Ceramics overview: Classification by microstructure and processing methods

Abstract

The plethora of ceramic systems available today for all types of indirect restorations can be confusing and overwhelming for the clinician. Having a better understanding of them is important. In this article, the authors use classification systems based on microstructural components of ceramics and the processing techniques to help illustrate the various properties.

Introduction

Many different types of ceramic systems have been introduced in recent years for all types of indirect restorations, from very conservative non-preparation veneers, to multi-unit posterior fixed partial dentures and everything in between. Understanding all the different nuances of materials and material processing systems is overwhelming and can be confusing. This article will cover what types of ceramics are available based on a classification of the microstructural components of the ceramic. A second, simpler classification system based on how the ceramics are processed will give the main guidelines for their use.

The term "ceramic" derives from the Greek "keramos", which means "a potter or a pottery". This word is related to a Sanskrit term meaning "burned earth", since the basic components were clays from the earth heated to form pottery. Ceramics are non-metallic, inorganic materials. Ceramics refer to numerous materials, including metal oxides, borides, carbides, nitrides and complex mixtures of these materials. The structure of these materials is crystalline, displaying a regular periodic arrangement of the component atoms, and may exhibit ionic or covalent bonding. Although ceramics can be very strong, they are also extremely brittle and...
will catastrophically fail after minor flexure. Thus, these materials are strong in compression but weak in tension.

Contrast that with metals: metals are non-brittle (display elastic behaviour) and ductile (display plastic behaviour). This is because of the nature of the interatomic bonding, which is called metallic bonds; a cloud of shared electrons that can easily move when energy is applied defines these bonds. This is what makes most metals excellent conductors.

Ceramics can be very translucent to very opaque. In general, the more glassy the microstructure (i.e. non-crystalline), the more translucent; and the more crystalline, the more opaque. Many other factors contribute to translucency, for example, particle size, particle density, refractive index and porosity to name a few.

Different types of ceramics used in dentistry

The term "ceramic" technically refers to a crystalline material. Porcelain is a mixture of glass and crystal components. A non-crystalline-containing material is simply a glass. However, dentistry typically refers to all three basic materials as dental ceramics. How ceramics are classified can be very confusing. Ceramics can be classified by their microstructure, (i.e. amount and type of crystalline phase and glass composition). They can also be classified by processing technique (powder/liquid, pressed or machined) and by their clinical application. We will give a classification based on the microstructure of ceramics, with the inclusion of how the ceramics are processed and the effect of this on durability, to help the reader better understand the ceramics available in dentistry.

Microstructural classification

At a microstructural level, we can define ceramics by the nature of their composition of glass-crystalline ratio. There can be infinite variability in the microstructures of materials but they can be broken down into four basic compositional categories with a few subgroups:

- Category 1: glass-based systems (mainly silica);
- Category 2: glass-based systems (mainly silica) with fillers, usually crystalline (typically leucite or a different high-fusing glass);
- Category 3: crystalline-based systems with glass fillers (mainly alumina); and
- Category 4: polycrystalline solids (alumina and zirconia).

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1. Category 1: Glass-based systems

Glass-based systems are made from materials that contain mainly silicon dioxide (also known as silica or quartz) and various amounts of alumina (or aluminium oxide, chemical formula Al₂O₃). Aluminosilicates found in nature that contain various amounts of potassium and sodium are known as feldspars. Feldspars are modified in various ways to create the glasses used in dentistry. Synthetic forms of aluminosilicate glasses are also manufactured for dental ceramics. We could not find any documented references that demonstrated that naturally occurring aluminosilicate glasses perform better or worse than synthetic glasses, even though there have been claims to the contrary. These materials were first used in dentistry to make porcelain denture teeth.

The mechanical properties are low flexural strength, usually in the 60–70 MPa range. Thus, they tend to be used as veneer materials for metal or ceramic substructures, as well as for veneers using either a refractory die technique or a platinum foil. The microstructure of a glass is shown in Figure 1. This is an electron micrograph of an acid-etched glass surface. The holes indicate a second glass, which was removed by the acid. The veneer restoration uses a glassy porcelain (Figs. 2a & b).

