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A review of engineered zirconia surfaces in biomedical applications

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Abstract

Zirconia is widely used for load-bearing functional structures in medicine and dentistry. The quality of engineered zirconia surfaces determines not only the fracture and fatigue behaviour but also the low temperature degradation (ageing sensitivity), bacterial colonization and bonding strength of zirconia devices. This paper reviews the current manufacturing techniques for fabrication of zirconia surfaces in biomedical applications, particularly, in tooth and joint replacements, and influences of the zirconia surface quality on their functional behaviours. It discusses emerging manufacturing techniques and challenges for fabrication of zirconia surfaces in biomedical applications.

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Keywords: Dental restotations; joint repalcement; manufacuture; surfece; zirconia

1. Introduction

Load-bearing surfaces are widely used in medicine and dentistry as medical and dental prostheses, such as joints and crowns/bridges, which are increasingly needed by millions of people in our aging societies. A wide spectrum of materials is used for these functional surfaces, including metals, ceramics, polymers and composites. Among these materials, zirconia combines high strength and fracture toughness, outstanding slow crack growth resistance, low thermal conductivity, high ionic conductivity, attractive biocompatibility and chemical inertness [1–5]. The tetragonal-to-monoclinic phase transformation in zirconia [6] results in a high damage tolerance and enhanced fracture toughness [2]. This transformation toughening mechanism makes zirconia the strongest and most fracture resistant material of all bio-structural ceramics [7–10].

Zirconia has long been used in engineering as ferrules in optic fiber connectors [11–13], environmental filters, and mechanical components [14,15]. Zirconia has been used in surgical implants such as femoral heads for total hip replacements for nearly a half century [16]. Over the last thirty years, zirconia has also been applied in restorative dentistry as dental implants and abutments [17]. Over the last twenty years, zirconia has been used as cores for bi-layered posteriors as dental crowns and bridges [2,4,9,18,19]. Recently, coloured zirconia with improved translucency has been developed to closely match colours of human teeth. This new material has a flexural strength of 900–1400 MPa and a fracture toughness of up to 6 MPa $m^{1/2}$ [20]. Such advantages have led to an exponential increase in the use of zirconia for monolithic crowns and bridges for posterior applications [21].

In the application of zirconia in medical and dental devices, the material must be machined and surface-treated to obtain not only mechanical functions such as wear and fatigue resistances, but also biomedical capabilities such as cell adhesion, bacterial decolonization and bonding strength. This paper reviews current manufacturing techniques for fabrication of zirconia surfaces in biomedical applications, particularly, in tooth and joint replacements, and influences of

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the zirconia surface quality on their functional behaviours. It discusses emerging manufacturing techniques and challenges for fabrication of zirconia materials.

2. Zirconia Materials

2.1. Zirconia microstructures

Most structural zirconia materials are various zirconia containing ceramic alloys, in which zirconia is doped with other oxides, such as magnesium oxide (MgO), yttrium oxide (Y_2O_3), calcium oxide (CaO), and cerium oxide (Ce_2O_3), to form stabilized tetragonal or cubic phases [8]. In medical and dental applications, yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP) is most popular.

Pre-sintered Y-TZP is porous and has low strength. An example of pre-sintered Y-TZP is IPS e.max Zir CAD (Ivoclar Vivadent), which is designed for dental crowns and bridges using chairside dental CAD/CAM systems. The material is 97% tetragonal and 3% monoclinic zirconia, containing approximately 87–95 wt. % ZrO_2, 4–6 wt. % Y_2O_3 as a stabilizer for retention of tetragonal grains to room temperature, 1–5 wt. % HfO_2 as binders, and 0.1–1 wt. % Al_2O_3 as sintering aids to facilitate the densification of zirconia [22,23]. It has a highly isolated or interconnected porous microstructure with a porosity of approximately 47.3–49.3 vol. % [2] and Y-TZP crystals of approximately 300-nm grain size [24].

Sintered Y-TZP is obtained at temperatures between 900 °C and 1600 °C depending on required microstructures. In general, coarse zirconia microstructures are produced at higher temperatures and longer dwell times [2]. For dental prostheses, sintering is often conducted at 1200 °C–1600 °C [2], resulting in highly compacted zirconia grains of 300 nm or less [2].

