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REVIEW ARTICLE

Reliability and properties of core materials for all-ceramic dental restorations

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Summary Various core materials have been used as all-ceramic dental restorations. Since many foreign zirconia product systems were introduced to the Japanese dental market in the past few years, the researches and the papers on zirconia for ceramic biomaterials have immediately drawn considerable attention. Recently, most of the manufactures supply zirconia blocks available to multi-unit posterior bridges using CAD/CAM, because zirconia has excellent mechanical properties comparable to metal, due to its microstructures. The properties of conventional zirconia were further improved by the composite in nano-scale such as zirconia/alumina nanocomposite (NANOZR). There are many interesting behaviors such as long-term stability related to low temperature degradation, effect of sandblasting and heat treatment on the microstructure and the strength, bonding to veneering porcelains, bonding to cement, visible light translucency related to esthetic restoration, X-ray opacity, biocompatibility, fracture load of clinical bridge as well as lifetime and clinical survival rates of the restoratives made with zirconia. From the recent material researches on zirconia not only in Japan but also in the world, this review takes into account these interesting properties of zirconia and reliability as core material for all-ceramic dental restorations.

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1. Introduction

Modern dentistry is said to have its beginnings during the year 1728, when Fauchard published a treatise describing many types of dental restorations, including a method for the construction of artificial dentures from ivory. The year 1792 is important as the date when de Chamant patented a process for the construction of porcelain teeth; this was followed early in the next century by the introduction of the porcelain inlay [1]. However, since Taggart introduced the technique of cast inlay restorations in 1907, metals have been accepted as the most reliable dental restorations because of higher mechanical strength and easier preparation than ceramics. Sixty years later, reinforcement of the jacket crown with aluminum oxide was achieved as a result of the work of McLean and Hughes. Further materials developments were based on increasing the crystalline content, for example leucite (Empress), mica (Dicor), hydroxyapatite (Cerapearl) or mixed glass oxides (In-Ceram). Pure crystalline oxide ceramics (*e.g.* Procera AllCeram) have only been used for about 15 years. Casting (Dicor), pressing (Empress) and grinding techniques (CEREC) are all used to create morphology. The idea of using CAD/CAM techniques for the fabrication of tooth restorations originated with Duret in the 1970s. Ten years later, Mörmann developed CEREC-system first marketed by Siemens (now Sirona), which enabled the first chairside fabrication of restorations with this technology. There has been a marked acceleration in the development of other CAD/CAM laboratory systems in recent years as a result of the greatly increased performance of personal computers and software [2] Then, reliable metal-free restorations were achieved using these CAD/CAM systems and zirconia, and various product systems were introduced in this decade: Cercon from DeguDent in 1998; Procera AllZircon (now Procera Zirconia) from Nobel BioCare in 2001; and In-Ceram YZ CUBES from VITA in 2004. In Japan, Cercon, Lava, and ZENO systems were accepted as dental restoratives from the Ministry of Health, Labour and Welfare, in 2005 and 2006. NANOZR, nanocomposite consisting of nano-sized and submicron-sized zirconia and alumina, was also accepted in 2006.

Most of these zirconia blocks for CAD/CAM must be made from the zirconia powders made in Japan. Unfortunately, interests of Japanese companies and researchers related to zirconia have been concentrated to connector of optical fiber, oxygen sensor, and catalyst for purifying of car exhaust. Then, the Japanese products monopolize these markets in the present world. On the other hand, biomedical applications of zirconia in Japan have not been of high interest. Once many foreign zirconia product systems were introduced to the Japanese dental market in the past few years, the researches and the papers on zirconia for ceramic biomaterials have immediately drawn considerable attention. Furthermore, Procera manufacturing facility opened in Chiba, Japan on 1 September 2007. It can be remarked that "The Era of Zirconia has begun in Japan".

In this review, from the recent material researches on zirconia not only in Japan but also in the world, interesting properties of zirconia are described and reliability as core material for all-ceramic dental restorations are discussed.

2. Core materials used as all-ceramic dental restorations

Table 1 shows major core materials for all-ceramic dental restorations and their main composition, forming method, and temperature for pressing or sintering. Most of the manufacturers supply zirconia blocks available to multi-unit posterior bridges using CAD/CAM. Other materials such as Ceraeste, Finess All Ceramic, and IPS Empress Esthetic, and IPS e.max Press, etc. are recommended to apply to a single crown. Although OCC, Crycera, and Ceraeste were developed in Japan, their systems cannot handle zirconia. Among the systems made in Japan, KATANA is the only system using zirconia and CAD/CAM system made in Japan. NANOZR was also developed in Japan in 1998 [3,4], and accepted from the Ministry of Health, Labour and Welfare, in 2006, and will be soon supplied upon the final check of productivity conditions. Most of the other zirconia are classified to a conventional 3Y-TZP, namely 3 mol% yttria-stabilized tetragonal zirconia polycrystals (TZPs).

Table 2 shows mechanical and physical properties of these core materials [5—7]. There is no doubt that zirconia core ceramics have mechanical properties better than other ceramic core materials. Although Procera AllCeram (now Procera Alumina) made with alumina showed the largest elastic modulus and the highest hardness, these properties are inadequate as dental restoratives because of the inadequacy for the deflection and the wear of natural teeth. Fig. 1 shows the relation between the fracture toughness and the flexure strength of core ceramic materials. Among them, NANOZR showed the highest flexural strength, the highest fracture toughness, slightly higher elastic modulus, slightly lower thermal expansion, and slightly lower density. It is due to the unique properties of NANOZR as described as follows.

3. Microstructure of zirconia

Fig. 2 shows the scanning electron micrograph of the surface of a conventional Y-TZP, Cercon (left) and a Ce-TZP/alumina nanocomposite, namely NANOZR (right). Cercon consists of

Table 1 Core materials and its composition, forming method, and temperature for pressing or sintering

Manufacturer	Brand name	Main composition	Forming method	Temperature for pressing or sintering
Dentsply	Dicor	Mica + glass	Casting	1360 °C
Olympus	OCC	Mica + β-spodiumen + glass	Casting	1100–1200 °C
Kyushu Dentceram	Crycera	β-Metaphosphate calcium + glass	Casting	1100–1200 °C
Ceramco	Finess All Ceramic	Leucite + feldspathic glass	Press	930 °C
Tokuyama	Ceraeste	Diopside + glass	Press	900 °C
Ivoclar	IPS Empress Esthetic	Leucite + feldspathic glass	Press	1075 °C
	IPS Empress CAD		CAD/CAM	–
	IPS e.max Press	$Li_2O{\cdot}2SiO_2$ + feldspathic glass	Press	915–920 °C
	IPS e.max CAD		CAD/CAM	Cryst. 850 °C
	IPS e.max ZirPress	Furuoroapatite + feldspathic glass	Press	900–910 °C
	IPS e.max ZirCAD	Y-ZrO_2	CAD/CAM	1500 °C
Vita	In-Ceram Spinell	Porous $MgO{\cdot}Al_2O_3$ + lanthanide glass	Dipping, CAD/CAM, electrophoresis and glass infiltration	Core: 1180 °C; glass: 1100 °C
	In-Ceram Alumina	Porous Al_2O_3 + lanthanide glass		Core: 1120 °C; glass: 1100 °C
	In-Ceram Zirconia	Porous $Al_2O_3{\cdot}$Ce-ZrO_2 (33 wt%) + lanthanide glass		Core: 1180 °C; glass: 1140 °C
	In-Ceram YZ CUBES	Y-ZrO_2	CAD/CAM	1530 °C (7.5 h)
KaVo	Everest G-Blank	Leucite + feldspathic glass	CAD/CAM	–
	Everest Z-Block	Y-ZrO_2		–
	Everest HPC	$ZrSiO_4$		1575 °C (4 h)
Sirona	CEREC Blocs	Feldspathic porcelain	CAD/CAM	–
	InCoris AL	Al_2O_3		–
	InCoris ZI	Y-ZrO_2		–
Nobel Biocare	Procera AllCeram	Al_2O_3	CAD/CAM	1700 °C
	Procera AllZircon	Y-ZrO_2		–
DeguDent	Cercon	Y-ZrO_2	CAD/CAM	1350 °C (6.5 h)
3M ESPE	Lava	Y-ZrO_2	CAD/CAM	1500 °C (11 h)
Wieland	ZENO Zr discs	Y-ZrO_2	CAD/CAM	1450 °C
Noritake	KATANA	Y-ZrO_2	CAD/CAM	1350 °C
Matsushita Electric Works	NANOZR	Ce-ZrO_2/Al_2O_3 nanocomposite	CAD/CAM	1450 °C (2 h)

homogeneous grains, average 0.32 μm. NANOZR is composed of 10 mol% CeO_2 stabilized TZP (white grains) as a matrix and 30 vol% of Al_2O_3 (black grains) as a second phase. The average grain size of the NANOZR was 0.49 μm. Nanosized particles were shown with arrows in the right photo. The significant characteristic of its structure is an intergranular-type of nanostructure, in which several 10–100 nm-sized Al_2O_3 particles are trapped within the ZrO_2 grains and several 10 nm-sized ZrO_2 particles are trapped within the Al_2O_3 grains (Fig. 3). This structural feature can explain why NANOZR has better mechanical properties than the conventional Y-TZP.

