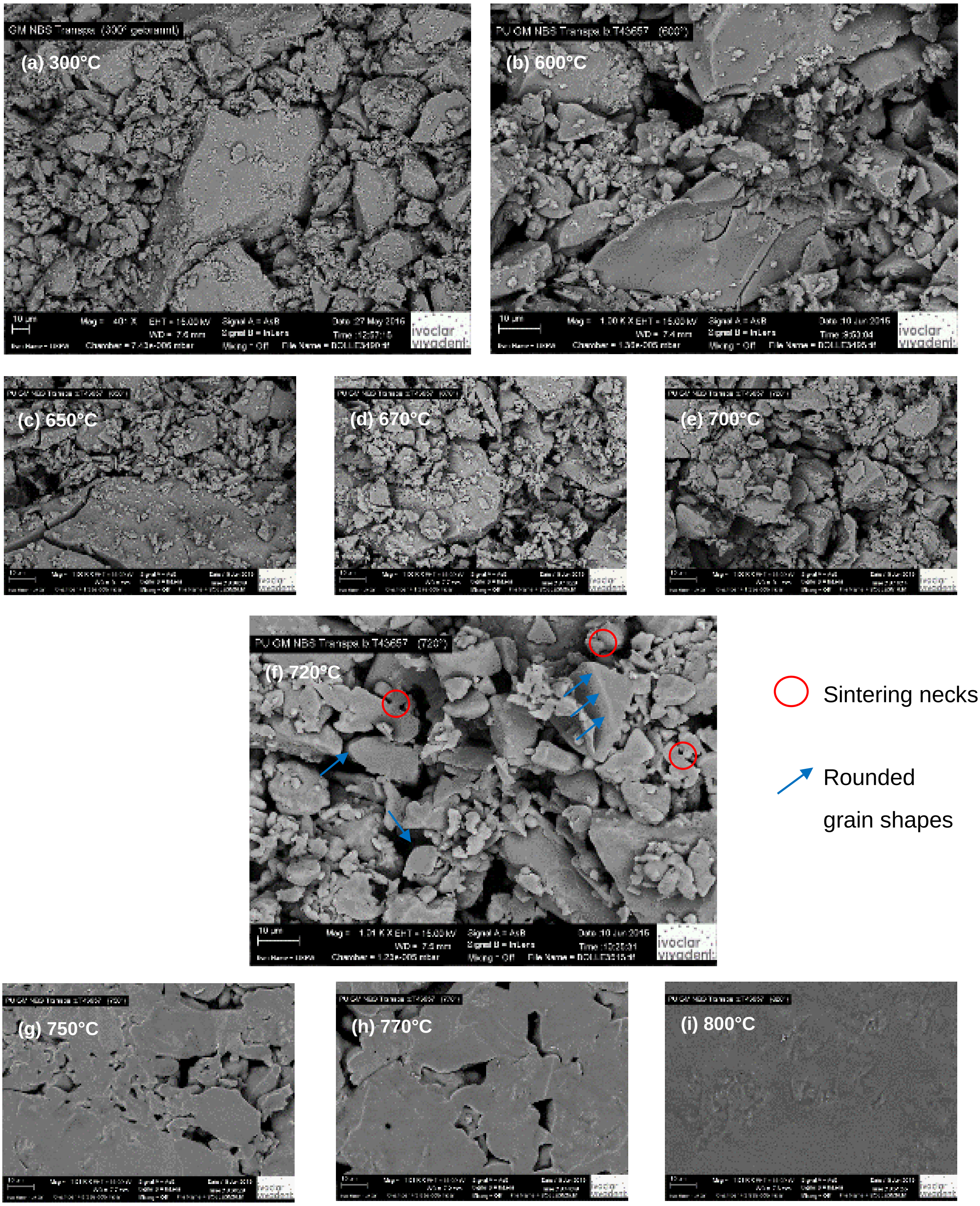


Figure 9: Temperature-controlled sintered structure formation in an IPS Style base material, between 300 °C and 800 °C (R&D Ivoclar Vivadent AG, Schaan, FL, 2015).

2.2 Metal-ceramic bond[1]

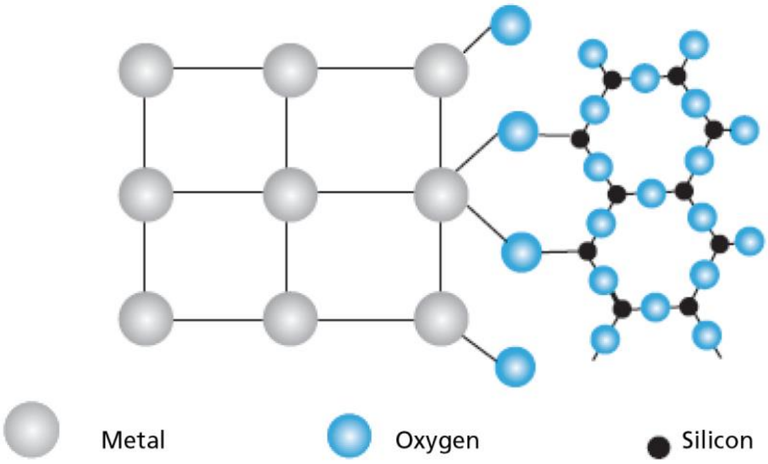
Good wettability of the alloy surface by the opaquer, which is very viscous at high temperatures, is a prerequisite for an optimal bond. In essence, however, the metal-ceramic bond is based on adhesion, i.e. the bonding effect between a solid interface (in this case the metal surface) and a second phase (in this case the ceramic). The most important of the numerous adhesive mechanisms are described below:

- **Adhesion by mechanical bond**

The ceramic bonds mechanically to the metal surface by filling depressions and/or enclosing protruding structures and anchor points which are present on the surface after metal conditioning. In addition to this mechanical bond, the ceramic demonstrates a certain compressive strain because its coefficient of thermal expansion is lower than that of the alloy, which increases retention.

- **Adhesion by chemical bond**

Processes such as chemical reactions, dissolution processes, redox processes, diffusion and precipitation result in the formation of a characteristic transition area at the interface between metal and ceramic. Particularly in the presence of non-precious alloy components, a certain saturation of both metal and ceramic with metal oxides occurs. Ideally, this results in the formation of an oxide monolayer, which is a component of both the metal and the ceramic. The resulting bond energies and electronic structures are then identical at each point of the interface. The prerequisite for this behaviour is an oxide layer that is formed on the metal surface during oxide firing.



This bonding mechanism can be depicted using the following model:

Figure 10: Model of the chemical bond adapted from[2]

The chemical bond is initiated by oxygen atoms which are present in both the metal layer and the ceramic and thus link the two materials.

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[1] "Focus on: Alloy/ceramic bond, IPS InLine", Ivoclar Vivadent AG, 2005.

[2] Schnettger A., Fachhochschule Osnabrück 2004.

- **Adhesion by intermolecular forces**

Short-range forces known as Van der Waals forces act between the alloy and ceramic. Their contribution to the adhesive bond is smaller than that of the mechanical and chemical bond.

2.3 Materials science of IPS Style and IPS Ivocolor

2.3.1 IPS Style and the functions of leucite, fluorapatite and oxyapatite

2.3.1.1 IPS Style

IPS Style is a feldspar-free metal-ceramic veneering material that emulates the high qualities of feldspar ceramics and even outperforms the latter as it is fired at a lower temperature and thereby offers an increased range of applications and reliability. In addition, the material sets new standards in terms of esthetics.

IPS Style is characterized by:

- high firing stability (contour stability) when fired several times
- optimum wetting of the opaquer without lifting or pulling away of the ceramic
- oxyapatite crystals for controlling the translucency
- fluorapatite crystals for controlling the brightness
- high shade stability and consistent brightness when fired several times (no greying)
- low firing temperature

The chemical basis of IPS Style consists of mixed glass-ceramics containing *leucite*, *fluorapatite* and oxyapatite as crystal phases (Figure 11). The content and distribution of the crystals in these glass-ceramics, as well as their viscosities, are responsible for the physico-chemical properties of IPS Style together with the carefully adjusted grain size distribution in the layering material. This material principle has been implemented for all materials, from the opaquer to the incisal, and ensures the optimum build-up of a restoration with regard to adhesion, compatibility and esthetic appearance.

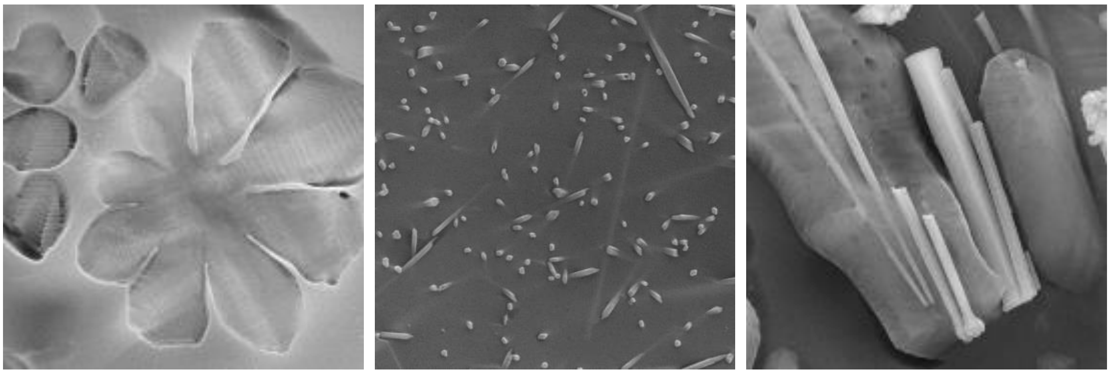


Figure 11: The three crystalline phases of IPS Style (not true to scale, for the purpose of better illustration). From left to right: leucite, fluorapatite and oxyapatite (R&D Ivoclar Vivadent AG, Schaan, FL, 2015)

The crystal phases are incorporated into the final product via glass-ceramic components, which contain at least one of these three phases. To manufacture these glass-ceramic components, glasses of a relevant composition are heat treated, either alone or in a blend, to precipitate the crystal phases. This process results in powdered leucite, fluorapatite and leucite oxyapatite glass-ceramics which are mixed with each other and/or with other base melts before they are tempered another time. After final grinding, the materials are dyed.