2.2. Mechanical properties

The mechanical properties of zirconia are determined by their microstructures and measurement scales [25, 26]. From a machining point of view, the indentation behaviour of zirconia at the micro/nano scales is essential to its manufacturability, because the micro/nano indentation properties are directly associated with material responses to diamond or tungsten carbide abrasive machining processes [24,27–29]. Several studies have focused not only on the indentation hardness and modulus but also on the resistance to plasticity and the resistance to machining-induced cracking based on the Sakai–Nowak model [30]. The resistance to plasticity indicates an independent property from the indenter geometry and represents the plasticity of a material. The resistance to machining-induced cracking is defined as the inverse degree of damage for a unit applied work [30]. These properties can be used to predict the machining behaviour of zirconia materials. Table 1 shows the comparison of the mechanical properties of pre-sintered (IPS emax ZirCAD) and sintered Y-TZP at 1200°C with a holding time of 2 hours [24,27–29]. Sintering decreased the porosity from approximately 47.3–49.3 vol. % to less than 0.5 vol. %. Sintered zirconia is more than 10 times harder and stronger, and approximately 5 times tougher than the pre-sintered state. The much higher resistance to machining-induced cracking for sintered Y-TZP indicates its higher degree of damage tolerance but it is less deformable than pre-sintered Y-TZP [24,27–29].

Table 1. Properties of pre-sintered and sintered Y-TZP materials [24,27–29]

Property	Pre-sintered	Sintered at 1200°C
Porosity (vol. %)	47.3–49.3	< 0.5%
Density (g/cm^3)	3.0–3.21	6.09
Nanohardness (GPa)	1.11±0.34	13.15
Young's modulus (GPa)	29.34±4.93	168.19
Fracture toughness (MPa m$^{1/2}$)	0.8	5.5
Flexural strength (MPa)	50–90	900
Resistance to plasticity (GPa)	3.28±0.98	43.22±9.59
Resistance to machining-induced cracking (J/m^2)	128.90±24.1	400

3. Manufacturing of Zirconia

The selection of white (or soft in dentistry) and hard machining processes for zirconia is based on the microstructure and mechanical properties of the material. Both processes have advantages and disadvantages.

3.1. White machining of pre-sintered zirconia

White machining is used to machine pre-sintered zirconia to obtain complex profiles such as dental crowns and bridges. It is a dry milling process using tungsten carbide milling tools [4,31]. This process enables a rapid and cost-effective generation of complex profiles of zirconia components. However, white machining also produces extensive surface damage on machined surfaces. Fig. 1 shows a scanning electron micrograph of surface fracture, crack and micro-chips produced in pre-sintered Y-TZP during a CNC white machining using a tungsten carbide milling tool [31]. Intragranular and transgranular fractures easily occurred due to weakly interconnected porous structures in pre-sintered state. This machining-induced damage cannot be naturally

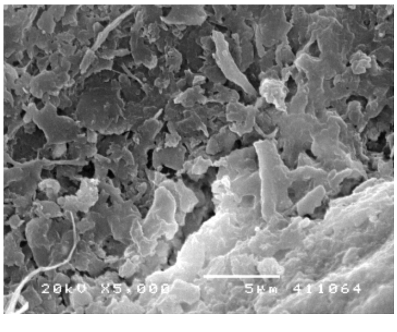

Fig. 1. Surface damage in pre-sintered Y-TZP produced in CNC milling process using a tungsten carbide milling tool [31].

removed during the subsequent sintering process, and thus can cause stress concentrations under mechanical loads.

All white machined zirconia components must be sintered at high temperatures to obtain the proper mechanical properties. Sintering unavoidably induces approximately 25% shrinkage and phase transformations, which cause severe changes in volumes, shapes and dimensions of the components [1,4,19]. Thus, precision design of initial pre-sintered components is required to compensate the sintering-induced shrinkage, and volume and shape changes, which can be very challenging. Nevertheless, white machining has become more popular in restorative dentistry and is utilised in many dental CAD/CAM systems [1,4,19] due to its cost effectiveness in which less stiff milling machines and cheaper carbide tools can be used.