2. Category 2: Glass-based systems with crystalline second phase, porcelain

This category of materials has a very large range of glass–crystalline ratios and crystal types. So much so that we can subdivide this category into three groups. The glass composition is similar to the pure glass of category 1. The difference is that varying amounts of different types of crystals have been either added or grown in the glass matrix. The primary crystal types today are leucite, lithium disilicate and fluorapatite. Leucite is created in dental porcelain by increasing the potassium oxide (chemical formula K₂O) content of the aluminosilicate glass. Lithium disilicate crystals are created by adding lithium oxide (chemical formula Li₂O) to the aluminosilicate glass. It also acts as a flux, lowering the melting temperature of the material.

These materials have also been developed into very fine-grained machinable blocks, VITABLOCS Mark II (VITA Zahnfabrik), for use with the CEREC CAD/CAM system (Sirona Dental Systems). This material is the most clinically successful documented machinable glass for the fabrication of inlays and onlays, with all studies showing a less than 1 % per year failure rate, which compares favourably with metal–ceramic survival data.²  ³ The benefit of a pre-manufactured block is that there is no residual porosity in the finished core that could act as a weak point, which could lead to catastrophic failure.

2.1 Subcategory 2.1: Low to moderate leucite-containing feldspathic glass

Even though other categories have a feldspathic-like glass, these materials have come to be called “feldspathic porcelains” by default. Leucite may alter the coefficient of thermal expansion (CTE) of the material, as well as inhibit crack propagation, which improves the strength of the material. The amount of leucite may be adjusted in the glass, based on the type of core and the required CTE. These materials are the typical powder/liquid materials that are used to veneer core systems and are the ideal materials for porcelain veneers.

The original materials had a fairly random size and distribution of leucite crystals, with the average particle size being around several hundred microns. This random distribution and large particle size contributed to the materials’ low fracture resistance and abrasive properties relative to enamel.⁴ Never generations of materials (e.g. VITA VM 13, VITA Zahnfabrik) have been developed with much finer leucite crystals (10–20 µm) and very even particle distribution throughout the glass. These materials are less abrasive and have much higher flexural strengths.⁵ An electron micrograph of a typical feldspathic porcelain reveals a glass matrix surrounding leucite crystals (Fig. 3). The most common use of these materials is as veneer porcelains for metal–ceramic restorations (Fig. 4).
2.2 Subcategory 2.2: High leucite-containing (approximately 50%) glass, glass-ceramics

The microstructure of these materials consists of a glass matrix surrounding a second phase of individual crystals. The material starts out as a homogeneous glass. A secondary heat treatment nucleates and grows crystals that give this class of materials improved mechanical and physical properties owing to the physical presence of the crystals and generation of compressive stress around the crystals. Glass-ceramic materials may be ideally suited for use as dental restorative materials. Glass-ceramics generally have improved mechanical and physical properties, such as increased fracture resistance, improved thermal shock resistance, and resistance to erosion. Improvements in properties are dependent upon the interaction of the crystals and glass matrix, as well as on the crystal size and amount. Finer crystals generally produce stronger materials. Glass-ceramics are in widespread use for cookware, missile nose cones, and even heat shields on space vehicles. They may be opaque or translucent, depending upon the chemical composition and percentage of crystallinity. A fundamental method of improving strength and fracture resistance is to add a second phase to a glass material, causing dispersion strengthening. The crystals may act as roadblocks to crack propagation. A crack spreading from a defect must go through or around the crystal, which takes some energy away from the propagating crack and may stop its progress. Thus, the restoration may continue to function instead of being cracked in half. In addition to the roadblock effect, compressive stresses around the growing crystals may help pin cracks and further enhance fracture resistance.

The most widely used version is the original pressable ceramic, IPS Empress (Ivoclar Vivadent), but there are several other products in this category (Figs. 5, 6a & b). A number of pressable materials with properties and microstructures similar to IPS Empress are available. This include Finesse (DENTSPLY), Authentic (Jensen), PM9 (VITA) and OPC (Pentron). A machinable version, IPS Empress CAD (Ivoclar Vivadent), designed for both the CEREC (Sirona) and E4D Technologies (Planmeca) CAD/CAM systems for high-leucite ceramics, has performed well clinically when used for posterior inlays and onlays, as well as anterior veneer and crown restorations. Machinable and pressable systems have much higher fracture resistance than powder/liquid systems, and have shown excellent clinical results for posterior inlay and onlay applications, and anterior veneer and crown restorations.