Zirconia is a well-known polymorph that occurs in three forms: monoclinic, tetragonal, and cubic. Pure zirconia is monoclinic at the room temperature. This phase is stable up to 1170 °C. Above this temperature, it transforms into tetragonal and then into cubic phase at 2370 °C. During cooling, a tetragonal–monoclinic transformation takes place in a temperature range of about 100 °C below 1070 °C. The addition of stabilizing oxide like CaO and MgO to pure zirconia allows to generate multiphase materials known as partially stabilized zirconia (PSZ) which consists of cubic zirconia as the major phase at room temperature, with monoclinic and tetragonal zirconia precipitates as the minor phase. Garvie et al. [8] called PSZ as "Ceramic Steel" and showed how to make the best of tetragonal–monoclinic transformation in PSZ improving mechanical strength and toughness of zirconia. They observed that tetragonal metastable precipitates finely dispersed within the cubic matrix were able to be transformed into the monoclinic phase when the constraint exerted on them by the matrix was relieved, *i.e.* by a crack advancing in the material. In that case, the stress field

Table 2 Properties of ceramic core materials

Forming method	Brand name	Flexural strength (MPa)	Fracture toughness (MPa m$^{1/2}$)	Elastic modulus (GPa)	Hardness (VHN)	CTE[a] ($\times 10^{-6}$ per °C)	Density (g/cm^3)
Casting	Dicor	152	2.5	70	620	7.2	2.7
	OCC	222–300	2.7	53	360–420	8.0	2.8
	Crys-Cera	160	2.7	80	420	11.5	2.7
Press	Finess All Ceramic	125	1.25	70	635	13.5	–
	Ceraeste	300[b]	–	80	580	5.7	2.8
	Empress 2	400[c]	3.3	96	540	10.6	–
Glass infiltration	In-Ceram Spinell	400	2.7	185	–	7.7	3.57
	In-Ceram Alumina	500	3.9	280	1122	7.4	3.84
	In-Ceram Zirconia	600	4.4	258	1122	7.8	4.24
CAD/CAM	Procera AllCeram	472–687[c]	3.84–4.48	370–420	1700[d]	7.0[d]	3.9[d]
	Procera Zirconia Y-TZP	1121	10	210	1200	10.4	6.05
	In-Ceram YZ CUBES	>900[b]	5.9	210	1200	10.5	6.05
	Cercon	900–1200[c]	9–10	200–210	1275	10.5	6.1
	Lava	1272[c]	10	210	1250	10	6.08
	NANOZR	1500[c]	18	245	1160	10.1	5.53

[a] Coefficient of thermal expansion at 25–500 °C.
[b] Three-point flexure test.
[c] Biaxial flexure test; no sign is no indication of flexural test method.
[d] Data of high-density alumina for engineering material.

associated with expansion due to the phase transformation acts in opposition to the stress fields that promotes the propagation of the crack. An enhancement in toughness is obtained, because the energy associated with crack propagation is dissipated both in the tetragonal–monoclinic transformation and in overcoming the compression stresses due to the volume expansion [9]. Fig. 4 shows a schematic illustration of this phenomenon.

PSZ can also be obtained in the Y_2O_3–ZrO_2 and CeO_2–ZrO_2 system. However, in these systems, it is also possible to obtain ceramics formed at room temperature with a tetragonal phase only, called TZPs. Then, both systems are abbreviated to Y-TZP and Ce-TZP, respectively. Ce-TZP shows a very high toughness and a complete resistance to low temperature aging degradation in comparison to those of Y-TZP [10,11]. However, the attractive properties are accompanied by a modest strength and a modest hardness. To compensate these disadvantages in Ce-TZP, recent investigations have been focused on 12 mol% Ce-TZP/Al_2O_3 composites. However, toughness decreased remarkably with increasing Al_2O_3 content.

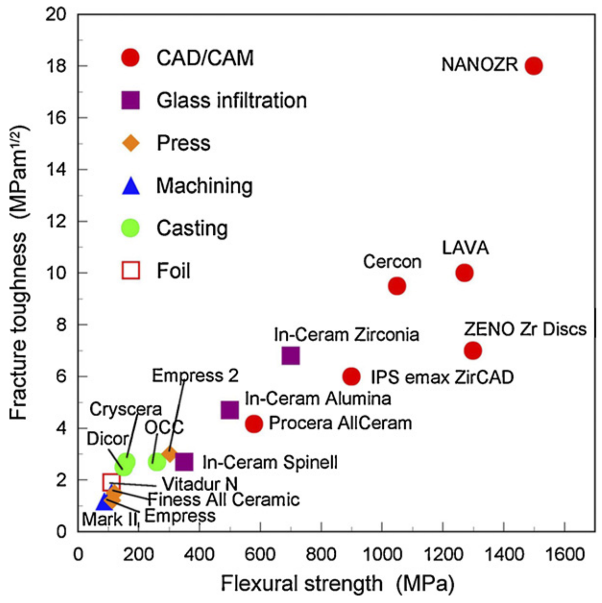

Figure 1 Relation between fracture toughness and biaxial flexure strength.

Niihara [12] developed nanocomposites, in which nanometer-sized second-phase particles are dispersed within the ceramic matrix grains and/or at the grain boundaries. Nawa et al. [3,4] applied this concept to improve the toughness of Ce-TZP/Al_2O_3 composite system and developed Ce-TZP/Al_2O_3 nanocomposite, called NANOZR, as shown in Figs. 2 and 3. The strengthening was determined as a result of two separate constituents. The first concerns a decrease in flaw size relating a reduction of the grain size for both the ZrO_2 and Al_2O_3 grains. Furthermore, associated with the interpenetrated intragranular nanodispersion, several 10–100 nm-sized inclusions are believed to have a role in dividing a grain size into more finer sized particles by forming sub-grain boundaries. The second constituent concerns the stress-induced transformation on strengthening for TZP ceramics, as shown in Fig. 4. It has been determined that the retention of the tetragonal phase is critically governed by the grain size [13]. That is, reduction of the grain size is predicted to increase the critical stress that induces the tetragonal–monoclinic transformation. These interactive contributions are considered to result in the improvement of the strength [4].

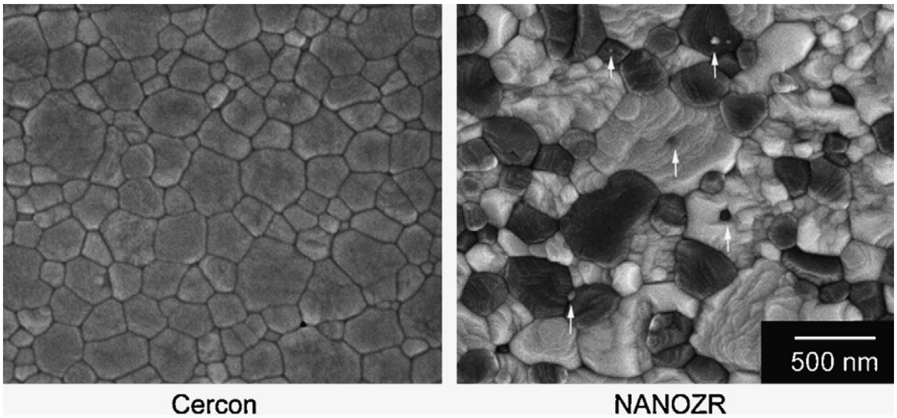

Figure 2 Microstructure of a conventional Y-TZP (left) and a Ce-TZP/alumina nanocomposite (right).

4. Long-term stability

As mentioned above, mechanical properties of zirconia depend on its fine grained, metastable microstructure. Then, the stability of this structure during the lifetime of TZP components is the key point to attain the expected performances. Mechanical property degradation in zirconia, known as aging, is due to the progressive spontaneous transformation of the metastable tetragonal phase into the monoclinic phase. This behavior is well known in the temperature range above 200 °C in the presence of water vapor, namely hydrothermal degradation. This phenomenon is called low-temperature degradation (LTD). So many papers about LTD have been published. Among them, Japanese researchers' studies are very important to biomedical application. For instance, Sato and Shimada [10] demonstrated that the transformation rate in water, which was much greater than that in air, was first order with respect to surface concentration of tetragonal zirconia. They [11] also reported that the transformation rate for Ce-TZP was extremely slower than that of Y-TZP, but the values of activation energies for both zirconia were almost the same, about 90 kJ/mol.