2.3.1.2 Functions of leucite, fluorapatite and oxyapatite crystals

The three different crystal phases fulfil different functions in the IPS Style glass-ceramic.
Leucite ($KAlSi_2O_6$) plays a central role in metal-ceramics. Due to its high coefficient of thermal expansion, the thermal expansion of a glass ceramic material can be defined by adjusting the crystalline leucite content. This is a basic requirement when a ceramic material is to be used as a veneering material on different dental alloys. In addition, the strength of the glass-ceramic is enhanced through the isotropic distribution of leucite crystals and their size in what is known as dispersion strengthening. The effect of dispersion strengthening is to retard crack propagation along the interfaces between the crystal phase and the glassy matrix by which all glass-ceramic materials are *a priori* afflicted.
While the leucite-containing base material forms the "backbone" of IPS Style, the other two crystal phases also play a decisive role in this mixed glass-ceramic. Both of them belong to the group of apatites. This group includes hydroxyapatite ($Ca_5(PO_4)_3OH$), which is the main component of human bones and teeth [6]. Fluor- and oxyapatite ($Ca_5(PO_4)_3F$ and $NaY_9(SiO_4)_6O_2$) differ from each other not only in terms of their structural units and their formation process, but also in terms of the functions they fulfil in IPS Style. Fluorapatite forms through microphase separation and volume cyrstallization, while oxyapatite is precipitated by surface crystallization. Needle-shaped fluorapatite (Figure 11) possesses a refractive index that is only slightly different from that of the surrounding glass. Therefore, a certain level of diffuse scattering occurs in respect to incident light, which causes the glass-ceramic material to look bright without a loss in translucency. This effect is also used in IPS Style to achieve a consistent brightness in layered restorations, regardless of the number of firings. Translucency is mainly controlled by the amount of oxyapatite. For the first time, a patented glass-ceramic that contains leucite, along with oxyapatite as additional crystal phase, has been developed. This allows the thermal coefficient of expansion of the layering material to be kept at a consistent level regardless of the level of opacity. Similar to fluorapatite, oxypatite also crystallizes in a needle-like form - a property that has a beneficial effect on the esthetic appearance of the restoration. The amount of oxyapatite present in the glass-ceramic determines its translucency. It therefore makes sense to include a high amount of oxyapatite in the opaquer to minimize its translucency, or inversely, to maximize its opacity. The Margin, Deep Dentin and Dentin materials contain significant amounts of oxyapatite, while the Incisal materials comprise only small amounts of this crystal phase. By nature, Transpa materials do not require oxyapatite.
The gradual progression in the amount of oxyapatite from the opaquer to Transpa Natural and exemplary sections of their microstructure are shown qualitatively in Figure 12.

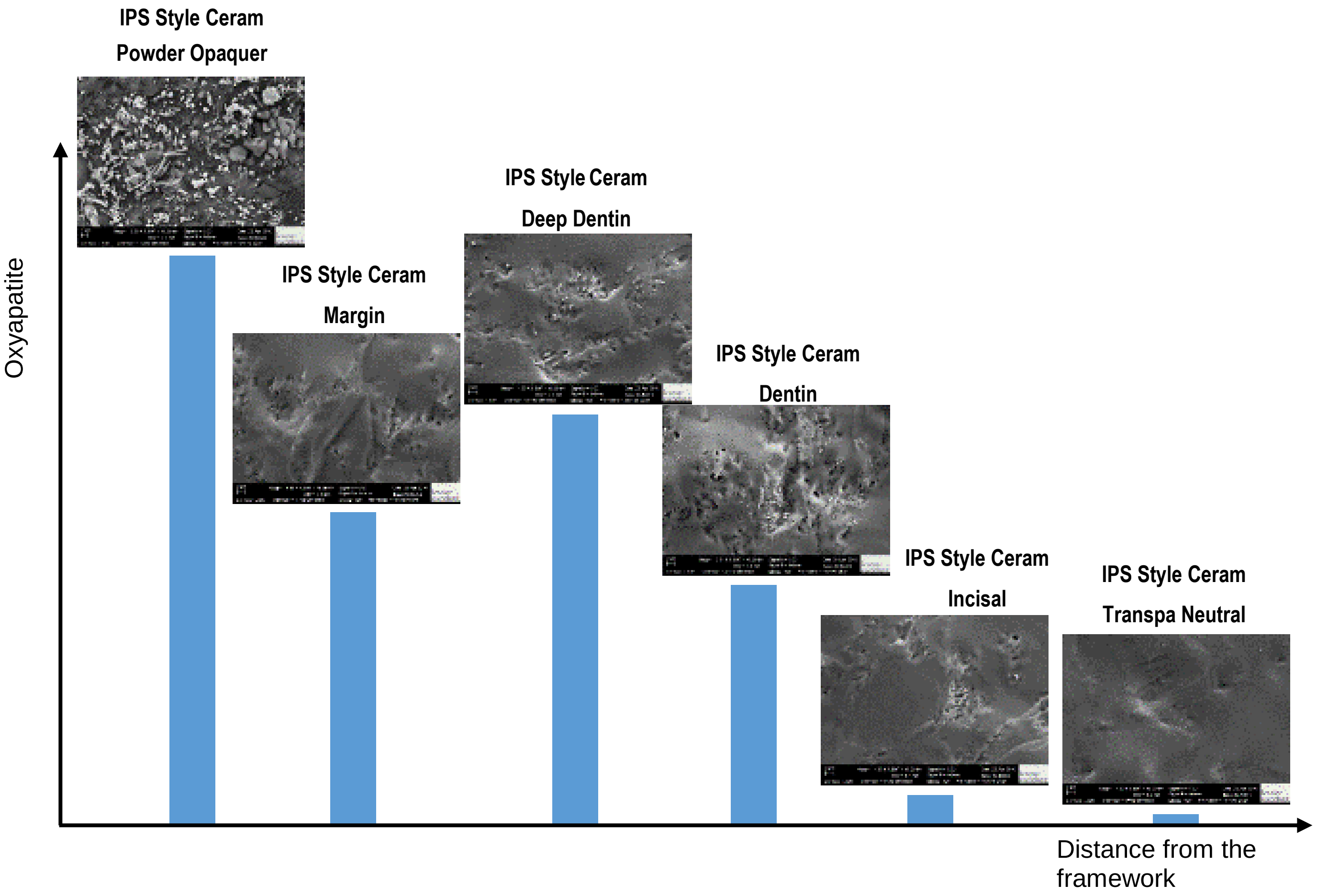


Figure 12: Qualitative progression of the oxyapatite content in a layered restoration (not to scale) (R&D Ivoclar Vivadent AG, Schaan, FL, 2015)

2.3.2 Metal-IPS Style bond and translucency

A metal-ceramic restoration is a material composition that is based on a durable bond between an alloy and a ceramic material. The interface between metal and ceramic is formed by the opaquer, which, if carefully applied and fired, produces a sound bond between the two materials. Figure 13 shows cross-sections of the transitional area between opaquer and ceramic at different degrees of magnification. The metal-ceramic bond in IPS Style is of good quality and exceptionally homogeneous. The differences in the translucency of the three layers can be clearly seen. As it contains a high amount of oxyapatite, the opaquer demonstrates the lowest translucency of the layers (see Figure 12). The translucency increases with increasing distance from the alloy.