2.3 Subcategory 2.3: Lithium disilicate glass-ceramic

This is a type of dental glass-ceramic originally introduced by Ivoclar Vivadent as IPS Empress II (and later in the form of IPS e.max pressable and machinable ceramics). Increasing the crystal content to about 70% and refining the crystal size achieved improvements in flexural strength. The glass matrix consists of a lithium silicate with micron-size lithium disilicate crystals in between submicron lithium orthophosphate crystals (Figs. 7, 8a & b). This creates a highly filled glass matrix. A veneer porcelain consisting of fluorapatite crystals in an aluminosilicate glass may be layered on to the core to create the final morphology and shade of the restoration. The shape and the volume of crystals increase the flexural strength to about 360 MPa, or about three times that of IPS Empress. This material can be very translucent even with the high crystalline content. This is due to the relatively low refractive index of the lithium disilicate crystals. This material is translucent enough that it can be used for full contour restorations or for the highest aesthetics and can be veneered with special porcelain. Veneer porcelain consisting of fluorapatite crystals in an aluminosilicate glass may be layered on the core to create the final morphology and shade of the restoration. Fluorapatite is a fluoride containing calcium phosphate (chemical formula \( \text{Ca}_5(\text{PO}_4)_3\text{F} \)). The fluorapatite crystals contribute to the veneering porcelain’s optical properties and CTE so that it matches the lithium disilicate pressable or machinable material. Both the veneering material and the lithium disilicate material are etchable.
I review ceramics owing to the glassy phase. Initial clinical data for single restorations with this material is excellent, especially if it is bonded.20

3. Category 3: Interpenetrating phase ceramic

VITA In-Ceram (VITA Zahnfabrik) consists of a family of all-ceramic restorative materials based on the same principle introduced in 1988. The family includes a range of strengths, translucencies and fabrication methodologies designed to cover the wide scope of all-ceramic restorations, including veneers, inlays, onlays, anterior and posterior crowns, and bridges. VITA In-Ceram SPINELL (alumina and magnesia matrix) is the most translucent, of a moderately high strength and used for anterior crowns. VITA In-Ceram ALUMINA (alumina matrix) is of high strength and moderate translucency, and is used for anterior and posterior crowns. VITA In-Ceram ZIRCONIA (alumina and zirconia matrix) has a very high strength and lower translucency, and is used primarily for three-unit posterior bridges. Additionally, these materials are supplied in a block form for producing milled restorations using a variety of machining systems.

VITA In-Ceram belongs to a class of materials known as interpenetrating phase composites.21 They consist of at least two phases that are intertwined and extend continuously from the internal to the external surface (Fig. 9). These materials possess improved mechanical and physical properties relative to the individual components owing to the geometrical and physical constraints that are placed on the path that a crack must follow to cause a fracture. A tortuous route through alternating layers of both components is required to break these materials.

Interpenetrating phase materials are generally fabricated by first creating a porous matrix, in the case of VITA In-Ceram a ceramic sponge. The pores are then filled by a second-phase material, a lanthanum aluminosilicate glass, using capillary action to draw a liquid or molten glass into all the pores to produce the dense interpenetrating material.

The system was developed as an alternative to conventional metal–ceramic restorations and has met with great clinical success.22, 23 The system utilises a sintered crystalline matrix of a high-modulus material (85% of the volume), in which there is a junction of the particles in the crystalline phase. This is very different from glasses or glass-ceramic materials, in that these ceramics consist of a glass matrix with or without a crystalline filler in which there is no junction of particles (crystals). Slip casting24 may be used to fabricate the ceramic matrix or it can be milled from a pre-sintered block.25 Flexural strength ranges from 350 MPa for VITA In-Ceram SPINELL, 450 MPa for VITA In-Ceram ALUMINA and up to 650 MPa for VITA In-Ceram ZIRCONIA. Several clinical studies support the use VITA In-Ceram ALUMINA for single units anywhere in the mouth. In those studies, VITA In-Ceram ALUMINA had the same survival rate as porcelain fused to metal up to the first molar, with a slightly higher failure rate for the second molar.26–28 VITA In-Ceram ZIRCONIA should only be used on molars owing to its very high opacity, which is not ideal for anterior aesthetics. For anterior teeth, VITA In-Ceram SPINELL is ideal, owing to its higher translucency (Figs. 10 a–c).