These facts indicate that the phase transformation was controlled by the chemical reaction between water and Zr—O—Zr bond on the surface as follows. On the basis of the corrosion mechanism, the reaction scheme for the reaction between water and Zr—O—Zr bonds at the crack tip [10] can be shown in Fig. 5. It suggests that Zr—OH bond is formed in

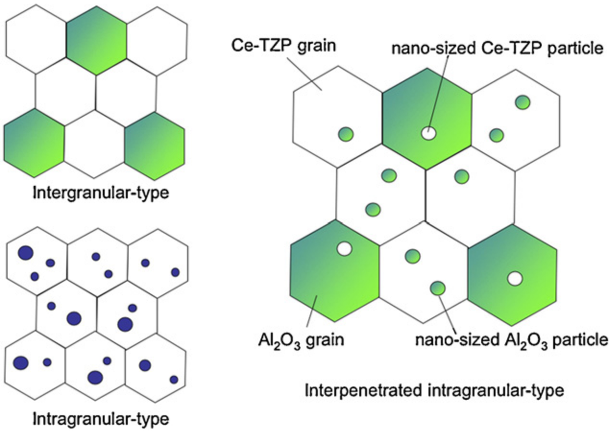

Figure 3 Schematic illustration of interpenetrated intergranular-type toughening of zirconia.

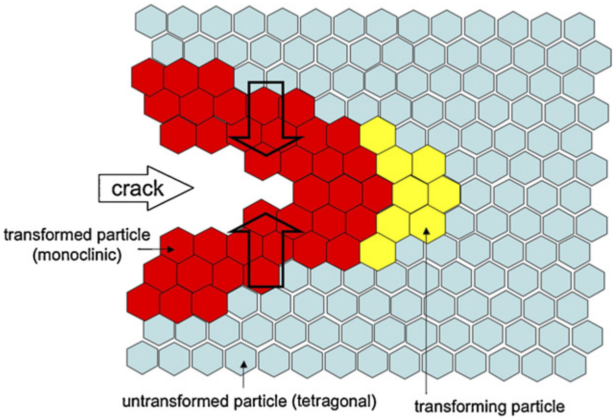

Figure 4 Inhibition of crack propagation by stress-induced phase transformation.

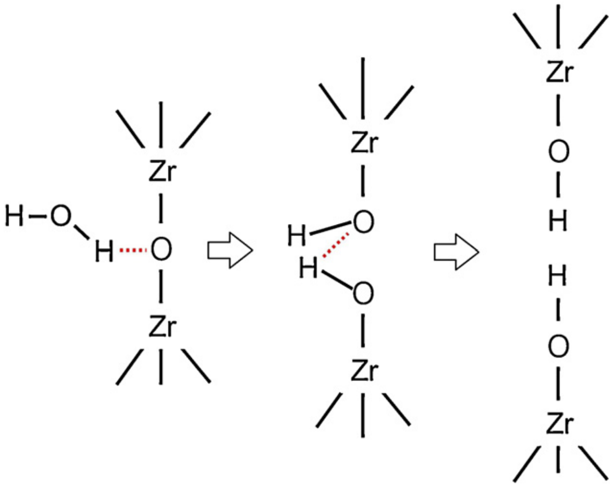

Figure 5 Schematic illustration of the reaction between water and Zr—O—Zr bonds at crack tip.

Table 3 Summary of some results of aging tests on zirconia for biomedical application

Ref. no. (year)	Material	Medium	Temperature (°C)	Time	Monoclinic content	Strength
[17] (1992)	Y-TZP	Implantation in subcutaneous tissue of rat	Body temperature	12 months	—	No change of three-point bending test
[15] (1996)	Y-TZP	Saline	37	30 months	With the rise in aging temperature, the speed of phase transformation changed markedly. After 36-month storage at 95 °C, 71.5% monoclinic phase was transformed	No serious decrease of three-point flexural strength was found over the 3-year period under the left conditions. It is concluded that zirconia can be used clinically because it retains a bending strength of over 700 MPa under any experimental conditions for over 3 years
			95	36 months		
		Steam	121	40 days		
[19] (2000)	(Y, Nb)-TZP/ 20 vol% Al_2O_3	Steam 4 MPa	200	5 h	No change	—
[20] (2002)	Y-TZP	4% acetic acid	80	168 h	The monoclinic phase increased to 20—25% from 2% of the unexposed ones	The three-point flexural strengths of the aged specimens were not significantly different than the corresponding unexposed specimens
[21] (2002)	Y-TZP NANOZR	Implant in rabbit tibia	Body temperature	18 months	Slight change for both materials	—
		Physiological saline	62	18 months	Y-TZP showed 78.7% monoclinic phase, whereas NANOZR showed only slight change	—
		Autoclave	121	190 h	Y-TZP showed 81% monoclinic phase, whereas NANOZR showed only slight change	The three-point flexural strength of Y-TZP was significantly lower after aging for 108 h than its initial strength
[22] (2003)	Y-TZP NANOZR	Autoclave	121	18 h	Y-TZP showed 25.3% monoclinic phase, whereas NANOZR showed only slight change	—
[23] (2005)	Y-TZP	Autoclave	134	15 h	Y-TZP showed 80% monoclinic phase at 12 h at 134 °C. 1 h of autoclave at 134 °C has theoretically the same effect as 3—4 years in vivo at 37 °C	—
[18] (2006)	Y-TZP (Vita In-Ceram YZ)	Boiling water	100	7 days	XRD showed a slight transformation from tetragonal to monoclinic phase	LTD, polishing, and sandblasting did not produce strength degradation effects on the Y-TZP. The sandblasting significantly increased the three-point flexural strength. However, the sandblasting is responsible for strength degradation if not neutralized by a region of compressive stresses
		Steam	250	7 days	The greatest amount of transformation was noted on the bar after storage in humidified air at 250 °C for 7 days	

[24] (2006)	Y-TZP NANOZR	Physiological saline	37	540 days	No significant difference	No significant difference
			80	30 days		
		4% acetic acid	80	30 days		
		Autoclave	120	14 days	The monoclinic content of Y-TZP remarkably increased with the autoclaving; 3.8 and 75.5 vol% before and after the autoclaving, whereas NANOZR showed no change	The biaxial flexure strength of Y-TZP after the autoclaving showed about 17% lower than the autoclaving, whereas NANOZR showed no significant difference
[25] (2007)	Y-TZP	Steam	140	24 h	An about 100 μm-thick layer transformed on the surface of the standard-grade Y-TZP	The biaxial flexural strength of standard-grade Y-TZP showed an almost 50% strength reduction. The biomedical-grade Y-TZP, containing 0.25% alumina, exhibited a considerably higher corrosion resistance, and only a minor (about 10%) strength reduction was observed

the lattice of zirconia and it starts the phase transformation [14,15].

Chevalier [16] reported that, until 2001, 400 of Y-TZP femoral heads failed in a short period, with the fracture origin clearly associated with the hydrothermal degradation in two particular batches of a commercial product. Even if limited in time and number, and clearly identified to be process controlled, these events have had a catastrophic impact for the use of zirconia. However, no such failure event for dental zirconia has been reported so far.

Table 3 shows the summary of some recent results of aging tests on zirconia for biomedical application [15,17—25]. We reported a short-term and a long-term stability of NANOZR in comparison to Y-TZP [24,26]. Monoclinic contents of both materials did not change with the aging under the various water-based conditions, except that of Y-TZP which dramatically changed with autoclaving at 120 °C (Fig. 6). Furthermore the biaxial flexure strengths of Y-TZP slightly changed with the autoclaving. Whereas those of NANOZR showed no significant change and was significantly stronger than Y-TZP (Fig. 7). This is probably because any Y_2O_3 reacts rapidly with water vapor to form yttrium hydroxide, resulting in instability of the tetragonal zirconia [27].

On the theory after Sato and Shimada, Chevalier et al. [28] predicted a 25-year aging period at 37 °C to reach 20% monoclinic content, in which the activation energy is the same order of the one measured by Shimidzu et al. [15]. Chevalier et al. [29] also calculated that 1 h of autoclave treatment at 134 °C has theoretically the same effect as 3—4 years *in vivo* at 37 °C.

Kosmac et al. [25] reported that, compared with standard-grade Y-TZP, the biomedical-grade material containing 0.25% alumina addition exhibited a considerably higher corrosion resistance.

From these facts, it is concluded that the well-controlled zirconia has a sufficient long-term stability in oral conditions even after the slight transformation occurs, because the initial strength is quite high and the transformation durability is sufficient.