3.2. Hard machining of sintered zirconia

Hard machining is performed on sintered zirconia to obtain high precision in tolerance, dimension, and shape using high-precision, high-stiffness, and conventional or high-speed grinding machines [14]. Diamond or CBN, or dense vitreous bond silicon carbide grinding wheels are used [14,15,32]. The process is much more expensive than white machining. Fig. 2 demonstrates the morphology of a sintered-Y-TZP surface produced in diamond grinding [33]. In comparison with the pre-sintered surface produced in white machining shown in Fig. 1, there are far fewer fracture defects on the ground sintered zirconia surface, because sintered zirconia has much higher fracture toughness and resistances to machining-induced damage evidenced in Table 1. Hard machining of zirconia is generally conducted in the quasi-plastic removal mode at both conventional and high-speed grinding conditions because sintered zirconia is more resistant to machining damage compared to many other polycrystalline ceramics [14,33].

In grinding of sintered zirconia, machining-induced micro damage can be diffused by shear stresses [14,33]. This is also proved in diamond indenting of sintered zirconia, in which plastic deformation and dislocation-induced pileups were observed around indented imprints [29] using atomic force microscopy. In contrast, microstructural compaction (pore closure and opening) and kink band formation were examined around the indented imprints of pre-sintered zirconia [29]. These nanomechanical behaviours of pre-sintered and sintered zirconia materials predict the different machining responses of these two materials states. Sintered zirconia can be partially plastically removed while pre-sintered zirconia undergoes brittle fracture due to breakdowns of pore networks during pore closure and opening and kink band formation in machining. In addition, the grinding-induced tetragonal-monoclinic phase transformation in sintered zirconia might have resulted in volume dilatation with compressive stresses, making crack propagation more difficult and surface flaws less detrimental to strength [7]. This phase transformation might have also occurred in machining of pre-sintered zirconia but would not have resulted in volume increase because of high porosities in pre-sintered structures.

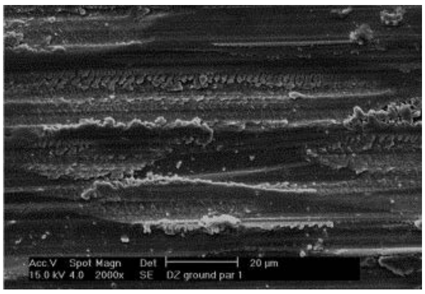

Fig. 2. Scratches and plastic deformation in sintered Y-TZP produced in diamond grinding [33].

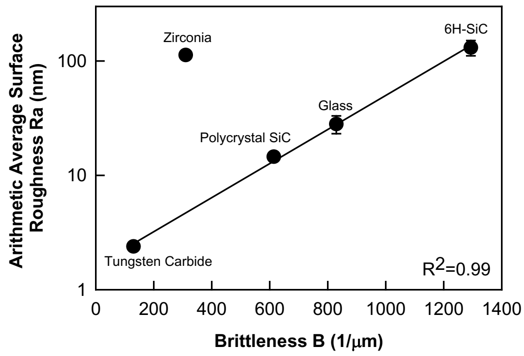

Fig. 3. Common logarithm of surface roughness Ra as function of material brittleness B for ceramics in nanogrinding, $R^2 = 0.99$, $\log_{10}(\mathrm{Ra}) = 0.209 + 0.0015B$, with the exception of zirconia [34].

Low-damage zirconia surfaces can be achieved in hard grinding. However, the surface roughness obtained is not far superior to other ceramic surfaces with more machining-induced micro cracks [34]. Fig. 3 shows that in nanogrinding of several ceramics with single crystals, polycrystals or amorphous microstructures, the dependence of the surface roughness *Ra*, on the brittleness *B* can be described by a power law. The brittleness B $(= HE/K_{IC}^2)$ is the deformation and fracture energy ratio [35], associated with the hardness H, the Young's modulus E, and the fracture toughness K_{IC}. Zirconia is an exception, which reflects the complex nature of its machinability. Although hard machining avoids subsequent sintering, the process costs are very high for components with complex shapes, such as bridges and crowns. Further, the martensitic tetragonal to monoclinic transformation during machining can create surface defects serving as stress concentration sites leading to catastrophic failure [7].

3.3. Polishing, sandblasting and surface modification

All zirconia surfaces for medical and dental applications require polishing processes to remove plastically deformed machining scratches, traces, and partially fractured scars. Different medical devices require different surface roughness values. In joint replacements, the ISO13356 approved in 1997 established 30 nm *Ra* for zirconia joint surfaces [36], which was later found inadequate to avoid excessive wear [36]. Nowadays, femoral heads and acetabular cups are generally manufactured with the surface roughness of 2–6 nm *Ra* to reduce wear particle release in the body [36]. In dentistry, clinically accepted surfaces of zirconia monolithic crowns are required to have the roughness threshold of 200 nm *Ra* to reduce bacterial plaque retention [37]. The average surface roughness *Ra*, for zirconia implants range from 130 nm to 360 nm [38].