4. Category 4: Polycrystalline solids

Solid sintered monophase ceramics are materials formed by directly sintering crystals together without any intervening matrix to form a dense, air-free, glass-free polycrystalline structure. There are several different processing techniques that allow the fabrication of solid sintered alumina or zirconia frameworks. The first fully dense polycrystalline material for dental applications was Procera AllCeram alumina (Nobel Biocare) with a strength of about 600 MPa.29 The alumina powder is pressed and milled on a die, and sintered at about 1,600 °C, leading to a dense coping but with about 20% shrinkage (Figs. 11, 12a & b).

The use of what is commonly referred to as zirconia in dentistry has increased rapidly over the past few years. This is not pure zirconia; it is partially stabilised by the addition of small
amounts of other metal oxides. Partially stabilised zirconia is one of the materials that allow production of reliable multi-unit all-ceramic restorations for high-stress areas, such as the posterior region of the mouth. Zirconia (or zirconium dioxide, chemical formula ZrO₂) may exist in several crystal types (phases), depending upon the addition of minor components, such as calcia (or calcium oxide, chemical formula CaO), magnesia (or magnesium oxide, chemical formula MgO), yttria (or yttrium oxide, chemical formula Y₂O₃), and ceria (or cerium(iv) oxide, chemical formula CeO₂). Specific phases are said to be stabilised at room temperature by the minor components. Typically for dental applications, about 3 wt% of yttria is added to the pure zirconia (Figs. 13, 14a & b).

Zirconia has unique physical characteristics that make it twice as strong and tough as alumina-based ceramics. Values for flexural strength for this material range from about 900 to 1,100 MPa. It is important to note that there is no direct correlation between flexural strength (modulus of rupture) and clinical performance. Another important physical property is fracture toughness, which has been reported to lie between 8 and 10 MPa m¹/² for zirconia. This is significantly higher than any previous dental ceramic. Fracture toughness is a measure of a material’s ability to resist crack propagation. Zirconia has the apparent physical properties to be used for multi-unit anterior and posterior fixed partial dentures. Clinical reports on zirconia have not demonstrated problems with zirconia frameworks. The problems have been associated with chipping and cracking of porcelain. Using a slow-cooling protocol at the glaze bake to equalise the heat dissipation from the zirconia and porcelain increased the fracture resistance of the porcelain by 20%.

Within categories 2 and 3, there can be great variation of composition and there are several commercial materials in these groups. Glass-based systems (categories 1 and 2) are etchable and thus easily bondable. Crystalline-based systems (categories 3 and 4) are not etchable and thus much more difficult to bond. Categories 1–3 can exist in a powdered form that is then fabricated using a wet brush technique, or they can be preprocessed into a block form that can be pressed or machined. As a rule, powder/liquid systems have much lower strength than pre-manufactured blocks, owing to a much larger amount of bubbles and flaws in the finished restoration.

Classification based on processing technique

A more user-friendly and simplistic way to classify the ceramics used in dentistry is by how they are processed. It is important to note that all materials can be processed by various techniques but in general for dentistry they can be classified as:

- powder/liquid glass-based systems;
- machinable or pressable blocks of glass-based systems; and
- CAD/CAM or slurry, die-processed, mostly crystalline (alumina or zirconia) systems.

It is an important classification method, as there appears to be a greater correlation with clinical success (and thus failure) due to processing technique. Even though a material may have the same chemistry and microstructure, the processing methodology used to produce a restoration may improve or decrease the final properties and clinical success. Specifically, machined blocks of materials have performed better than powder/liquid versions of the same material.
1. Powder/liquid

1.1 Conventional

These are typically veneer materials, which may be all glass or a mixture of glass and crystal components. These include veneers for all-ceramic and metal frameworks, and may also be used alone as anterior veneer restorations. Typically, these materials are mixed by hand with deionised water or a special modelling liquid supplied by the manufacturer. They are built up by hand and vibrated (condensed) to remove water and air. These are fired in a vacuum to help remove remaining air and improve the density and aesthetics of the veneer. Since these restorations are made by hand, there are often voids present in the fired material. This is inherent to the process and may be worse or better depending upon environmental conditions, the skill of the technician, and the firing cycle. Often, one sees bubbles remaining in the hand-layered veneer material.