5. Effect of sandblasting and heat treatment

To realize esthetic appearance, the machined frameworks, namely cores, made with ceramics for crown and bridges were fired with some veneering porcelains at 800—950 °C. Before firing, the frameworks were sandblasted after final firing and subsequently were subjected to heat treatment to regeneration of crystal phase. It is known that the mechanical properties of Y-TZP frame are strongly affected by these treatments [30—33]. Not only the initial strength, but the long-term stability is also affected by these mechanical and heat treatments [34,35]. Usually, the sandblasting increases the strength of Y-TZP, and decreases with annealing. It can be explained that the transformation from tetragonal to monoclinic by sandblasting expands their volume to generate compressive stress on the surface, resulting in increasing of strength and the regeneration of tetragonal phase by annealing reduces the compressive stress on the surface, resulting in decreasing of strength. We compared the effects of these treatments of NANOZR to Y-TZP [36—41].

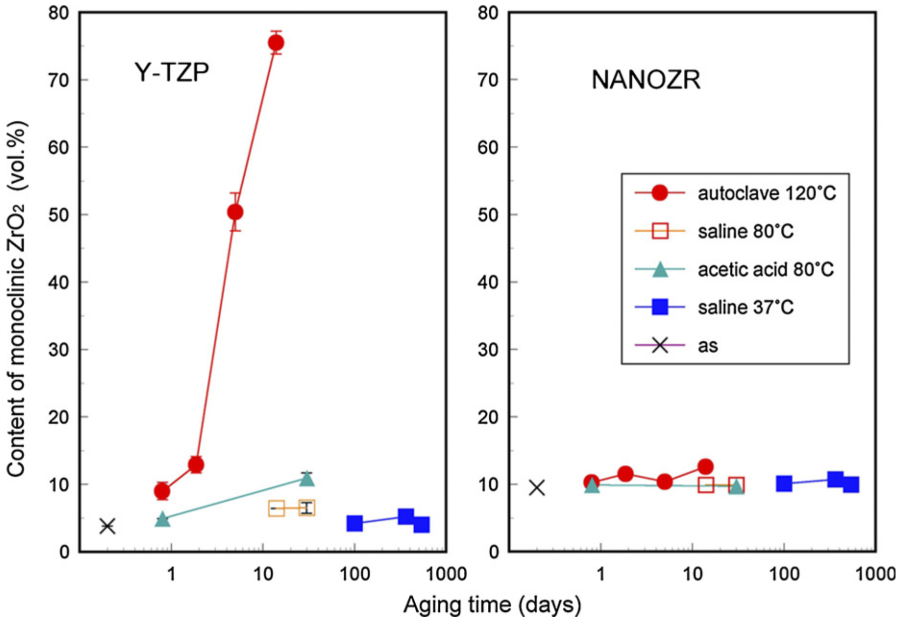

Figure 6 Monoclinic zirconia content of Y-TZP and NANOZR before and after aging test.

Monoclinic zirconia content of the Y-TZP and NANOZR changed with various treatments (Fig. 8). The monoclinic content of both the materials increased with sandblasting and decreased with heat treatment. Furthermore, the monoclinic content of the Y-TZP dramatically increased with autoclaving and those of NANOZR remarkably increased with sandblasting.

The biaxial flexure strengths of the Y-TZP and NANOZR also varied with these treatments (Fig. 9). The biaxial flexure strengths of NANOZR were definitely larger than those of Y-TZP ($p < 0.01$). The biaxial flexure strengths of both materials as machined significantly decreased by heat treatment ($p < 0.01$), but did not change by sandblasting. The strength of both materials after the heat treatment significantly increased with the sandblasting ($p < 0.01$). The biaxial flexure strengths of both materials were independent on the soaking in acetic acid. However, the strength of Y-TZP decreased with autoclaving, whereas those of NANOZR did not change.

These results suggest that the stress-induced transformation from tetragonal to monoclinic occurs more easily than Y-TZP and the resistance of LTD of NANOZR is superior than Y-TZP as mentioned above. It also means that the transformation of the NANOZR by sandblasting, resulting in increasing of strength, may be more susceptible than Y-TZP.

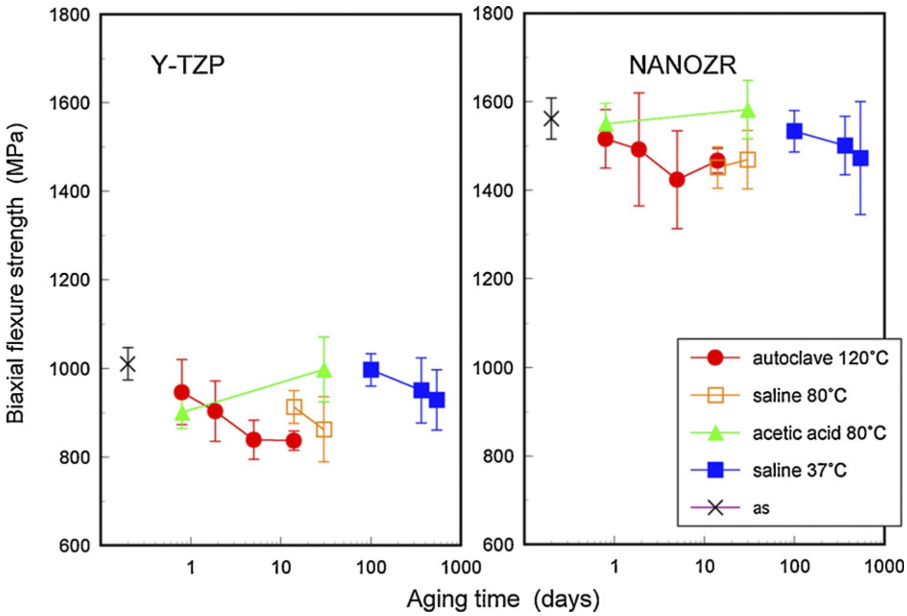

Figure 7 Biaxial flexure strength of Y-TZP and NANOZR before and after aging test.

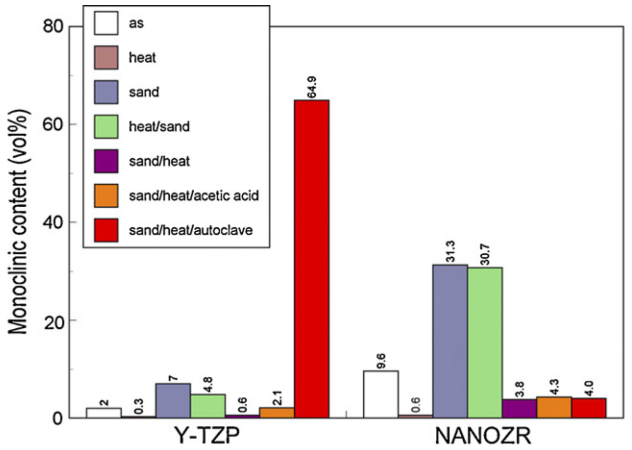

Figure 8 Monoclinic zirconia content of Y-TZP and NANOZR before and after various treatment and storage.

6. Bonding to veneering porcelain

Zirconia core should be covered by veneering porcelains to realize the esthetic restoration. Although there are some information on the bonding between zirconia and veneering porcelains (Table 4) [42—48], the test method for the bonding strength is not consistent. We measured the bonding strengths between zirconia and veneering porcelains according to ISO9693 [47]. It was assumed that the debonding/crack initiation strength due to ISO9693 is available for the bonding test between zirconia and veneering porcelain, although this specification originally prescribes for the test method of bonding strength between metal substrate and veneering porcelain. Three veneering porcelains, 8 mm in length and 1 mm in thickness, were fused to the central area of the zirconia plate, 25.0 mm × 3.0 mm × 0.5 mm. Using the three-point bending test, the debonding/crack initiation strengths were determined as bonding strength before and after 20,000 thermal cycles at 60 °C and 4 °C with a dwelling time of 1 min each. Bonding strengths between zirconia and veneering porcelains were 26.5—31.6 MPa on average for each group, and were independent to the kind of porcelain and the thermal-cycling (Fig. 10). The coefficients of thermal expansion of veneering porcelains for zirconia (8.8—10.0 × 10^{-6} per °C) are compatible to those of zirconia (10.0—10.5 × 10^{-6} per °C) [5,49]. However, there was no fact of the chemical bonding between the zirconia and the veneering porcelains, because SEM observation could not confirm the presence of reaction layer between the zirconia and the veneering porcelain. It seems that the veneering porcelains are mainly bonded to zirconia with mechanical interlocking and compressive stress due to the small difference between the zirconia and the veneering porcelain in thermal shrinkage by cooling after sintering. Further studies should be done to improve the bonding strength.