Polishing processes influence the low temperature degradation (ageing sensitivity) in zirconia, compromising its fracture toughness and strength after prolonged exposure to water vapor at intermediate temperatures (~30°–300°C) [36,39]. Studies have found a strong influence of surface finish on the ageing kinetics of zirconia [36]. Polishing-induced residual stresses can prompt preferential transformation in the polishing zone, resulting in induction of scratches and consequent acceleration of ageing in zirconia [36]. Studies found a compressive surface stress layer on the roughly polished zirconia surface, which appeared to be beneficial for the ageing resistance [40]. In contrast, smoothly polished zirconia surfaces might have produced the preferential transformation nucleation in the polishing zone to accelerate its aging, which required a thermal treatment to relax the residual stresses [2]. Given the complex nature of phase transformations in zirconia, the surface roughness alone cannot be used for ensuring long-term reliability. Thus, both the quality of surface finish and the associated polishing-induced residual stresses are inseparable issues and need to be controlled when considering clinical applications of zirconia.

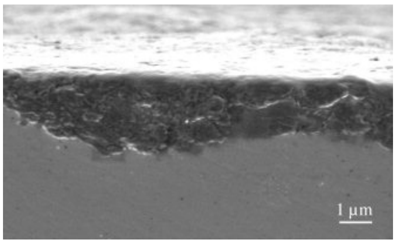

Fig. 4. Subsurface damage of up to 4 μm on the Y-TZP surface blasted by 50-μm Al_2O_3 particles [41].

Zirconia devices also require surface roughening treatment. For instance, all interior surfaces of zirconia crowns and bridges must be sandblasted to enable the bond between the luting agent and the zirconia restorations. This is critical in clinical practice in all-ceramic crown restorations [31,41,42]. Sandblasting is conducted using abrasives to impact interior crown surfaces at a low pressure in a sandblaster [41], in which zirconia undergoes indentation, scratches and impact by moving hard abrasives. The process roughens zirconia surfaces and also introduces surface flaws and defects that can compromise the strength of the zirconia restorations. Fig. 4 reveals the sandblast-induced subsurface damage of up to 4 μm on a Y-TZP surface blasted by 50 μm alumina particles [41], which is severe for the material with submicrometer grain sizes and deteriorates the mechanical strength and fatigue behaviour of the material [42,43].

Surface modification techniques have recently been developed to improve the aesthetics and bonding strength of zirconia monolithic crowns. These techniques include ceramic coating on zirconia surfaces and glass infiltration into zirconia surface/d subsurface microstructures. In particular, glass-infiltrated, functionally graded glass/zirconia/glass structures showed improved damage resistance, aesthetics, and cement adhesion [44–47]. Fig. 5(a) shows the coating layer of nanostructured and needle-shaped alumina particles on zirconia for modification of intaglio surfaces of zirconia-based restorations with the improved cementation strength [48]. Fig. 5(b) reveals the glass-infiltrated layer on zirconia, providing improved aesthetics for monolithic zirconia crowns and significantly increasing the flexural strength of monolithic structures by mechanically outperforming bi-layered porcelain-veneered zirconia and lithium disilicate glass–ceramic counterparts [46,48].

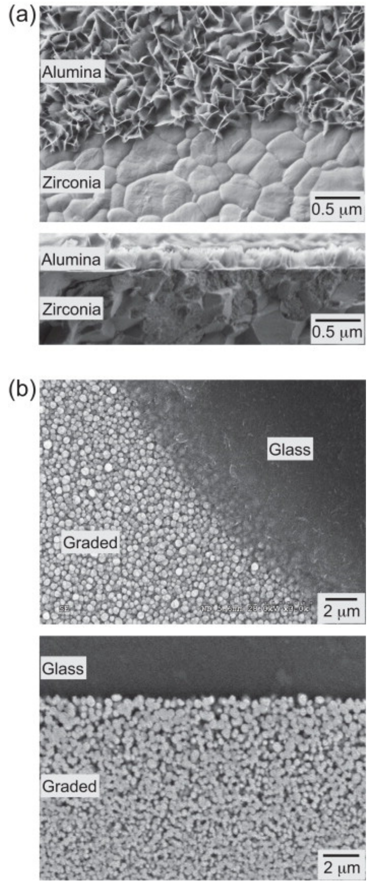

Fig. 5. (a) Nanostructured alumina coating on the zirconia substrate: surface view (top) and section view (bottom); (b) Glass-infiltrated zirconia: surface view (top) and section view (bottom), showing the outer surface glass layer and the graded glass zirconia layer [48].