1.2 Slip casting

The original VITA In-Ceram and some partially stabilised zirconia blocks are fabricated based on slip casting of alumina or zirconia. The slip is a homogenous dispersion of ceramic powder in water. The pH of the water is often adjusted to create a charge on the ceramic particles and the ceramic powder is coated with a polymer to cause the particles to be evenly suspended in the water. In the case of VITA In-Ceram, the slip is painted on a gypsum die with a brush to form the underlying core for the ceramic tooth. The water is removed via capillary action of the porous gypsum, which packs the particles into a rigid network (Fig. 15). The alumina core is then slightly sintered (0.2% shrinkage) in a furnace to create an interconnected porous network. The lanthanum glass powder is placed on the core, and the glass becomes molten and flows into the pores by capillary action to produce the interpenetrating network. The last step in the fabrication of the restoration involves application of aluminous porcelain to the core to produce the final form of the restoration. Other powder dispersions, such as those created with zirconia, may be poured into a gypsum mould that withdraws the water and leads to a homogeneous block of zirconia being formed.

2. Pressable

Pressed ceramic restorations are fabricated using a method similar to injection moulding. Monochromatic porcelain or glass-ceramic ingots are heated to allow the material to flow under pressure into a mould formed using a conventional lost-wax technique. The restoration may be cast to its final contours and subsequently stained and glazed to provide an aesthetic match. Alternatively, a coping may be moulded upon which porcelain is added to achieve the final shape and shade of the restoration. IPS Empress restorations and other materials with a similar leucite/glass structure are fabricated in this manner. The glass-ceramic IPS e.max is also fabricated this way. Pressables may be used for inlays, onlays, veneers and single-unit crowns.

3. CAD/CAM

3.1 Subtractive (removal of excess material to fabricate the restoration, milling)

3.1.1 Full contour

Full contour restorations, such as inlays, onlays, crowns and veneers, may be fabricated from various blocks of materials. In general, these blocks are fabricated from starting powders that are mixed with a binder and then pressed into a mould or extruded like a sausage into a block form. The binder helps hold the powder together so that the shape is maintained after pressing or extrusion. The blocks are then transferred to a furnace to remove the binder and sintered to full density. As mentioned
previously, restorations milled from blocks tend to have improved density and mechanical properties compared with powder/liquid or pressed restorations owing to the standardised manufacturing process [Fig. 16].

3.1.2 Glass/crystal

VITABLOCS Mark II are fabricated using fine-grained powders, which produce a nearly pore-free ceramic with fine crystals. This was the first material specifically produced for the CEREC system and has an excellent history of clinical success for inlays, onlays, and anterior and posterior crowns. The restoration may be characterised with external stains or porcelain may be added to produce a layered effect (Figs. 17a & b). These blocks are available as monochromatic, polychromatic with stacked shades as in a layer cake, and more recently in a form replicating the hand-fabricated crowns for which an enamel porcelain is layered on dentine porcelain.

3.1.3 Glass/leucite

IPS Empress CAD is based on the pressable IPS Empress and has the same microstructure, a feldspathic glass with about 45 % leucite crystal. These blocks also have a fine leucite crystal structure (about 5–10 µ) and may be further characterised using external stains or porcelain. IPS Empress CAD is available in monochromatic and polychromatic stacked shades. Its strength properties are similar to that of VITABLOCS Mark II. Common to all of these blocks is a microstructure with a fine particle size that helps resist machining damage, improve mechanical properties and decrease the polishing time of the finished restoration.

3.1.4 Lithium disilicate

The IPS e.max block (lithium disilicate) is not initially fully crystallised. This improves milling time and decreases chipping from milling. The milled restoration is then heat-treated for about 20–30 minutes to crystallise the glass, and produce the final shade and mechanical properties of the restoration. The crystallisation process changes the restoration from a blue colour to a tooth shade. The microstructure and chemical composition are essentially the same as those of IPS e.max Press. The IPS e.max block has several translucency levels, the least translucent used primarily as a framework material and the higher translucency blocks used for full contour restorations.