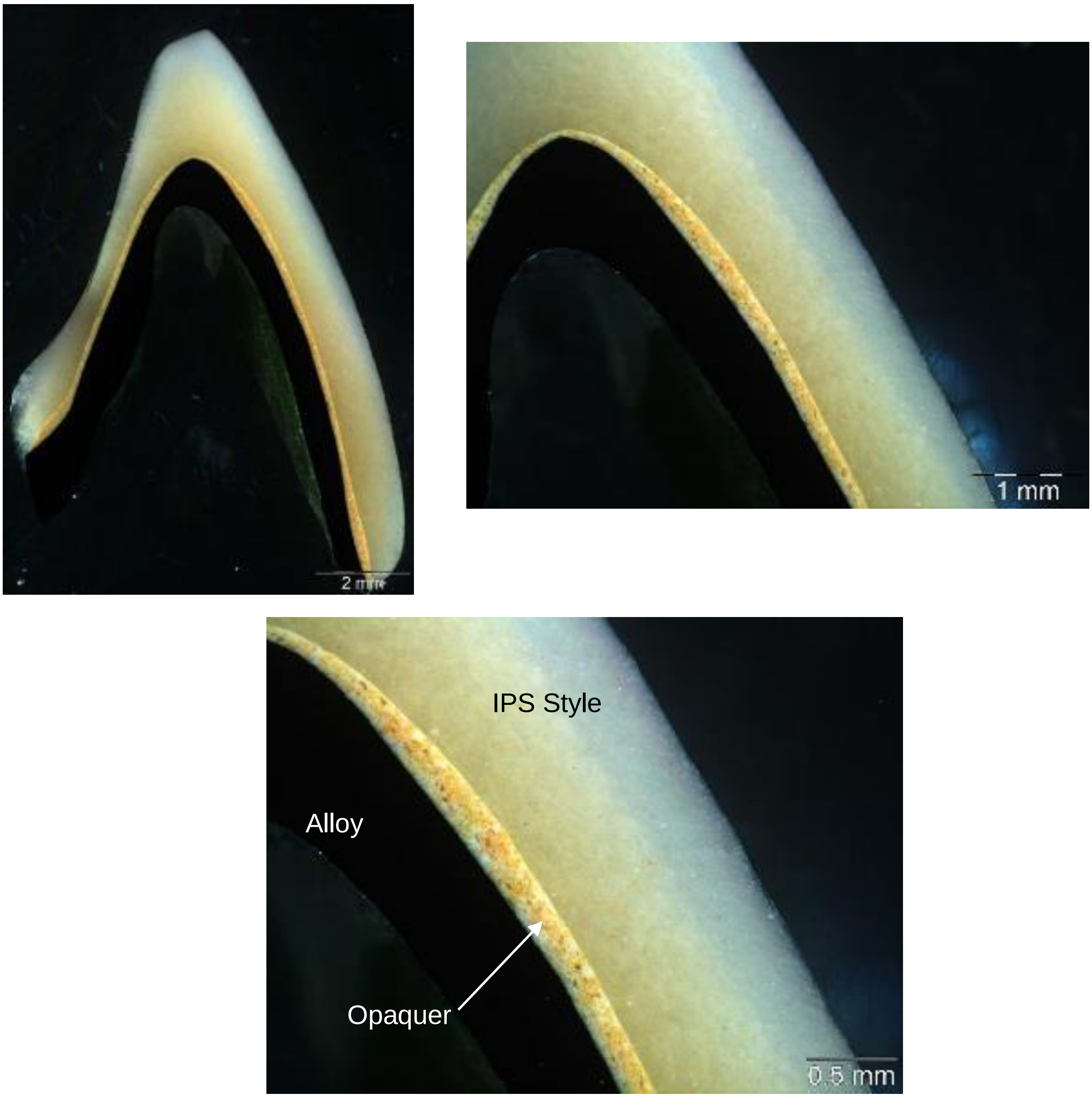


Figure 13: Optical cross-sections of the metal/opaquer/ceramic transition in an IPS Style crown (R&D Ivoclar Vivadent AG, Schaan, FL, 2015)

2.3.3 IPS Ivocolor

IPS Ivocolor is a universal range of Stain and Glaze materials for the individualized staining and characterization of ceramic materials. The chemical composition and grain distribution of the newly developed glass allow it to be applied to the layering, press and CAD/CAM ceramics from Ivoclar Vivadent and the zirconium oxide materials from Wieland Dental, regardless of the coefficient of thermal expansion of the ceramic. Optimum esthetic results can be achieved on every ceramic substrate due to the optimized sintering behaviour of the new formulation. Finely ground glasses, select shade compositions, a newly developed gel delivery form for the pastes and coordinated liquids facilitate the handling of the stains and glazes and lead to esthetically brilliant restorations. The formulation of the paste was designed to achieve optimum application and firing characteristics. Adaptation to the modified sintering behaviour has been achieved by reducing the additives and by targeted selection of the ingredients. This enables highly esthetic results to be achieved even within the lower firing temperature range. In addition, the pastes feature shear-thinning thixotropic properties to ensure that they remain stable when stored in their undiluted state. The gel-like consistency can be adjusted to suit the ceramic onto which the material is to be applied. That is, the degree of dilution and thus desired flow behaviour can be individually adapted. If the material is only slightly diluted, it can be applied in larger thicknesses and it is less likely to flow off and accumulate in certain areas, such as fissures. Conversely, a somewhat higher degree of dilution facilitates the application of material in a homogeneous layer on large surfaces.

Figure 14: Stained crown after the Stain and Glaze firing

3 Technical data

3.1 IPS Style

- Composition:

Product	**Product category**
IPS Style	**Ceramic for metal-ceramics**

IPS Style Ceram Powder Opaquer 870, Margin, Intensive Margin, Dentin, Deep Dentin, Incisal, Transpa, Mamelon, Occlusal Dentin, Incisal Edge, Opal Effect, Special Incisal, Inter Incisal, Cervical Transpa, Add-On, Bleach Bl, Gingiva, Intensiv Gingiva, Basic Gingiva,

IPS Style Ceram One

Function	**Substance/Component**	**Weight**	**(%)**	
		IPS Style Ceram Powder Opaquer	IPS Style Ceram Add-On	IPS Style Ceram - other
Ceramic	**Dental ceramic**	75 - 85	20 - 30	> 99
Glass	**Glass**	---	70 - 80	---
Pigment	**Pigments**	15 - 25	< 1	< 1

- Physical properties:

Product	**Product category**		
IPS Style **IPS Style Ceram** **IPS Style Ceram One**	**Metal-ceramic (powder)**		
Description			Product name
Principal group	Subgroup	ID	Included in subgroup
---	Pastes	1.1	---
IPS Style Ceram IPS Style Ceram One	Powder	2.1	IPS Style Ceram Margin
		2.2	IPS Style Ceram (Deep Dentin, Dentin, Incisal, Transpa, Impulse, Gingiva)
		2.3	IPS Style Ceram Opal Effect
		2.4	IPS Style Ceram One
		2.5	IPS Style Ceram Add-On Margin
		2.6	IPS Style Ceram Add-On Dentin/Incisal/BL
		2.7	IPS Style Ceram Add-On 690 °C
		2.8	IPS Style Ceram Powder Opaquer 870

Properties		Group **IPS Style Powder**					**Unit of measure**
		Specification or subgroup ID					
		2.1	**2.2**	**2.3**	**2.4**	**2.5**(1)	
* **Flexural strength** (σ)		≤ 50					MPa
* **Coefficient of thermal expansion** (span: 25-500 °C or Tg)	2 firings	12.8 ±0.5	12.0 ±0.5	12.0 ±0.5	12.0 ±0.5	12.7 ±0.5	$10^{-6}K^{-1}$
	4 firings	13.1	12.4	12.4	12.4	13.3	
	Mean	13.0	12.2	12.2	12.2	13.0	
* **Glass transition temperature** Tg		580 ±20	525 ±20	510 ±20	525 ±20	480 ±20	°C
* **Firing temperature**		840	790	790	790	750	°C
* **Chemical solubility**		< 100					µg·cm^{-2}
* **Radioactivity U^{238}**		≤ 1					Bq/g
** **Metal-ceramic bond** (crack initiation) (Schwickerath)		> 25					MPa

Properties		Group **IPS Style Powder** **Specification or subgroup ID** **2.6**(1)	**2.7**(1)	**2.8**			**Unit of measure**
* **Flexural strength** (σ)		≤ 50					MPa
* **Coefficient of thermal expansion** (25–400°C)	2 firings	13.0	12.0	13.5			$10^{-6}K^{-1}$
	4 firings	13.6 ±0.5	12.4 ±0.5	13.7 ±0.5			
	Mean	13.3	12.2	13.6			
* **Glass transition temperature** Tg		475 ±20	455 ±20	540 ±20			°C
* **Firing temperature**		750	690	870			°C
* **Chemical solubility**		< 100					$\mu g \cdot cm^{-2}$
* **Radioactivity** U^{238}		≤ 1					Bq/g
** **Metal-ceramic bond** (crack initiation) (Schwickerath)		> 25					MPa

The product meets the relevant performance criteria as described in

*** EN ISO 6872:2015 Dentistry - Ceramic materials**

**** EN ISO 9693-1:2012 Dentistry - Compatibility testing - Part 1:Metal-ceramic systems**

3.2 IPS Ivocolor

- Composition:

Product	**Product category**
IPS Ivocolor IPS Ivocolor Essence IPS Ivocolor Shade IPS Ivocolor Glaze	**Powders and pastes for the characterization/staining and glazing of ceramic materials**

Function	**Substance/Component**	**Weight**			
		Essence	**Shade**	**Glaze**	
				Paste	Powder
Glass	**Glass**	75 - 99	59 - 69	65 - 75	100
Viscosity control	**Glycols, water**	---	25 - 35	25 - 35	---
Pigment	**Pigments**	1 - 25	1 - 11	---	---

- Physical properties

Product	**Product category**		
IPS Ivocolor Glaze, Shade, Essence	**Staining/characterization of ceramic materials**		
Properties	**Unit of measure**	**Specification**	
Flexural strength	MPa	≤ 50	
Chemical solubility	$\mu g/cm^2$	< 100	
Coefficient of thermal expansion (25-T_G°C)	$10^{-6}K^{-1}$	Glaze	9.3 ± 0.5
		Glaze FLUO / Essence / Shade; concentration of pigments ≤ 4%	9.2 ± 0.5
		Essence / Shade; concentration of pigments > 4 to ≤ 12%	9.1 ± 0.5
		Essence / Shade; concentration of pigments > 12%	8.8 ± 0.5
Glass transformation temperature	°C	460 ± 20	
Radioactivity U^{238}	Bq/g	≤ 1	
The product meets the performance criteria defined in EN ISO 6872:2015 Dentistry – Ceramic materials (ISO 6872:2015) Type 1 Class 1.			

4 Characteristic material properties of IPS Style

4.1 Investigation of the glass-ceramic microstructure of IPS Style

Examinations of the glass-ceramic microstructure were conducted at the R&D department of Ivoclar Vivadent AG, Schaan, FL, in 2015.

For the microstructure analysis, fragments of freshly broken specimen discs were etched with 3% hydrofluoric acid (HF) for 10 seconds and then carefully cleaned and dried. The resulting fracture surfaces were coated with a conductive layer and then examined by SEM for typical structural properties.

Crystalline components can be identified by their distinct shape (habit) and by phase analysis using X-ray diffraction (XRD) on powder. The results of both methods are shown below.