4. Zirconia Surface Functions

4.1. Mechanical functions

The integrity of final surfaces conditions the mechanical behavior of zirconia devices [2,19]. Machining-induced surface defects, such as grain pullouts, micro-craters and deep subsurface damage, are the origins of surface cracks and crack propagation, resulting in fracture failures of zirconia restorations and joint replacements. Particularly, both joints and dental restorations undergo cyclic loading, which promotes microcrack extensions by mechanical degradation processes. Compared to the initial flexure strength, the dynamic fatigue strength reduced by 86.3%, 73.4% and 42.3% for polished, 50-μm grit ground and 120-μm ground zirconia surfaces, respectively, at 1,000,000 cycles, 1 hz and 0.5 s contact time in three-point flexure strength testing [49]. Polishing can successfully improve surface roughness with optical mirror finish by producing thin layers of monoclininc phase and compressive stresses in zirconia. It can also increase the sensitivity to zirconia aging by enhancing monoclinic phase nucleation around residual scratches [36].

Published data have shown a possible role of phase transformation-induced compressive residual stresses on machined zirconia surfaces. Studies claim that machining-induced compressive residual stresses can be beneficial to the fatigue behavior of zirconia because these stresses can counter with the slow crack growth which extends microcracks for radial crack initiations. Thus, it is expected to have compressive residual stresses on top occlusal surfaces in zirconia monolithic crowns to shield the subsurface damage to some extent from the immediate occlusal forces [21]. Other studies indicate that grinding or aging did not result in any deleterious impact on the mechanical properties of zirconia although a high monoclinic phase content and roughness were observed on ground surfaces [50]. The machining-induced degradation might not be sufficient enough to introduce catastrophic strength losses from crack coalescence during fatigue and fretting in zirconia [50].

4.2. Bio-functions

Traditional mechanical prerequisites of engineered surfaces for medical/dental devices are obviously important but biological parameters of theses surfaces also determine the functional performance and quality of the devices [51,52], because these surfaces are often colonized by human pathogens that can form biofilms and cause infections. It is critical to evaluate biological functions of these interfaces.

Biofilms are a major concern for biomaterial surfaces. In a comparison of commercial titanium and zirconia surfaces with the same roughness ranging from 730 to 760 nm *Ra*, zirconia surfaces attracted much less bacteria than titanium surfaces [53]. In joint replacements, bacterial colonization of zirconia has compromised the effectiveness of joint implants and resulted in persistent infections. In dental restorations, bacterial plaque accumulation at restorative surfaces can cause dental caries, gingival inflammation and periodontal problems [54]. In general, plaque retention is associated with the surface roughness and the surface energy of restorative materials. The clinical evidence proves that rougher surfaces enhance bacterial colonization. Bacterial contamination appeared extensively on cracks in symptomatic vital teeth [55]. In *in vivo* studies of materials (human enamel, gold, amalgam, acrylic resin, resin composite, glass ionomer and porcelains) responses to different surface treatments (polishing, scaling, brushing, condensing, glazing or finishing), a threshold surface roughness of 200 nm *Ra* was established for bacterial plaque retention [37]. However, it is not clear whether this threshold can be applied to zirconia surfaces because some bacterial adhesion was detected on all zirconia implant surfaces with roughness ranges of 119–259 nm *Ra* [56].

5. Outlook

Abrasive machining has been the key processing technique for fabrication of complex shape of zirconia devices. Intricate complexities in machining-induced phase-transformation, which will affect the mechanical and chemical behavior of Y-TZP, have not yet been fully explored. Many publications have claimed that zirconia ceramics exhibit stress-induced transformation. However, little published work is available to reveal directly measured stress distributions on machined zirconia surfaces and how these stresses affect the surface performance. This is probably due to the thin affected surface layer at the micron scale, which makes many methods insensitive [57]. Both x-ray and neutron beams have been used for internal stress measurement of zirconia materials [58,59]. Synchrotron x-ray diffraction may be the useful for mapping the strain tomography of zirconia surfaces [58]. These techniques require very complex data interpretation and tomographic reconstruction of strain, and have not been used for machined surface residual stress measurement in zirconia.