3.1.5 Framework

(a) Alumina: Interpenetrating phase or glass-infused

VITA In-Ceram blocks are fabricated by pressing the alumina-based powder into a block shape in a manner similar to that of VITABLOCS Mark II. However, these blocks are only fired to about 75 % dense. Porous blocks of VITA In-Ceram materials are milled to produce a framework. The blocks are then infused with a glass in different shades to produce a 100 % dense material, which is then veneered with porcelain. Glass infusion only requires about 20 minutes for a coping and 1.5 hours for a three-unit bridge. The microstructure is the same as that of slip-cast alumina. The blocks are available in all three types of VITA In-Ceram.

(b) Alumina: Porous

Alumina frameworks may be fabricated from porous blocks of material. Pressing the alumina powder with a binder into moulds produces the blocks. The blocks may be partially sintered to improve resistance to maching damage or used as pressed in a fully green state (unfired, with binder). The frameworks are milled from the blocks and then sintered to full density at about 1,500 °C for 4–6 hours. The alumina has a fine particle size of about 1 µ and a strength of about 600 MPa, and is designed for anterior and posterior single units, as well as anterior three-unit bridges.
Zirconia frameworks milled from porous blocks are fabricated in a similar fashion to those milled from alumina blocks. There are a variety of methods to press the powder into a mould. Uniaxial pressing involves pressing from one direction, biaxial pressing involves pressing from two equal and opposite directions, and isostatic pressing involves uniform pressing in all directions. There are advantages and disadvantages to all methods but the desired result is the same: to produce a homogeneous block that shrinks uniformly. As is the case with the alumina block, the milled zirconia framework shrinks about 25% after a 4–6 hour cycle at around 1,300–1,500 °C. The particle size is about 0.1–0.5 µ.

(d) Partially stabilised zirconia: Hot isostatic pressing

Fully dense zirconia is produced by a method called hot isostatic pressing. The zirconia powder may be pre-pressed into a block or the powder itself may be packed into a flexible mould. Either the block or mould is then vacuum sealed in an airtight rubber or plastic bag and placed into a fluid-filled chamber. Pressure is then applied to the fluid and this pressure is transmitted evenly all around the zirconia. Heat is applied to the chamber, which sinters the zirconia to full density (Fig. 18). Zirconia blocks produced in this manner may achieve flexural strength values of about 1,200–1,400 MPa. However, it requires extended milling to produce the framework and the higher strength value does not generally justify the loss in productivity. The accuracy may be improved versus the porous block method and may be preferred for large frameworks that span the arch.

3.2 Additive

3.2.1 Electrodeposition

VITA In-Ceram powder dispersions used in the slip-casting technique have been applied to electrodeposition systems, which apply a current across the dispersion and deposit the powder particles automatically on the surface of a conductive die. This approach is efficient for single units, but becomes cumbersome and potentially unreliable for multi-unit frameworks.

Discussion and summary

Ceramics can be classified in many ways. Two classification systems were given to aid the reader in understanding the types of ceramics available for dental use. The processing technique has a very large impact on strength and thus clinical performance, and should be one of the primary considerations in choosing a material.

There are many clinical aspects that are important for success with all-ceramic materials that are not as critical with metal-based restorations that cannot be covered here (e.g. preparation design, management of stresses, and cementation techniques). The reader is advised that significant knowledge and training in these areas are requisite for success with all-ceramic materials.

About the authors

Prof. Edward A. McLaren, DDS, MDC, is the founder and director of UCLA postgraduate aesthetic dentistry, and Director of the UCLA Center for Esthetic Dentistry in Los Angeles in California in the US.

Prof. Russell Giordano, DMD, CAGS, DMSc, is an associate professor in Materials Science and Engineering at the Boston University College of Engineering in Massachusetts in the US. He received his DMD from the Harvard School of Dental Medicine in the US. Presently, he has several private and federally funded projects. His major research foci are ceramics and ceramic matrix composites.