4.1.1 *IPS Style Ceram Powder Opaquer 870*

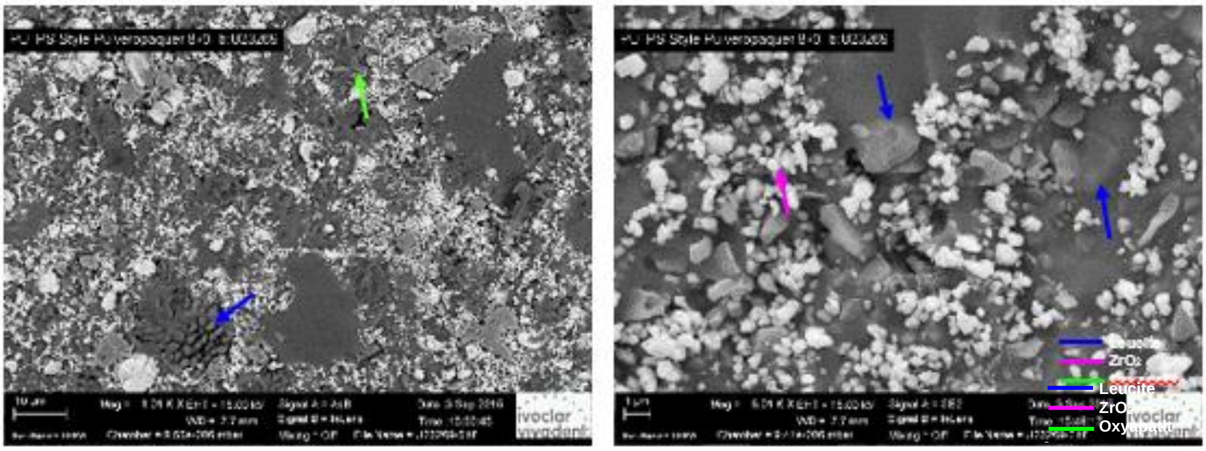


Figure 15: Microstructure of IPS Style Ceram Powder Opaquer 870 A2

Figure 15 shows the typical microstructure of dyed IPS Style Ceram Powder Opaquer 870, containing leucite (blue arrows), needle-like oxyapatite (green arrow) and various pigments to impart colour and opacity to the material. In the case of shade A2, the white bulb-shaped particles are mainly zirconium oxide (ZrO_2) or zirconium-oxide-based pigments. The crystalline phases are detectable on the X-ray diffraction image:

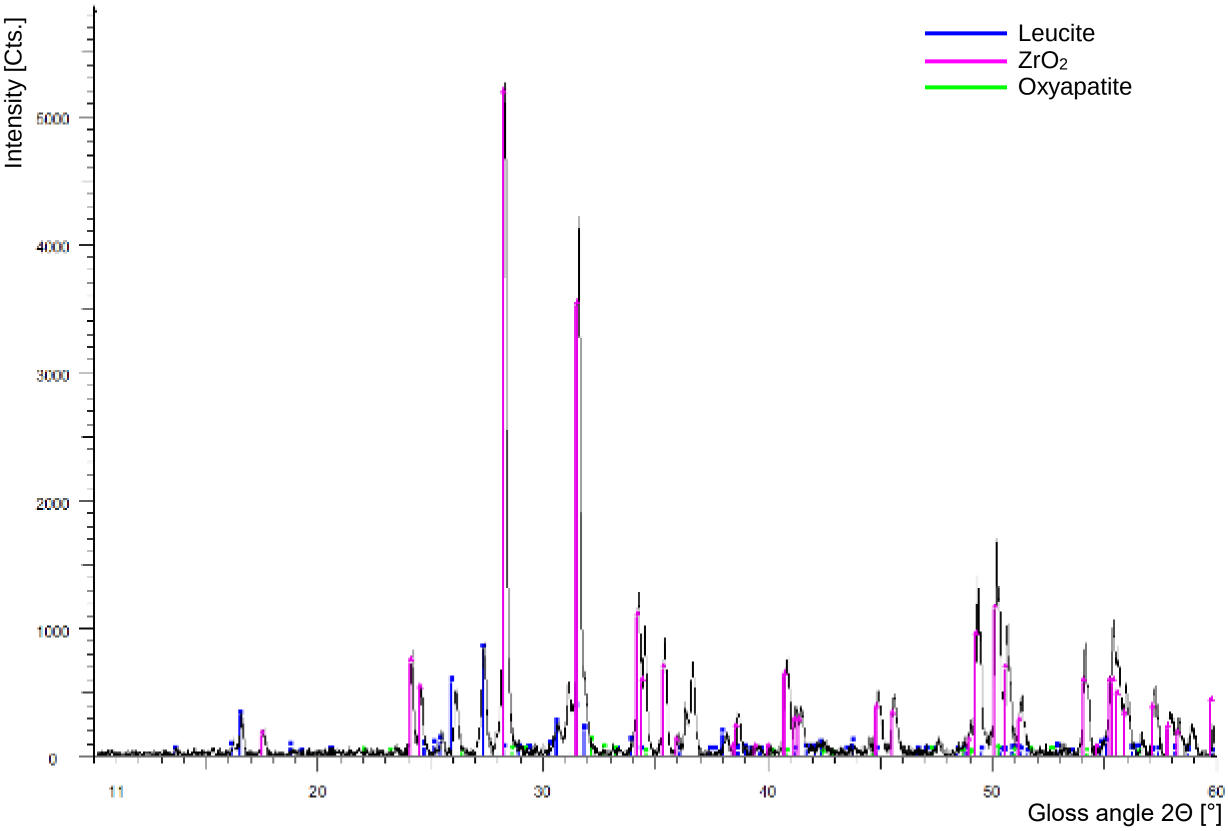


Figure 16: X-ray diffraction image of IPS Style Ceram Powder Opaquer 870 A2, with superimposed images of leucite, ZrO_2 and oxyapatite

In both the base materials of the powder opaquer and the coloured end products, the amount of crystalline zirconium oxide and/or zirconium-oxide-based pigments prevails and so these crystal phases dominate the X-ray diffraction images due to their high intensities (> 5000 Cts). The intensities produced by the crystal phases of the individual glass-ceramics (leucite, oxyapatite) are significantly lower (< 1000 Cts).

4.1.2 IPS Style Ceram Margin

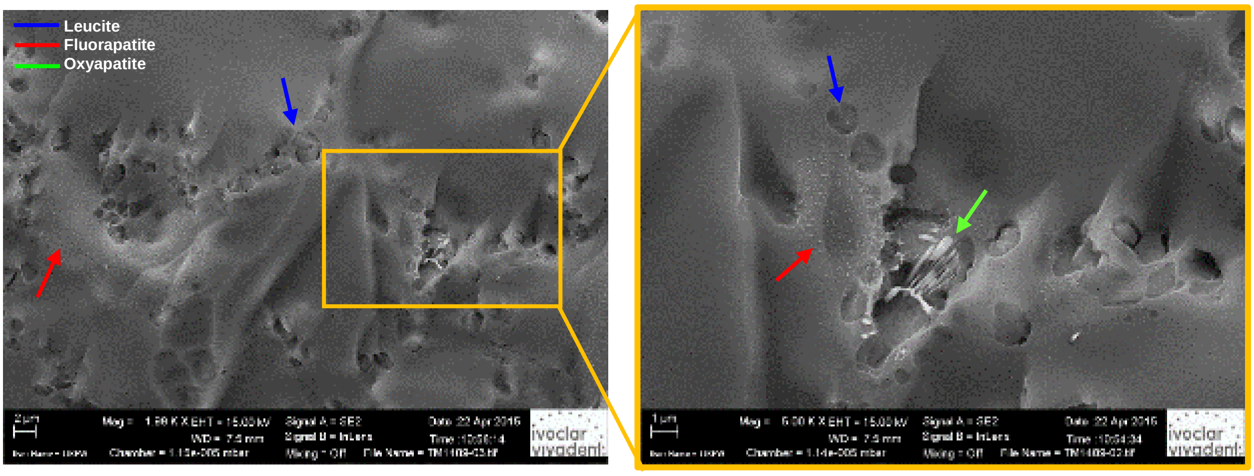


Figure 17: Typical microstructure of IPS Style Ceram Margin containing leucite, fluorapatite and oxyapatite

All three crystal phases (leucite, fluorapatite and oxyapatite) can be detected in the microstructure of IPS Style Ceram Margin. The tufts of oxyapatite needles (green arrow) in particular catch the eye. All three crystal phases are detectable on the X-ray diffraction image.

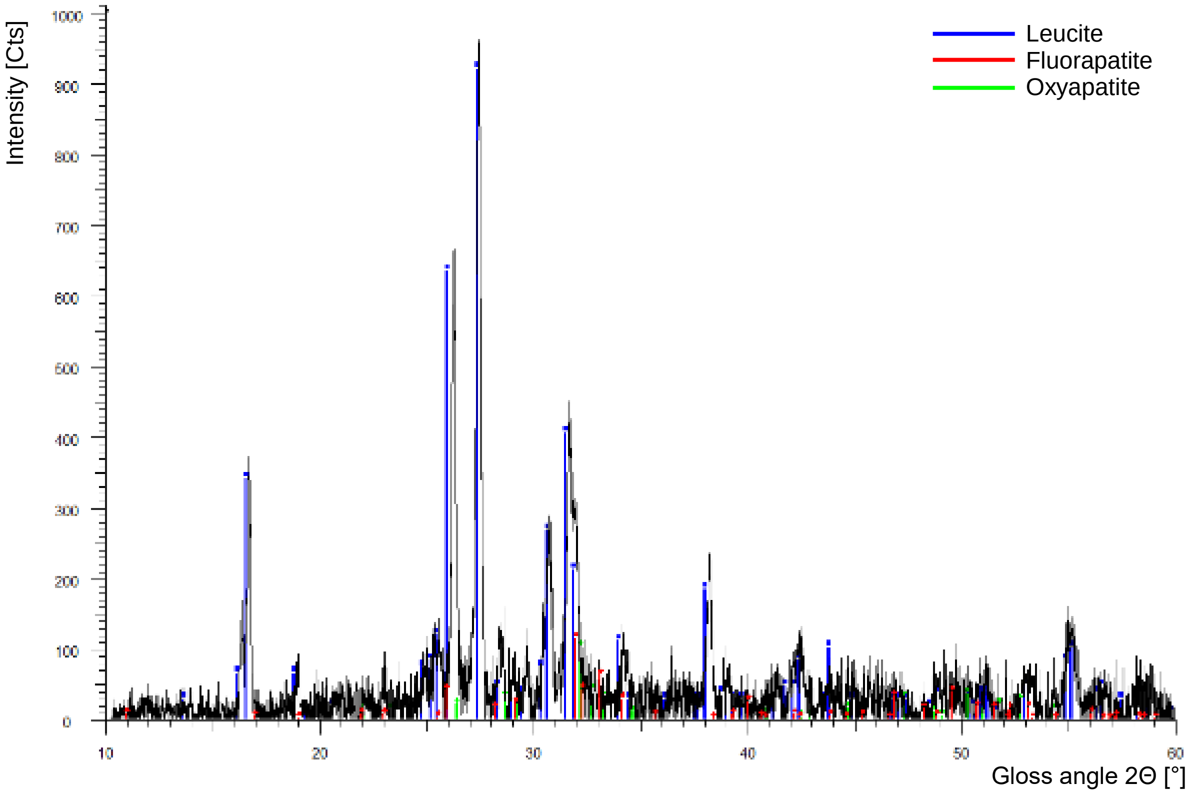


Figure 18: X-ray diffraction image of IPS Style Ceram Margin M2, with superimposed images of leucite, fluorapatite and oxyapatite