Although zirconia is generally considered chemically stable, the consequences of various environments on crack growth and strength degradation of zirconia in a human body have been questioned [60]. The aqueous dissolution-induced destabilization of Y-TZP and low temperature degradation of zirconia have to be considered when applying zirconia as a biomedical material [60]. The performance of zirconia femoral heads in orthopaedics and zirconia dental restorations in different pH conditions is not clearly understood. How machined surfaces of zirconia respond to chemical/environmental degradation is of particularly importance to the long-term functionality of zirconia devices in human bodies.

Abrasive machining-induced surface and subsurface damage in zirconia has been a bottleneck for the reliable application of the material. There is a need to develop non-abrasive machining processes for ceramics, zirconia included. More recently, nonconventional and novel manufacturing techniques have been used for zirconia materials. For instance, laser micromachining of Y-TZP, has been developed for sintered zirconia to achieve smooth, microcrack-free surfaces and high material strength [61]. However, in terms of processing costs, more research needs to be conducted to make the process acceptable by medical and dental clinicians. Many additive manufacturing techniques have also been applied to fabricate zirconia for dental restorations. Direct inkjet printing of zirconia prostheses was invented in which extensive laser-sintering-induced thermal cracks were found in the zirconia microstructure [62]. Further, dimensional accuracy and surface roughness are crucial issues because of the nature of the layer-by-layer formation of the zirconia components [63–65]. Indirect selective laser sintering of zirconia also induced cracks in zirconia, which can be diminished by pressure infiltration and warm isostatic pressing. However, the achieved density was 85%, lower than in conventional processes [66]. Selective laser melting/sintering also causes severe deformation of zirconia components [64]. Laser re-melting has been applied for improved density, surface quality and microstructure of metal materials in additive manufacturing [63]. It may be used for ceramics with improved quality. Robotic assisted deposition or robocasting is also used for fabrication of zirconia for dental and medical application [65]. Another novel extrusion-based additive manufacturing process for ceramics, called the ceramic on-demand extrusion (CODE), was successfully developed to produce alumina. The process is currently being investigated for complex zirconia components [67].

To overcome ageing sensitivity and improve aesthetics in zirconia, zirconia-based ceramics have been developed. Ageing-free zirconia materials have been achieved by doping and composites for long-term reliability of all zirconia devices [5]. Silica-, or ceria-doped zirconia materials [5] and zirconia toughened alumina composites or zirconia toughened lithium disilicate composites [59] have been developed. For precision tooth colour reproduction with monolithic zirconia restorations [2,9], nanostructured zirconia restorations have been invented with combined translucency and mechanical properties [20,68]. However, little is known about the machinability of these new multi-phase zirconia-containing materials, in which multiple phase transformations and heterogeneous fractures of different crystals, can be very complex and dominant in machining processes [69]. The improved mechanical properties of nano-structured zirconia will make the material more difficult to machine in its sintered state using conventional diamond machining techniques.

6. Conclusions

(a) Microstructure and mechanical properties of zirconia determine the selection of soft and hard machining with different machines and tools at different machining cost. Soft machining is predominant by brittle mode removal while hard machining is quasi-plastic. Both processes induce surface and subsurface damage which is a bottleneck in the application of zirconia as medical devices and dental restorations.

(b) Due to the complex nature of phase transformations in zirconia, the coupling issue of surface roughness and residual stresses on polished zirconia surfaces needs to be further investigated. Particularly, the quantitative measurement of residual stresses on polished zirconia layers with micro-scale thicknesses is very challenging. Synchrotron-based x-ray or neutron radiation might be useful tools for the measurements.

(c) Machined surface texture and roughness play key roles in determining the mechanical and biofunctional performance. It is very necessary to establish the quantitative relationships between surface roughness and performance indicators for quality assurance of zirconia surfaces with mechanical reliability and biofunctions.

(d) Emerging manufacturing techniques (e.g., additive manufacturing) will be alternative tools for fabrication of zirconia surfaces. However, technical hurdles such as internal defects, poor surface roughness, and lower density associated with laser sintering and layer-by-layer processing are challenging. Laser re-melting might be a useful technique for zirconia to achieve reduced material defects and improved surface quality.

(e) Many newly developed zirconia-containing composites with improved mechanical properties and functionality impose new manufacturability challenges.

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