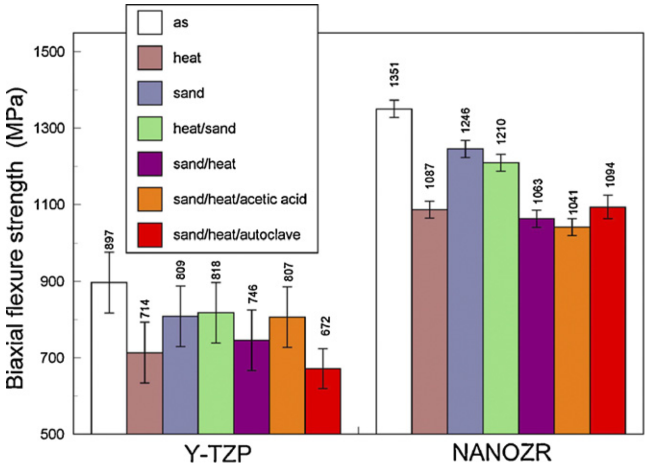

Figure 9 Biaxial flexure strength of Y-TZP and NANOZR before and after various treatment and storage.

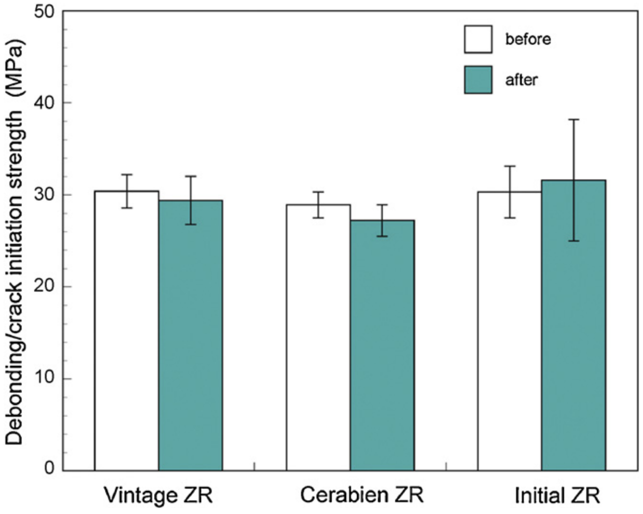

Figure 10 Debonding/crack initiation strength of NANOZR to veneering porcelains.

7. Bonding to cement

The clinical success of high strength ceramics such as zirconia strongly depends on the adhesion to natural teeth and other dental materials. The surface treatment of dental ceramics is one of the most important factors for the improvement of the adhesion [50—55]. We evaluated the effect of sandblasting on the bonding strength of dental ceramics to resin cements [36,56,57]. The surfaces of Y-TZP and NANOZR were sandblasted by 70 μm alumina and 125 μm SiC powders. The surface roughness of both zirconia sandblasted by SiC was twice larger than those by alumina, whereas the shear bonding strength of them to a resin cement (Resicem) showed no significant difference (Fig. 11). These results demonstrated that the surface roughness prepared by alumina is enough to produce the bonding of zirconia to resin cement. The shear bonding strengths of alumina-sandblasted zirconia to three resin cements varied with the cement and they decreased with the thermal-cycling in all the resin cements. Especially, the bonding strengths of Superbond C&B and Panavia F 2.0 decreased after the thermal-cycling ($p < 0.01$). There was no effect of silane coupling agent on bonding strength and durability between zirconia and resin cement. Resicem showed the best durability in the bonding strength among them [57].

Table 4 Summary of some results of bonding strength test of zirconia to veneering porcelain

Ref. no. (year)	Method and dimension	Surface treatment	Core materials	Veneering materials	Bonding strength (MPa)
[42] (2005)	Shear buttons (4 mm × 4 mm) on 3 mm-thick disc	–	Lava	VM9 bonder at 950 °C	13.8 (2.6)
				VM9 dentin wash at 950 °C	33.5 (8.6)
				Lava Ceram	19.2 (5.1)
				Noritake	16.4 (9.2)
[43] (2006)	Microtensile 1 mm × 1 mm × 6 mm	120 μm alumina sandblasting	Cercon	Ceram S	28.1 (4.5)
				Ceram Express	37.0 (6.7)
				Rond Dentine	48.8 (15.2)
				Lava Dentine	44.4 (15.1)
[44] (2006)	Shear	–	Y-TZP	Cerabien ZR	11.2 (2.0)
			NANOZR		11.1 (1.3)
[45] (2006)	Shear button (5 mm × 5 mm × 3 mm) on 10 mm × 10 mm × 1 mm disc	70 μm alumina sandblasting and heat treatment	NANOZR	Vintage ZR	27.1 (5.7)
[46] (2006)	Three-point bend (ISO9693) 25 mm × 3 mm × 0.5 mm	70 μm alumina sandblasting	NANOZR	Cercon Ceram Kiss	30.4 (1.52)
				Cerabien ZR	29.3 (1.44)
[47] (2006)	Three-point bend (ISO9693) 25 mm × 3 mm × 0.5 mm	70 μm alumina sandblasting	NANOZR	Vintage ZR	30.4 (1.8)
				Cerabien ZR	28.9 (1.4)
				Initial ZR	30.3 (2.8)
[48] (2007)	Shear button (Ø 4 mm × 3 mm) on 2 mm-thick disc	No treatment	Vita Y-TZP	VM9	54.59 (22.5)
		Grind with 120 μm diamond disc			76.38 (26.35)
		Grind and heat treatment			97.69 (27.57)

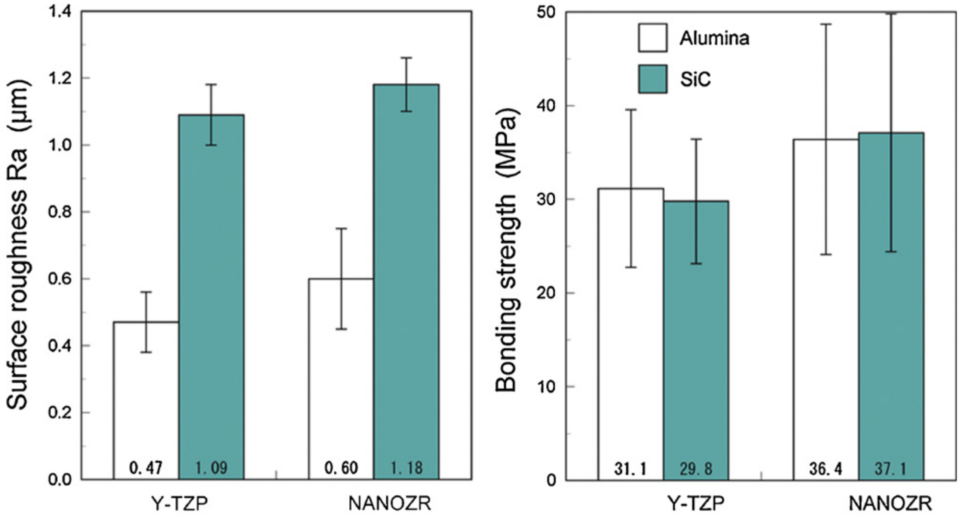

Figure 11 Surface roughness of zirconia (Y-TZP and NANOZR) sandblasted with alumina and/or SiC and their bonding strength to a resin cement (Resicem).

8. Visible light translucency

Single crystals of the cubic phase of zirconia are translucent and commonly used as a substitute for diamond in jewellery. Like diamond, cubic zirconia has a cubic crystal structure and a high index of refraction: zirconia, 2.15—2.18; and diamond 2.42—2.44. On the other hand, because most of the zirconia such as TZP and NANOZR are polycrystals, the majority of light passing through the ceramics is intensively scattered and diffusely reflected, resulting in opaque appearance [58]. We measured the transmission spectra of NANOZR of 0.1—1.0 mm in thickness (Fig. 12). In the wavelength above 380 nm, the transmission increased with increasing of the wavelength and with decreasing of the thickness. The overall transmittance at 380—700 nm of NANOZR as a function of the thickness showed that the transmission decreased with increasing of the thickness up to 0.5 mm and no remarkable change in the thickness between 0.5 and 1 mm (Fig. 13). It can be confirmed that the light transmission of NANOZR mainly depends on the scattering and the diffuse reflection. Since NANOZR consisting of zirconia and alumina particles having different index of refraction (1.76), the scattering of NANOZR is more larger than Y-TZP made with only zirconia.

The apparent translucency of zirconia is very important; some zirconias exhibit a bright white, rather opaque color, while others do not. Accordingly, it is essential to choose the bright zirconia type and optimize the production conditions such as raw material, presintering, and final sintering in order to achieve maximum strength and translucency.

Even if the optimum translucency of zirconia is achieved, the translucency of zirconia is commonly lower than those of alumina, spinell, and feldspathic porcelains. However, it is better than metal, because zirconia has no metallic color and slight transmission.