4.1.3 IPS Style Ceram Dentin

All characteristic crystal phases can be identified in the microstructure of IPS Style Ceram Dentin. Along with the needle-like oxyapatite crystals (green arrows), the smaller fluorapatite needles (pink arrows) are clearly detectable in the amorphous mother glass phase. The leucite crystals (blue arrows), which are present in different sizes and shapes, are easy to identify.

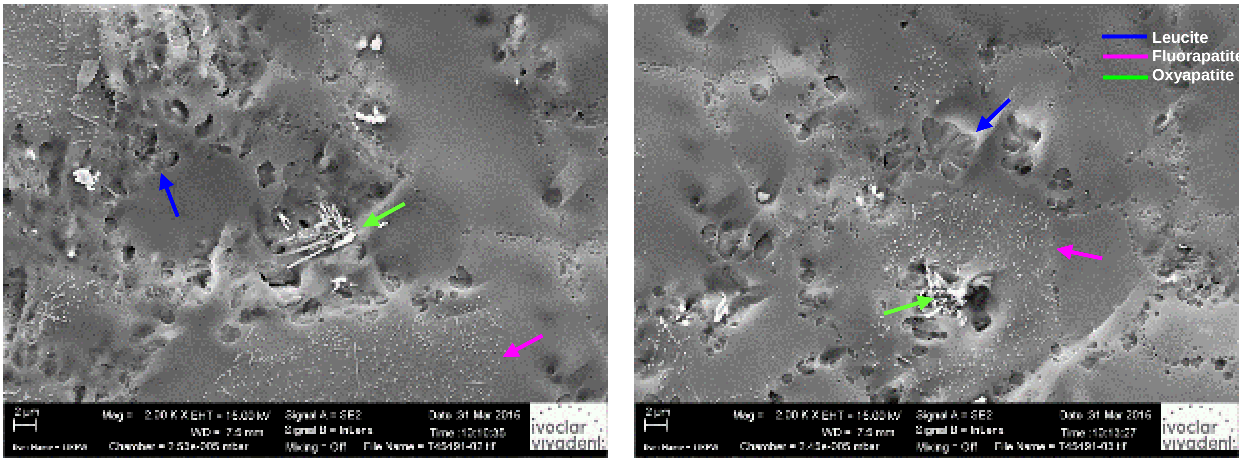


Figure 19: Typical microstructure of IPS Style Ceram Dentin A2, showing leucite, fluorapatite and oxyapatite

All the phases can be identified on the respective X-ray diffraction images.

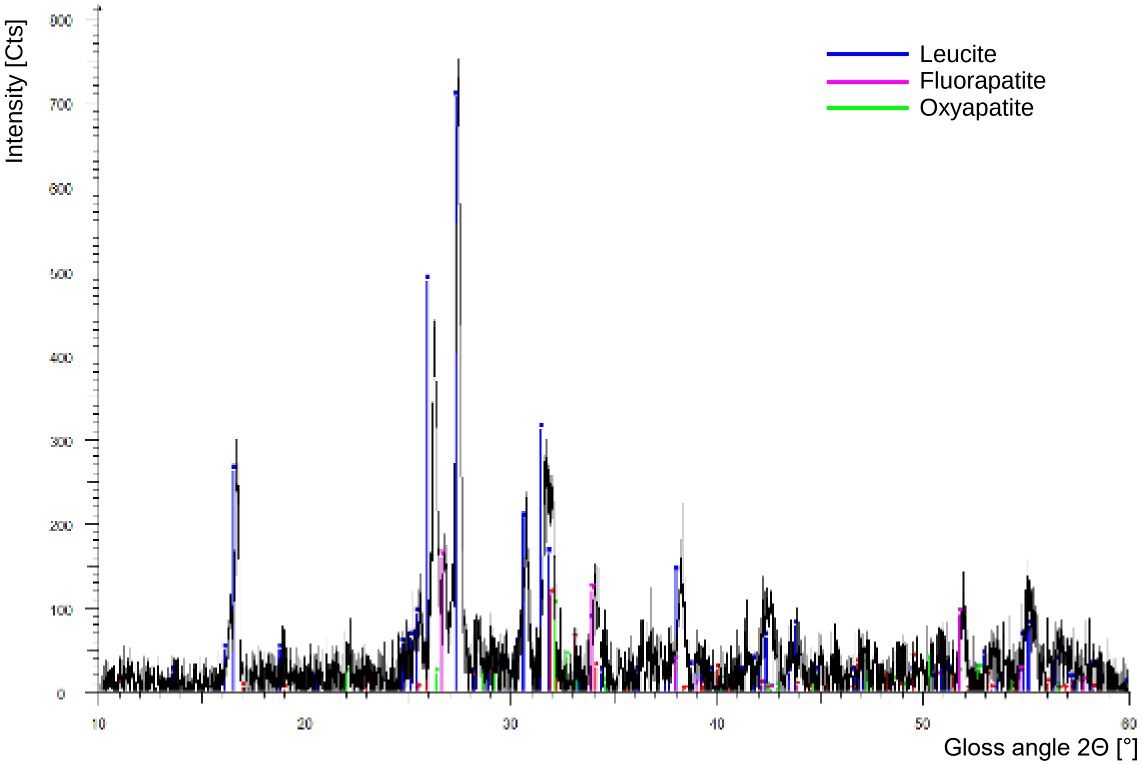


Figure 20: X-ray diffraction image of IPS Style Ceram Dentin A2, with superimposed images of leucite, fluorapatite and oxyapatite

4.1.4 IPS Style Ceram Incisal

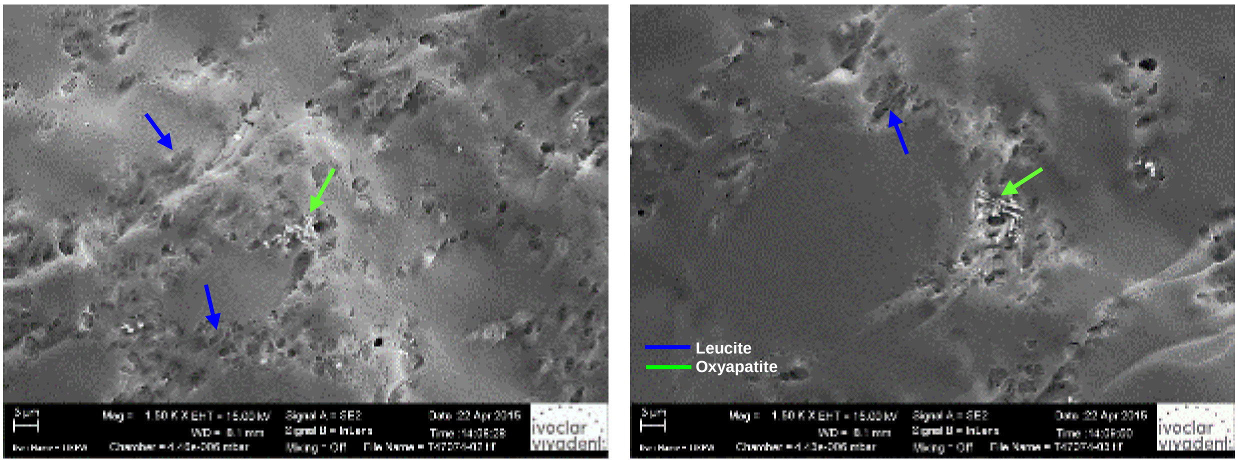


Figure 21: Typical microstructure of IPS Style Ceram Incisal I5 containing leucite and oxyapatite

The microstructure of IPS Style Ceram Incisal contains leucite and small amounts of oxyapatite, but no fluorapatite. This formulation enhances the brightness of the incisal without reducing its translucency. Accordingly, only these two crystal phases can be seen on the X-ray diffraction image (Figure 22).

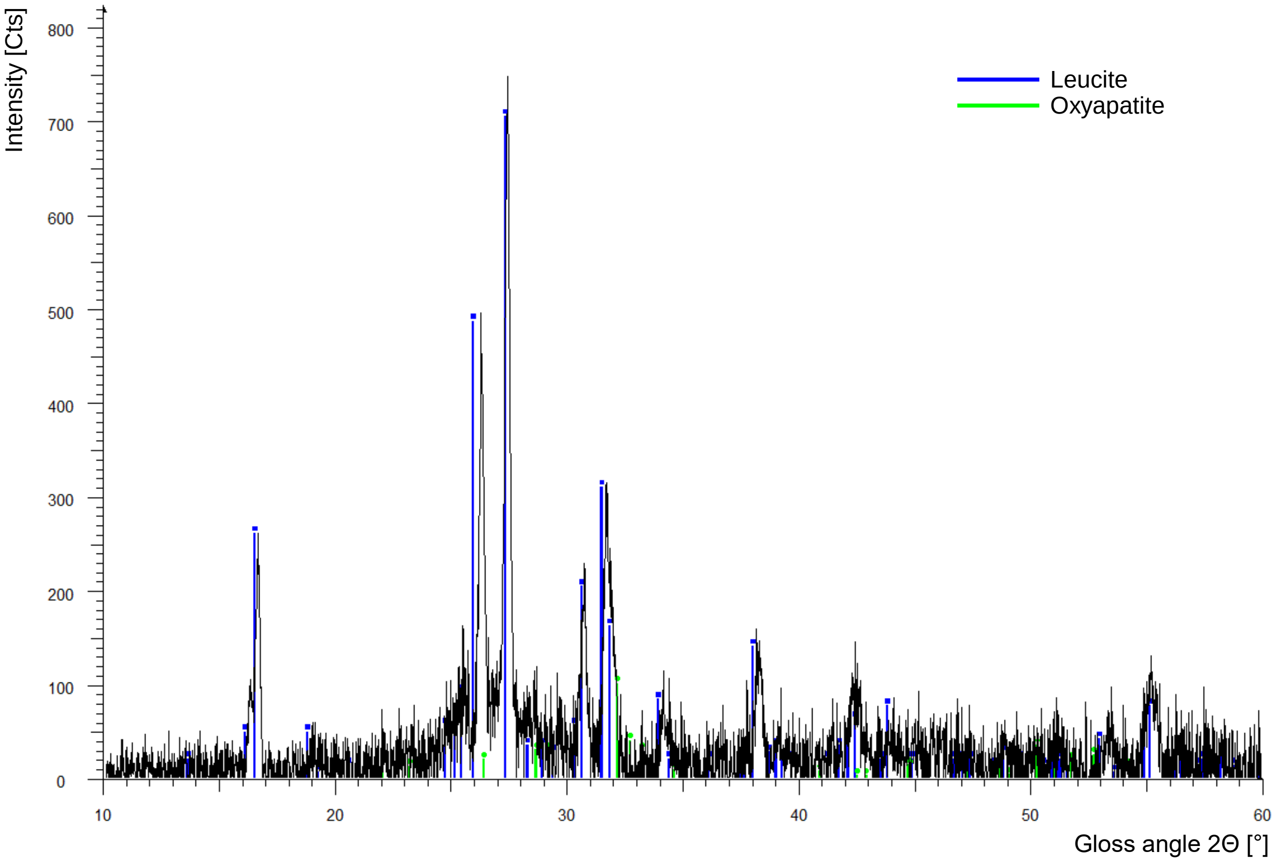


Figure 22: X-ray diffraction image of IPS Style Ceram Incisal I5, with superimposed images of leucite and oxyapatite

4.2 Properties of IPS Style according to ISO 6872:2015

IPS Style has been assessed with regard to the requirements described below and documented in the following sections:

- Biaxial flexural strength
- Fracture toughness K_{1C} using the SEVNB method
- Chemical solubility (see Section 5.3)
- Glass transition temperature
- CTE (25 - 500°C) 2x, CTE (25 - 500°C) 4x, Ø CTE (25 - 500°C) 2x/4x

4.2.1 Flexural strength and fracture toughness of IPS Style layering materials

ISO 6872:2015 stipulates a minimum of ≥ 50 MPa for the flexural strength (σ) of dental ceramics classified as Type I and Class 1. Fracture toughness is merely informative. A limiting value has therefore not been defined.

	Biaxial flexural strength [MPa]			Fracture toughness [MPam$^{1/2}$]	
Description	σ	SD	Limiting value*	K_{1C}	SD
IPS Style Ceram Powder Opaquer 870 A2	122	7	≥ 50	1.25	0.06
IPS Style Ceram Margin M2	86	10	≥ 50	0.73	0.09
IPS Style Ceram Dentin A2	107	15	≥ 50	0.80	0.08
IPS Style Ceram Opal Effect OE5	89	6	≥ 50	n. a.	n. a.
IPS Style Ceram Incisal I5	112	22	≥ 50	0.87	0.02

*) acc. to ISO 6872:2015

Table 1: Typical biaxial flexural strength and fracture toughness values of various IPS Style layering materials (SD: standard deviation, n/a: not assessed) (R&D Ivoclar Vivadent AG, Schaan, FL, 2015)

- ► The biaxial flexural strength of the IPS Style layering materials is clearly above the minimum of 50 MPa stipulated in the standard.

4.2.2 Thermal parameters of IPS Style layering materials

The linear thermal expansion of a material is determined by means of a dilatometer. The specimen is continuously heated/cooled and the linear dimensional change recorded. The resulting dimensional change can be continuous or erratic. A jump in the curve can be seen if a phase transition occurs in the material. The linear coefficient of thermal expansion (CTE) is determined per unit length for 1 degree change in temperature (1 Kelvin). The CTE largely depends on the temperature range within which it is measured. Therefore, the temperature range has to be stated at all times. ISO 6872:2015 prescribes that the CTE should be determined between 25 – 500°C (or T_G). The CTE is utilized to assess the potential stress levels that the ceramic may have to endure in conjunction with the framework and/or layering material. Glass-ceramics at temperatures above the glass transition value (T_G) are soft and the stress is dissipated by the flow of the material.

The thermal expansion of a ceramic is decisive for its compatibility with the framework material. Ceramic materials are far less sensitive to compressive stresses than to tensile stresses. Therefore, in dental restorations the ceramic has to be applied in such a way that it is subjected to compressive stress in the restoration. This is achieved by choosing the CTE of the ceramic to be about one unit ($1x10^{-6} \cdot K^{-1}$) lower than the CTE of the alloy.

The CTE of the ceramic changes with the thermal treatment (e.g. with the number of firing cycles), as the microstructure may alter depending on the temperature (grain growth, precipitation of a higher amount of leucite).

An overview of the CTEs of various IPS Style layering materials according to ISO 6872:2015 and their T_G values is given in Table 2:

Description	CTE (25-500°C) 2x [10^{-6} K^{-1}]	CTE (25-500°C) 4x [10^{-6} K^{-1}]	Ø CTE (25-500°C) 2x/4x [10^{-6} K^{-1}]	T_G [°C]
PU IPS Style Ceram Powder Opaquer 870 A2	13.5 ± 0.5	13.7 ± 0.5	13.6 ± 0.5	540 ± 20
PU IPS Style Ceram Margin M2	12.8 ± 0.5	13.1 ± 0.5	13.0 ± 0.5	580 ± 20
PU IPS Style Ceram Dentin A2	12.0 ± 0.5	12.4 ± 0.5	12.2 ± 0.5	525 ± 20
PU IPS Style Ceram Opal Effect OE5	12.0 ± 0.5	12.4 ± 0.5	12.2 ± 0.5	510 ± 20
PU IPS Style Ceram Incisal I5	12.0 ± 0.5	12.4 ± 0.5	12.2 ± 0.5	525 ± 20

Table 2: Thermal parameters of various IPS Style layering materials (R&D Ivoclar Vivadent AG, Schaan, FL, 2015)

4.3 Shade stability of IPS Style after multiple firings

Users attest to the high brightness of IPS Style, which, along with the shade concept and excellent shade stability, accounts for the esthetic effect of this veneering material.

To examine the changes in shade and opacity of IPS Style, five defined shade samples each were fabricated. The samples were fired once, three times, five times, seven times and ten times, prepared and then measured. The first firing process was carried out to sinter the samples to their final density. All the subsequent firing cycles were carried out at the technical firing temperatures. Samples of competitive materials were fabricated using the same method. The following incisal and dentin materials were examined:

Manufacturer	**Incisal material**	**Batch no.**	**Dentin material**	**Batch no.**
Ivoclar	IPS Style Ceram Incisal I2	U14689	IPS Style Ceram Dentin A2	UZ1073
Heraeus Kulzer	HeraCeram Sun S1	2012X	HeraCeram Sun D A2	2513X
GC	GC Initial LF E-58	201401221	GC Initial LF D-A2	201309121
WG Creation	Creation LF E-58	8806	Creation LF D-A2	9512
Vita	Vita Base Enamel ENL	51550	Vita Base Dentin 2M2	44930

Table 3: Overview of the materials for shade stability testing in conjunction with multiple firings

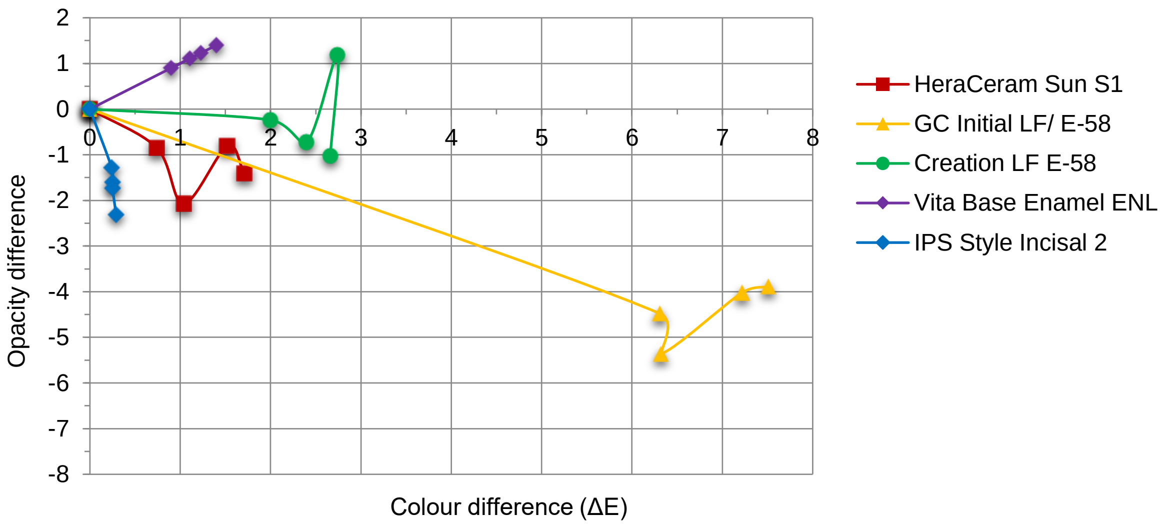


Figure 23: Comparison of the colour and opacity difference after multiple firings of IPS Style Ceram Incisal and competitive materials. The opacity difference is shown in relation to the colour difference. The dotted line marks the threshold for the perception of differences in colour by the average observer (R&D Ivoclar Vivadent AG, Schaan, FL, 2015).