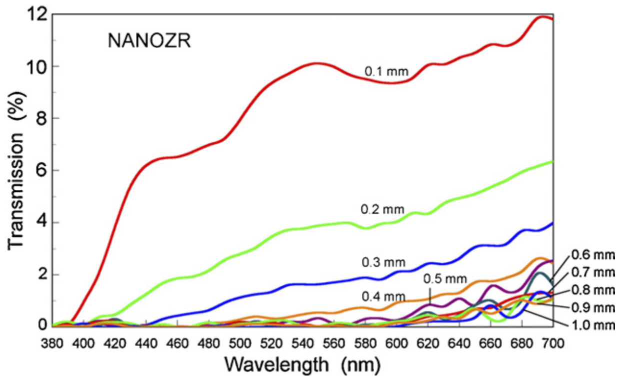

Figure 12 Transmission spectra of NANOZR of 0.1—1.0 mm in thickness.

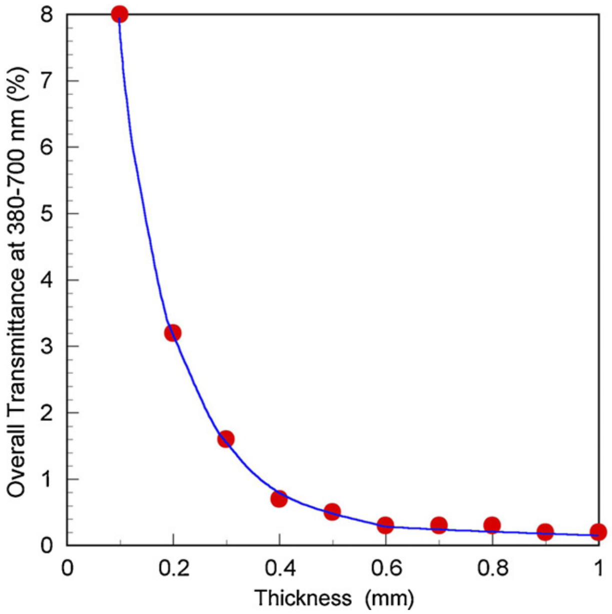

Figure 13 Overall transmittance at 380—700 nm of NANOZR as a function of thickness.

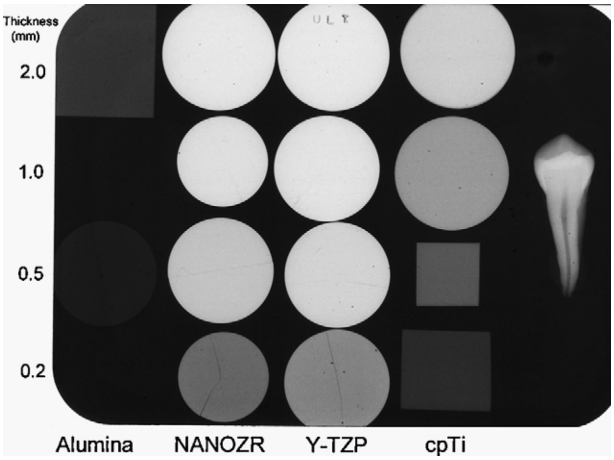

Figure 14 X-ray developed film of a commercially pure titanium, Y-TZP, and alumina plates, 0.2—2.0 mm in thickness, and a human tooth.

9. X-ray opacity

X-ray opacity of the dental restorative materials is an important information for dental treatments. It is already known that zirconia is suitable to X-ray opaque agent in the dental filling composite materials. Fig. 14 shows X-ray-developed film of a commercially pure titanium, Y-TZP, NANOZR, and alumina plates, 0.2—2.0 mm in thickness, and a human tooth. X-ray image was observed using MAXF1-F (Morita) with Kodak DF49 film under 60 kV—10 mA at 40 cm for 0.45 s. The alumina plate showed the most transparency against X-ray. Both 2 mm of Y-TZP and NANOZR showed the most opacity. cpTi showed moderate transparency. The X-ray image film was scanned using digital scanner and the darkness of the central area of the specimen image was quantitatively analyzed by free software (Scion Image 1.63). The transmittance was calculated from the darkness of the specimen plate image divided by that of the background. Fig. 15 shows the X-ray transmittance (I/I_0) of four kinds of specimen plates as a function of thickness (x). It can be assumed that the apparent absorption coefficient (μ) consists of the mass absorption coefficient (μ_{abs}) and scattering (μ_{scatter}) as follows:

$$\mu = \mu_{\text{abs}} + \mu_{\text{scatter}}$$

$$I = I_0\, e^{-\mu x}$$

From the regression curve as a function of $I = I_0\, e^{-\mu x}$, the apparent absorption coefficients were derived from the data in the thickness range $\leq$0.5 mm of NANOZR and Y-TZP, and $\leq$2.0 mm of alumina and Ti, because thicker specimen showed the saturated values.

The apparent absorption coefficients of X-ray of Y-TZP, NANOZR, Ti and alumina in this exposed condition were calculated as 3.53, 2.96, 0.84, and 0.11 mm^{-1}, respectively. Then, it is concluded that X-ray opacity is in the order of alumina $\ll$ Ti $\ll$ NANOZR $<$ Y-TZP.

The absorption coefficients of X-ray strongly depend on the effective atomic number calculated by taking the fraction portion of each atom in the compound and multiplying that by the atomic number of the atom: O 8; Al 13; Ti 22; Y 39; Zr 40; and Ce 58. Alumina showed a high transparency against X-ray, because alumina consists of Al and O having small atomic number. The X-ray opacity of NANOZR was slightly smaller than that of Y-TZP, because NANOZR contained 30 vol% of alumina and its density was also slightly smaller than Y-TZP.

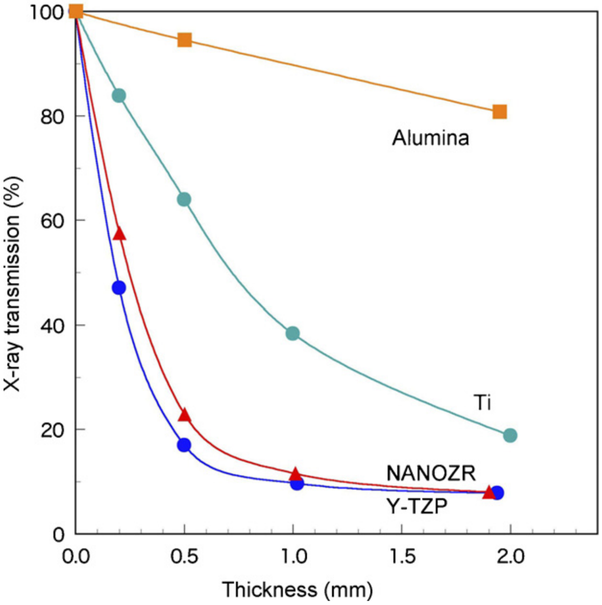

Figure 15 X-ray transmittance of four kinds of materials as a function of thickness.

Then, it is concluded that zirconia has a stronger X-ray opacity than titanium and is convenient to the implant fixture made with zirconia.

10. Biocompatibility

From more than 20 years ago, biocompatibility of zirconia has been investigated as dental implant material *in vitro* and *in vivo* (Table 5) [17,19,22,59—66]. It is conceived that zirconia is bioinert. Ichikawa et al. [17] demonstrated that tissue reaction and stability of partially stabilized zirconia ceramic *in vivo* was evaluated with the use of the subcutaneous implantation test. During the experimental period, zirconia ceramic was completely encapsulated by a thin fibrous connective tissue with less than 80 μm thickness. No changes of weight and three-point bending strength were detected after 12 months of implantation. The result suggests that zirconia ceramic is biocompatible and no degradation of zirconia ceramic occurred.

We also reported that biocompatibility of two kinds of zirconia, Y-TZP and NANOZR, was similar to commercially pure titanium [65]. Fig. 16 shows SEM photographs of MC3T3-E1 on the surface of Ti, Y-TZP and NANOZR at 1, 3, and 6 days after incubation. No inhibition was observed in all the plates. MC3T3-E1 satisfactorily has been coming in contact and proliferating with incubation. These results demonstrated that MC3T3-E1 on all the plates appeared to be attached and proliferated well. Fig. 17 shows osteoblast-like cell MC3TC-E1 proliferation on Ti, Y-TZP, and NANOZR at 1, 3, 6, and 9