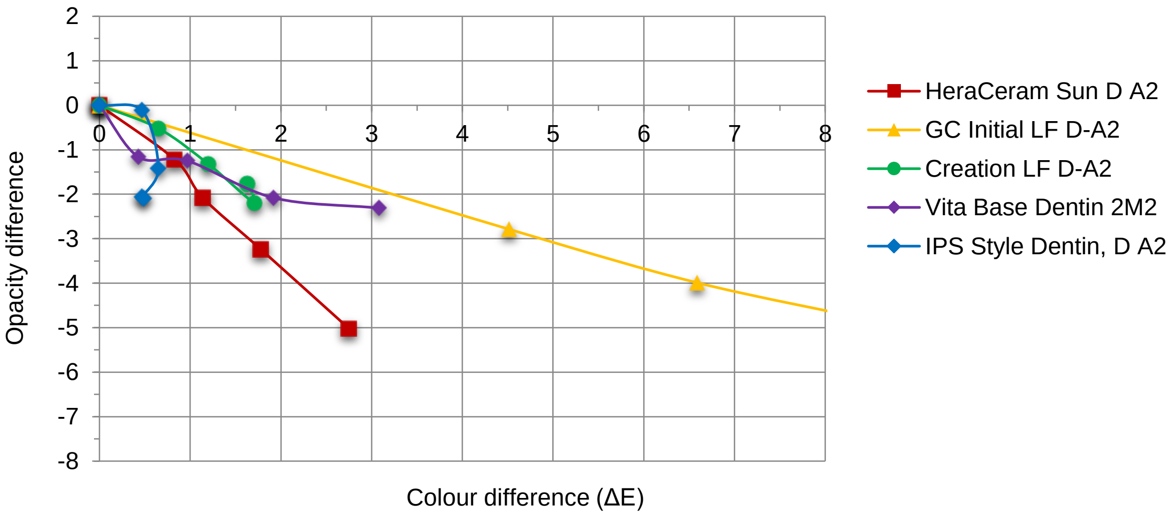


Figure 24: Comparison of the colour and opacity difference after multiple firings of IPS Style Ceram Dentin and competitive materials. The opacity difference is shown in relation to the colour difference. The dotted line marks the threshold for the perception of differences in colour by the average observer (R&D Ivoclar Vivadent AG, Schaan, FL, 2015).

Both the IPS Style Ceram Incisal and Dentin materials showed an excellent opacity and colour stability when fired several times. The colour differences (ΔE) measured for the samples are not perceptible by the average observer. The competitive materials were examined for comparative purposes and some of them showed clearly perceptible opacity and colour differences as early as after the third firing. After ten firings, the colour of some samples had changed to such an extent that a new shade was present. To enhance the comparability between the incisal and dentin materials, the same ranges were used for the axes of the two graphs (Figure 23 and Figure 24). The values measured for the GC Initial LF D-A2 dentin samples after the seventh and tenth firing fell outside of the range used in these graphs. IPS Style Ceram Dentin A2 produced nearly identical results after seven and ten firings, which on the graph looks as if one measuring point is missing.

4.4 Qualitative observation of shrinkage and wetting of IPS Style

The shrinkage and wetting behaviour of the layering material can be qualitatively assessed and compared with competitive materials with the help of metal-ceramic test specimens. The method for this test is described below. The starting point of the procedure is the fabrication of rectangular metal-ceramic green bodies of exactly identical dimensions. These bodies are used to measure the volumetric changes after sintering (ceramic firing) and this serves as a measure for the shrinkage of the individual materials. In addition, the procedure is used to examine the wetting behaviour of the sintered material on the substrate, i.e. the opaquer.

To conduct the test, metal samples measuring 14.5 x 10.7 x 1.1 mm were produced using Zenotec® NP according to the manufacturer's instructions and then coated with the relevant opaquer. Subsequently, the ceramic being examined was layered onto the metal samples. In the process, the investigated ceramic powder was processed into a slurry using the accompanying modelling fluid or deionized water and then applied in layers onto the metal plates in several stages and, if necessary, excess fluid was removed by suction. The surface of the ceramic was drawn smooth by carefully guiding an inclined razor blade back and forth over it. The thickness of the layer and the lateral boundaries were determined by the steel template (Figure 25). A ceramic thickness of 1.5 mm in the green state proved to be ideal. The front was smoothed out with a microscope slide. Finally, the ceramic layer was cut in the middle down to the opaquer using a razor blade (Figure 25 (d): completed sample, shortly before being fired).

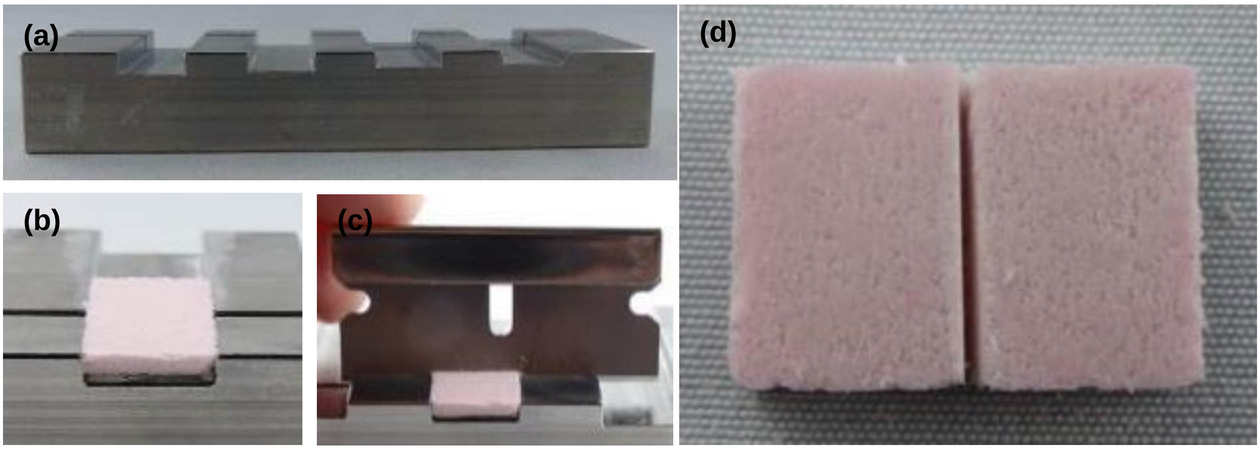
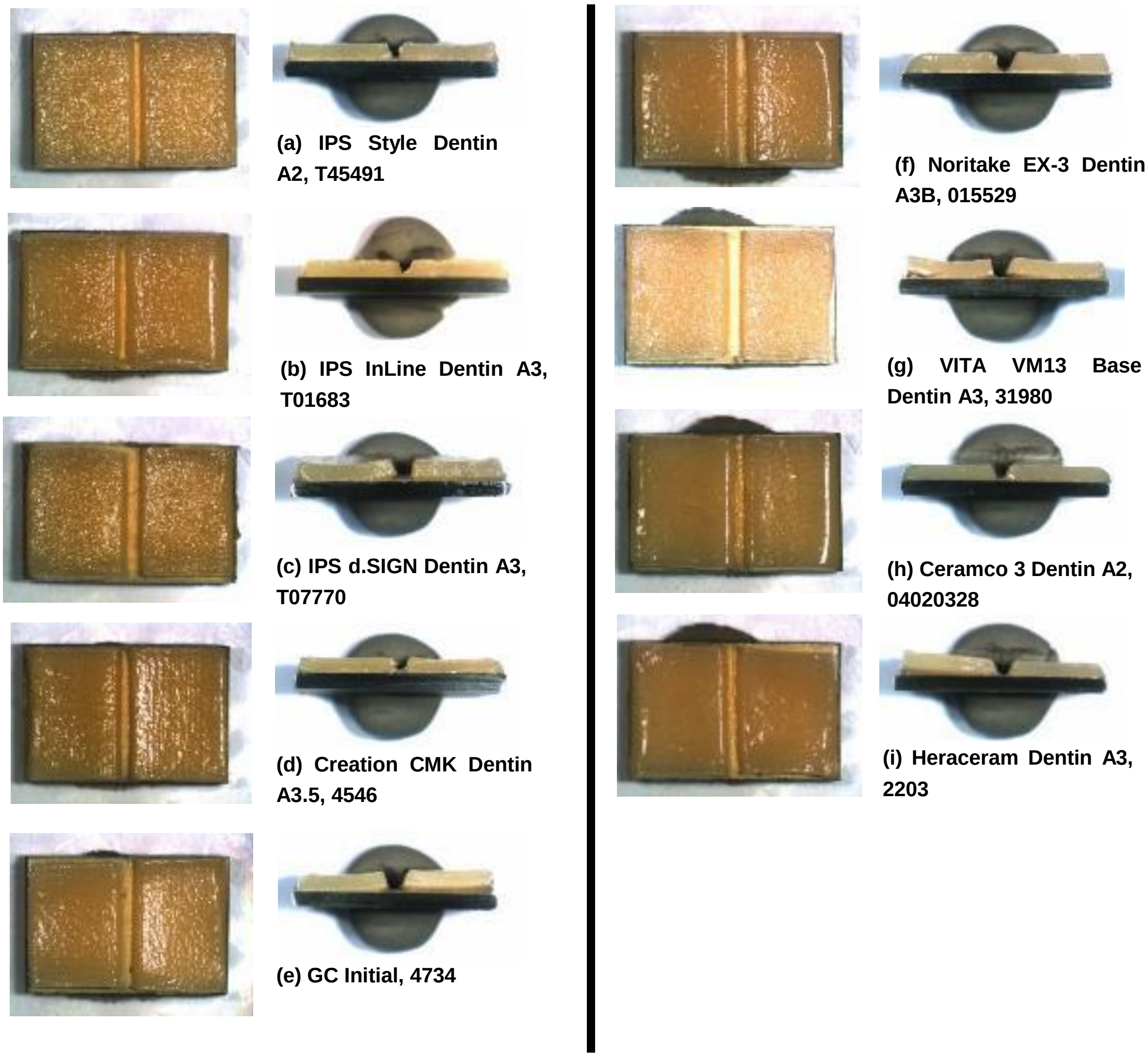


Figure 25: Fabricating the samples for testing the wetting behaviour. Template (a), different stages in the fabrication process (b)–(d) (R&D Ivoclar Vivadent AG, Schaan, FL, 2015)

After firing (according to the manufacturer's firing parameters), the plate was visually examined to assess the contact between the ceramic and opaquer.

The results of the Ivoclar Vivadent layering materials (a-c) are shown in comparison with the results of the competitive materials (d-i) in Figure 26:



(a) IPS Style Dentin A2, T45491

(b) IPS InLine Dentin A3, T01683

(c) IPS d.SIGN Dentin A3, T07770

(d) Creation CMK Dentin A3.5, 4546

(e) GC Initial, 4734

(f) Noritake EX-3 Dentin A3B, 015529

(g) VITA VM13 Base Dentin A3, 31980

(h) Ceramco 3 Dentin A2, 04020328

(i) Heraceram Dentin A3, 2203

Figure 26: Samples for testing the wetting behaviour of different dental ceramics. Only the dentin layer is shown for each ceramic system (R&D Ivoclar Vivadent AG, Schaan, FL, 2015).

The test specimens show the typical shrinkage and wetting behaviour of the metal-ceramics being investigated. The shape of the sintered powder is indicative of the shrinkage behaviour of the ceramic. Delamination at the edge is symptomatic of the unwilling wetting behaviour of some dentin materials during firing. The wetting test revealed clear qualitative differences in the firing behaviour of the different veneering materials.

These results, however, cannot be transferred, just as they are, to the sintering behaviour encountered in the fabrication of dental restorations, since different geometrical conditions come into play in restorations (concave/convex shapes instead of flat surfaces with right angles).

5 Biocompatibility

Biocompatibility (*bios* [Greek] = life) refers to "the ability of a material to perform with an appropriate biological response in a specific situation". Biocompatibility is therefore concerned with the interaction between the patient and a material and its function. Biocompatibility involves an ongoing dynamic process and is complex to assess. Biocompatibility assessments require an extensive schedule of *in vitro* and *in vivo* investigations.
In vitro investigations on biocompatibility involve tests in artificial environments, e.g. in cell culture dishes. By contrast, *in vivo* investigations are performed in the living organism in the form of clinical studies [10].

Ceramic materials are highly resistant to acid and corrosion attacks and are therefore regarded as exceptionally biocompatible. The conditions found in the oral cavity (pH and temperature changes) are not severe enough to dissolve components from dental ceramics. Nonetheless, mechanical destruction and chemical reactions (erosion) may have an effect on the constituents of the ceramic. Mechanical abrasion, however, does not affect the biocompatibility because the fragments do not remain in the mouth/body for long and the composition of the ceramic does not change if pieces break off. Chemical reactions and the associated dissolution of components would lead to problems. However, the composition of dental ceramics is biologically harmless and the amounts of dissolved material would be so small that they would not present a risk to biocompatibility. ISO 6872 prescribes the *evaluation of chemical solubility* to provide proof of the safety of ceramic materials in terms of their solubility [11].

5.1 *In vitro* biocompatibility investigations

The biocompatibility of IPS Style and IPS Ivocolor was evaluated by a series of different tests as well as by literature and database searches. The materials were examined for potential cell-damaging effects (cytotoxicity) and potential harmful effects on genetic material (genotoxicity). Sensitization, irritation and acute systemic toxicity were evaluated by literature research. The absence of subchronic toxicity was demonstrated via chemical solubility testing (Section 5.3).

5.1.1 Cytotoxicity

Cytotoxicity refers to the capability of a substance to damage cells. The XTT assay is used to determine whether or not the substance being investigated inhibits cell proliferation or causes even cell death. The resulting XTT_{50} value refers to the concentration of a substance sufficient to reduce the cell number by half.

In vitro cytotoxicity of IPS Style and IPS Ivocolor was assessed by means of an XTT assay on L929 cells according to ISO 10993-5. Sintered samples were used to obtain extracts of different concentrations and the cells were incubated with these extracts. None of the IPS Style or IPS Ivoclar samples investigated, demonstrated any cytotoxic effect, not even with the highest concentrations of the extract. As the material did not show any cytotoxicity at all, there was no basis for calculating the XTT_{50} value (1-6). An additional literature research also revealed no evidence to the contrary.

5.1.2 Genotoxicity

Genotoxicity refers to the capability of substances or external influences to damage or alter the genetic material of cells.

The mutagenicity of IPS Style and IPS Ivocolor samples was tested on five different bacterial strains. None of the samples investigated induced gene mutation by base pair changes or frameshifts in the genome of the strains used in the course of the test. Given these results, we can proceed on the assumption that IPS Style and IPS Ivocolor have no mutagenic potential (7-12).

5.1.3 Sensitization, irritation and acute systemic toxicity

Literature and databases (toxicology databases) were searched to evaluate if IPS Style and IPS Ivocolor may present any other potential risks to health. Dental ceramics have been on the market for more than 30 years and no sensitization has ever been reported so far [12-14].

As both products were found to demonstrate no cytotoxicity, we can assume that the possibility of IPS Style and IPS Ivocolor causing irritation, is negligible. Comparable reactions related to ceramics have not been reported to date. As ceramics have been around for a long time, a number of clinical studies could be conducted over long periods and the patients involved in these studies were followed up at regular intervals. The study protocols required the clinicians to document unwanted side effects. No serious side effects have been reported hitherto for Ivoclar Vivadent ceramics.

- ► In view of the information available, it can be assumed that IPS Style and IPS Ivocolor do not present a cytotoxic, mutagenic, irritating or sensitizing risk for patients or users.

5.2 Radioactivity

The ISO 6872 standard on ceramic materials requires that the radioactivity be determined. The radioactivity of dental ceramic materials has been and is the subject of controversial debate in the general public [11]. There are several reasons for this. One reason can be attributed to the fact that radioactive fluorescent substances were historically employed in dental ceramic materials in the nineteen-seventies [12]. Another reason is that some of the base materials used in ceramic materials are produced from natural rock, which has its own natural radiation. Although the radiation level caused by dental ceramics is negligible, it is absolutely necessary that the activity concentration of ceramic raw materials be of the lowest possible level (< 1.0 Bq/g ^{238}U) due to the cumulative nature of radiation [11].

Some of the disintegration products of the ^{232}Th and ^{238}U decay series are γ-emitting nuclides, which can be measured by means of γ-spectroscopy.

The activity concentration values measured for IPS Style were <0.03 Bq/g for ^{238}U and ^{232}Th (13-15).

The activity concentration values of IPS Ivocolor were ≤ 0.16 Bq/g for ^{238}U and ^{232}Th (16-18).

- ► These readings show that the activity concentration of ^{238}U is consistently clearly below the limiting value of 1 Bq/g specified by ISO Standard 6872.

(Comparison: the activity of the earth's crust is in the range of 0.03 Bq/g for ^{238}U and ^{232}Th).

5.3 Chemical solubility

The chemical solubility of a range of different IPS Style samples was measured:

Description	Chemical solubility [µg/cm^2]:
IPS Style Ceram Powder Opaquer 870 A2	23
IPS Style Ceram Margin M2	10
IPS Style Ceram Dentin A2	15
IPS Style Ceram Opal Effect OE5	6
IPS Style Ceram Incisal I5	8

Table 4: Chemical solubility of IPS Style according to ISO 6872 (R&D Ivoclar Vivadent AG, Schaan, FL, 2015)

- ► The chemical solubility of all the IPS Style samples examined was clearly below the maximum of < 100 µg/cm^2 stipulated in the ISO 6872 standard.

The chemical solubility of a range of different IPS Ivocolor samples was also measured:

Description	Chemical solubility [µg/cm^2]:
IPS Ivocolor Glaze clear	14
IPS Ivocolor Essence Copper	73

Table 5: Chemical solubility of IPS Ivocolor according to ISO 6872 (R&D Ivoclar Vivadent AG, Schaan, FL, 2015)

- ► The chemical solubility values measured for the IPS Ivocolor samples were all clearly below the maximum of < 100 µg/cm^2 stipulated by the ISO 6872 standard.

5.4 Literature on biocompatibility

(Reports on investigations commissioned by Ivoclar Vivadent AG, references not published)

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(11) Sokolowski A. Salmonella typhimurium and Escherichia coli reverse mutation assay. Harlan Report No. 1630708. 2014.
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Ivoclar Vivadent AG
Research and Development
Scientific Services
Bendererstrasse 2
FL - 9494 Schaan
Liechtenstein

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