Table 5 Summary of some results of biocompatible tests on zirconia

Ref. no. (year)	Material	Shape	Medium or target	Time	Results
[17] (1992)	Y-TZP	Plate	Implantation in subcutaneous tissue of rat	12 months	Zirconia was completely encapsulated by a thin fibrous connective tissue with less than 80 μm thickness
[59] (1998)	0.6–4.5 μm Al_2O_3 0.6 μm ZrO_2 4.5 μm high-density polyethylene	Particle	TNFα J774 macrophage cell	24 h	No significant difference in cell mortality and TNFα release was found between Al_2O_3 and ZrO_2
[60] (1999)	Unpurified raw zirconia Y-TZP	Plate	Clone 8 of $10T_{1/2}$ mouse embryo-derived cell	72 h	Y-TZP did not elicit either mutagenic or transforming effect on the cells
[19] (2000)	(Y, Nb)-TZP/20 vol% Al_2O_3	Disk	Mouse fibroblast cell growth and survival test	2, 4, and 6 days	No cytotoxity
		Plate	Implant in guinea pig	3 months	No significant adverse soft-tissue response
[22] (2002)	Alumina NANOZR	Bar	Implant in paraspinal muscles of Wister rats	24 weeks	Thin fibrous capsules with almost no inflammation were observed around both materials
[61] (2003)	Y-TZP Empress and Empress 2 Veneering porcelains	Extraction solutions of ceramics	Human gingival fibroblast cells	24 + 48 h	No significant cytotoxity was observed in all ceramic extractions
[62] (2003)	2.1 ± 0.4 μm Al_2O_3 1.5 ± 0.6 μm ZrO_2 1.1 ± 1.3 μm Ti_6Al_4V 4.1 ± 1.4 μm high-density polyethylene	Particle	Implant in miraine carvariae of mice	1 week	No significant differences in proinflammatory mediators or osteolytic area among Al_2O_3, ZrO_2, and control groups
[63] (2004)	Acryl cpTi Zirconia	Plate	Buccal aspect of removal of acrylic device	24 h	Bacteria on zirconia was significantly lower than the control
[64] (2004)	Ti Zirconia	Implant fixture	Implant in upper anterior-extraction sites in monkeys	5 months	Zirconia implants osseointegrated to the same extent as titanium and show the same peri-implant soft tissue dimensions
[65] (2006)	Ti Y-TZP NANOZR	Plate	MC3T3-E1 osteoblast-like cell	9 days	No significant differences were observed in proliferation among Ti, Y-TZP, and NANOZR
[66] (2007)	Ti (Y, Nb)-TZP/20 vol% Al_2O_3	Disk	Human osteosacroma cell	8 days	No difference from Ti

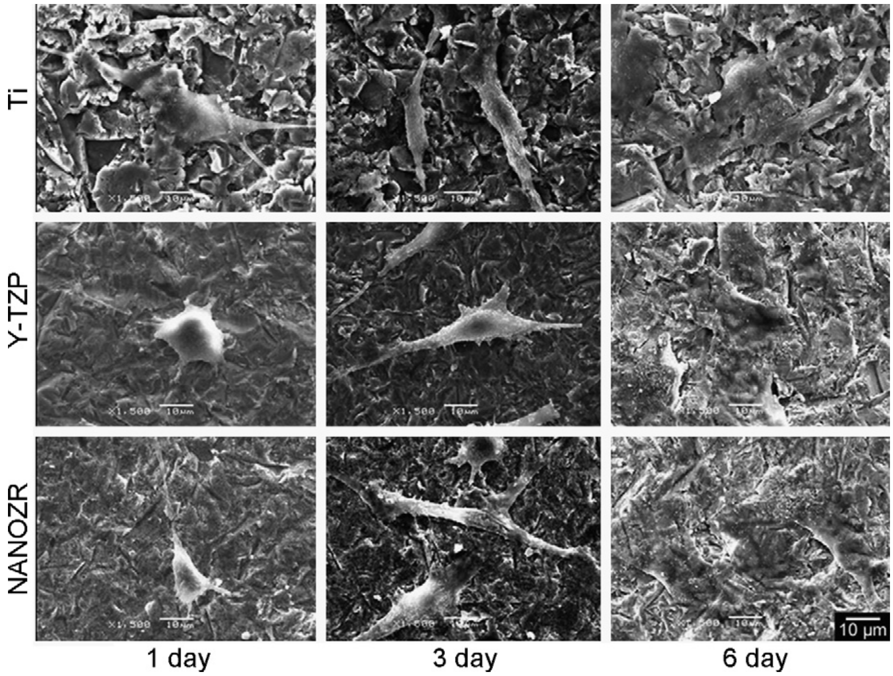

Figure 16 SEM photographs of osteoblast-like cell on a commercially pure Ti, Y-TZP, and NANOZR at 1, 3, and 6 days after incubation.

days after incubation. Although these plates have different composition and surface roughnesses, MC3T3-E1 increased with culture period in all the plates. Furthermore, there were no significant differences in the proliferation between them ($p > 0.05$). It implies that both zirconia are chemically stable to be inert on the cell proliferation such as titanium.

11. Fracture load of bridge

As described above, in comparison to Y-TZP, NANOZR has a higher biaxial flexure strength and toughness along with satisfactory durability for LTD. Furthermore, it is required to know the fracture load of the core having clinical shape.

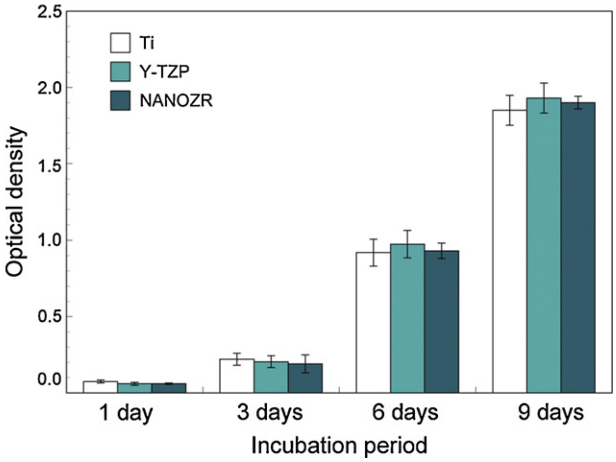

Figure 17 Osteoblast-like cell proliferation on a commercially pure Ti, Y-TZP, and NANOZR at 1, 3, 6, and 9 days after incubation.

The framework corresponding to the situation of a three-unit bridge from the second premolar to the second molar of the maxilla was designed and machined (Fig. 18). The mesial and distal connectors have a cross-section of approximately 12 and 11 mm^2, respectively. These bridge-frameworks were fixed on a metal post (16.4 mm in span length) without any cementing and loaded on the center of the bridges at 0.5 mm/min in cross-head speed with a metal rod rounded 8 mm in diameter (Fig. 19). The fracture strength of the bridge-frameworks fabricated from NANOZR was 6719 N (S.D.

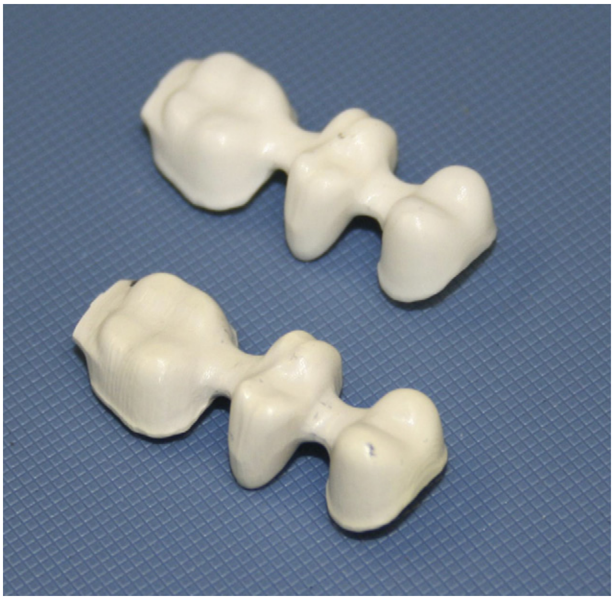

Figure 18 Appearance of three-unit bridge core made with Y-TZP (upper) and NANOZR (lower) prepared by a CAD/CAM system.

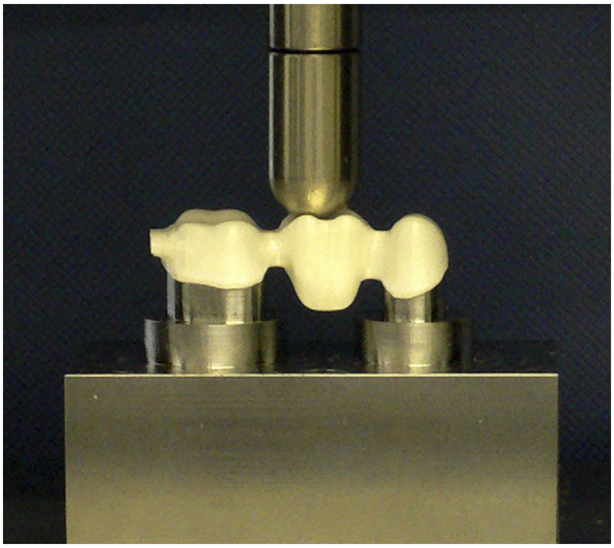

Figure 19 Appearance of test jig for fracture test of three-unit bridge.

2044 N) which is 49% higher than that of the Y-TZP, 4506 N (S.D. 1348 N) ($p < 0.05$), although the strength varied with the average thickness of wall of the frame [5]. Weibull analysis demonstrated that the fracture strengths of NANOZR and Y-TZP showed similar Weibull moduli, 2.9 and 3.1, respectively, whereas the characteristic strengths of NANOZR (7613 N) was much higher than that of the Y-TZP (5121 N), indicating a better reliability against the load-bearing (Fig. 20) [67].

12. Lifetime

Because of the brittle properties of ceramic materials, the indication spectrum for dental ceramics was considerably limited in the past. However, a supercritical loading that results in the immediate brittle fracture of a ceramic restoration is rarely observed *in vivo*, *e.g.* in case of trauma or extreme para function. In contrast, the subcritical stresses are of greater clinical importance. For instance, such stress occurs during cyclic masticatory loading and also when very small manufacturing-related structural flaws are exposed to the corrosive oral environment. This can lead to crack initiation and further propagation. If the external loading continues, the initial subcritical crack growth may reach a critical crack length and causes an unstable spread of cracks ultimately followed by failure of the ceramic restoration [68].

Various studies have already used the fracture mechanics to measure the subcritical crack extension and provide information on the susceptibility of zirconia to subcritical crack growth and thus a characterization of its long-term behavior (Table 6) [69—74].

Teixeira et al. [73] reported that the lifetime predictions after 10 years indicate a reduction of 50%, 36%, and 29% in strength for porcelain, alumina and Y-TZP, respectively.

On the other hand, Studart et al. [72,75] demonstrated that posterior bridges with zirconia frameworks can exhibit lifetimes longer than 20 years if the diameter of the bridge connector is properly designed. Each prediction of the lifetime depends on each assumption of many factors such as design, dimension, environment, and initial strength, etc. Although the conclusion of the lifetime of zirconia is still not consistent, it is commonly concluded that the lifetime of zirconia bridge is longer than those of alumina and others, and comparable to metal-based restorations.

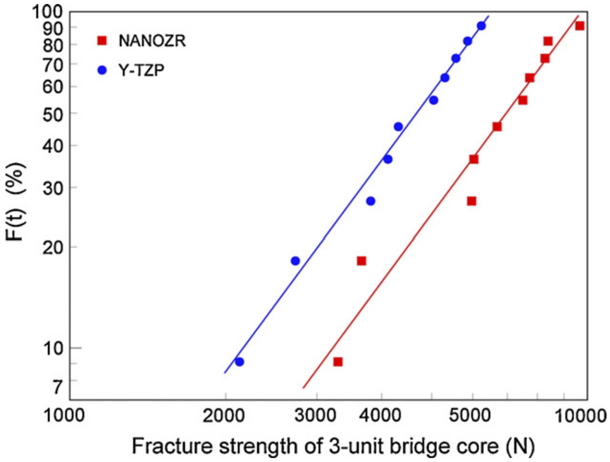

Figure 20 Weibull plot of the fracture load of three-unit bridges made with Y-TZP and NANOZR.

13. Clinical survival rate

In recent years, clinical studies with medium follow-up reported promising success rates for zirconia frameworks in anterior and posterior areas [75—78].

These studies on zirconia frameworks are listed in Table 7 in comparison with other ceramic frames and metal frames [78].

The three studies on zirconia frames provided data on the survival rate of a total of 135 prostheses after a mean follow-up of 3.4 years. Twelve prostheses were reported to be lost and its survival rate is 91.1%. For metal ceramic, four studies provided data on a total 1090 prostheses after a mean follow-up time of 8.1 years, of 119 were reported to be lost and its survival rate is 89.1%. From these data, survival rates after 5 years were estimated [78] and summarized in Table 7. Significantly lower survival rates of all-ceramic prostheses at 5 years were seen compared with meta-ceramic ones. The most frequent reason for failure of the prostheses made with glass-ceramics or glass-infiltrated ceramics was fracture of the reconstruction (framework and veneering porcelain). However, when zirconia was used as framework material, the reasons for failure were primarily biological and technical complications other than framework fracture [78].

14. Summary

Within the limitation of this review, it can be concluded that all-ceramic prostheses made with zirconia has the potential to withstand physiological occlusal forces applied in posterior region, and can alternatively replace porcelain-fused to metal restorations. Though further assessments for a long-term clinical performance must be undertaken, all-ceramic restoration made with zirconia can be recommended in daily practice.

Table 6 Summary of some results of lifetime tests on zirconia

Ref. no. (year)	Material	Shape	Load	Cycle	Environment	Strength
[69] (2005)	Y-TZP (Denzir) after machining, after heat-treatment, and after porcelain veneering	Three-unit posterior framework	0–50 N	100,000 (90 loads/min)	Water at 37 °C	Cyclic loading in water did not significantly affect the fracture resistance. The load necessary to fracture the frameworks as delivered after machining was significantly higher than for the heat-treated and veneered specimens
[70] (2006)	Alumina (Procera Alumina) Y-TZP (Procera Zirconia)	Single molar crown	30–300 N	10,000 (1 Hz) in air	Thermal cycling 5–55 °C in water, 5000 cycles (1 cycle/min) + 10,000 pre-loading	There is a significant difference in the fracture mode, suggesting that zirconia core is stronger than the alumina core. Crowns made with zirconia cores have significantly higher fracture strength after pre-loading
[71] (2006)	Y-TZP (Lava) Ra = 0.21 ± 0.3 μm	Disk Ø 13 mm × 1.48–1.54 mm	500, 700, and 800 N 80 N	2000 (1.8–2.7 Hz) 10^4 and 10^5 (2.8 Hz)	Air or water at 37 °C Air at 37 °C	No significant difference was identified in the bi-axial flexure strength of the simulated masticatory loading regimes and the control specimens loaded dry or wet. However, the extended loading regime to 10^5 cycles resulted in a significant reduction in the Weibull moduli of the Y-TZP specimens compared with the control
[72] (2007)	Y-TZP (Cercon) Empress 2 In-Ceram Zirconia	Bar 2 mm × 4 mm × 50 mm	0–500 MPa 0–160 MPa 0–300 MPa	Determination of number of cycles to failure (10 Hz)	Water at 25 °C	Y-TZP was found to be particularly suitable for the preparation of posterior all-ceramic bridges due to its high initial mechanical strength. This allows for the preparation of 3-, 4- and 5-unit posterior bridges with lifetime comparable to that achieved with metal-based restorations (>20 years) with advantageous esthetics and biocompatibility of all-ceramic prosthesis, if the diameter of the bridge connector is properly designed

[73] (2007)	Machinable leucite-reinforced ceramic (ProCAD) Alumina Y-TZP	Bar 2 mm × 2 mm × 15 mm	0.01—50 MPa/s	Dynamic fatigue	Water at 25 °C	Lifetime predictions after 10 years indicate a reduction of 50%, 36%, and 29% in strength for porcelain, alumina and Y-TZP, respectively
[74] (2007)	Y-TZP (Lava) Uncolored, FS1-FS7	Disk Ø 15 mm × 1.3 mm	50—90% of the mean flexural strength	Determination of number of cycles to failure (2 Hz)	Water at 37 °C	There was no significant difference in flexural strength of uncolored and colored Y-TZP ceramic. The fatigue limit at 5×10^5 cycles may be defined as lying between 60 and 65% of the stress to failure (600 N) and fatigue did not significantly affect the flexural strength of zirconia at 10,000 and 20,000 cycles

Table 7 Annual failure rates and survival of all-ceramic and metal-ceramic fixed dental prosthesis (FDP) [78]

Study	Year of publication	Materials	Total no. of FDPs	Mean follow-up time (years)	No. of failure	Total FDPs exposure time (years)	Estimated failure rate (per 100 FDP years)	Estimated survival after 5 years (in %)
Sailer et al.	2007	Zirconia	57	4.5	12	210	5.71	75.1
Raigrodsky et al.	2006	Zirconia	13	3	0	39	0	100
Tinschert et al.	2005	Zirconia	65	3.2	0	202	0	100
Wolhart et al.	2005	IPS e.max Press	36	4	0	120	0	100
Marquardt and Strub	2006	IPS Empress 2	31	4.2	6	129	4.65	79.3
Suarez et al.	2004	In-Ceram Zirconia	18	3	1	53	1.89	91
Olsson et al.	2003	In-Ceram Alumina	42	6.3	5	266	1.88	91
von Steyern et al.	2001	In-Ceram Alumina	20	5	2	95	2.1	90
De Backer et al.	2006	PFM	322	11.4	69	3671	1.88	91
Hochman et al.	2003	PFM	49	6.3	6	324	1.85	91.2
Walton	2002/2003	PFM	515	7.4	37	3363	1.1	94.6
Näpänkangas et al.	2002	PFM	204	7.6	7	1478	0.47	97.7

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