

THE INFLUENCE OF SANDBLASTING AND AGING ON Y-TZP CERAMIC DURING STATIC AND DYNAMIC LOADING

NIKOLAOS ZIOZIOS*, ELEANA KONTONASAKI**, NIKOLAOS KANTIRANIS***,
LAMBRINI PAPADOPOULOU***, YIANNIS PAPADOGIANNIS****, #PETROS KOIDIS*****

*Department of Fixed Prosthesis and Implant Prosthodontics, Faculty of Dentistry, Aristotle University of Thessaloniki, Greece

**Department of Fixed Prosthesis and Implant Prosthodontics, Faculty of Dentistry, Aristotle University of Thessaloniki, Greece

***Department of Mineralogy-Petrology-Economic Geology, Aristotle University of Thessaloniki, Greece

****Department of Operative Dentistry, Faculty of Dentistry, Aristotle University of Thessaloniki, Greece

*****Department of Fixed Prosthesis and Implant Prosthodontics, Faculty of Dentistry,
Aristotle University of Thessaloniki, Greece

#E-mail: pkoidis@dent.auth.gr

Submitted November 29, 2016; accepted January 12, 2017

Keywords: Yttria-stabilized zirconia, Aging, Young's modulus, Sandblasting, Dynamic loading

The purpose of this in vitro study was to investigate the mechanical response of zirconia ceramic during torsional, bending and vibrational loading, before and after aging and surface treatments. Three types of loading were imposed upon zirconia specimens, torsion, bending and vibration. The effects of aging, sandblasting and both on the shear modulus, Young's modulus, Poisson's ratio, storage modulus, loss modulus and $\tan\delta$ were investigated, while surface characterization was performed through Scanning Electron Microscopy (SEM) and X-Ray Diffraction Analysis (XRD). Sandblasting as well as the aging environment caused a raise of zirconia's shear modulus, Young's modulus and storage modulus. The most prominent increase, in these parameters was observed when both treatments were combined. Under the limitations of this in-vitro study it can be concluded that the proposed sandblasting and aging procedures do not impair the mechanical properties of zirconia ceramics, as long as the amount of monoclinic zirconia content is kept under low values, capable to induce the transformation toughening mechanism.

INTRODUCTION

The strong esthetic demands of dental patients have caused a turnover of material science towards materials that exhibit optimal esthetic results combined with increased strength. The major problem however of all-ceramic restorations is their low fracture resistance [1]. To this critical issue, zirconia ceramics were introduced as the optimal solution [2]. The introduction of ceramic restorations with higher fracture strength ($> 1000\text{MPa}$) and toughness ($6\text{-}10\text{MPa}/\text{m}^{1/2}$), compared with other all-ceramic materials, resulted in the wide spread of zirconia [3,4].

Zirconia is a polymorphic material existing in three different crystal structures: monoclinic, tetragonal and cubic. At room temperature pure zirconium is consisted of only monoclinic crystals. Above 1170°C the monoclinic crystals transform into the tetragonal phase and remain until temperature reaches 2370°C . At these conditions tetragonal grains transform into cubic until melting point 2680°C [5]. During cooling this phenomenon is reversed and accompanied by a voluminous crystal expansion of 3-5% during transformation from tetragonal to monoclinic structure ($t \rightarrow m$) [3]. The addition of stabilizers, such as yttria, into zirconia led to partially stabilized materials (PSZ) and later to fully stabilized (TZP) containing only tetragonal crystal structure [3]. External stress can

initiate the $t \rightarrow m$ transformation, along with the volume expansion [6]. This phenomenon leads to crack blunting, because the energy needed for the crack to propagate is dissipated in the transformation of zirconia crystals and in overcoming the volume expansion. This is the transformation toughening mechanism which enhances the toughness of the material. In particular the strain induced due to phase transformation around a crack tip generates microcracks opposing the opening of cracks, leading to greater resistance in crack propagation [2].

There are different kinds of stimulations that can initiate the transformation of zirconia crystals. However spontaneous transformation at rather low temperature in a humid environment is called aging or low-temperature degradation [7]. This phenomenon was firstly described by Kobayashi et al. 1981 [8].

According to Chevalier (Chevalier 1999) zirconia kinetics can be interpreted through the Mehl- Avrami-Johnson (MAJ) law, which suggests nucleation and growth. This law is an attempt to combine parameters like temperature, pressure and time with the percentage of zirconia grains transformation.

$$f = 1 - \exp[(-b \cdot t)^n] \quad (1)$$

where f is the transformation fraction, t is the time and b , n are constants. In order to predict zirconia's performance a great number of accelerated tests were

performed at temperatures higher than the average body temperature. Through the MAJ law, Chevalier [9] was able to calculate that “1 hour of autoclave treatment has theoretically the same effect as 3 to 4 years in vivo”. In addition, the need for advanced specifications for ISO standards of zirconia led Chevalier to recommend that zirconia-based biomaterials when treated in steam at 134°C, under 2 bars of pressure for 5 hours should not present monoclinic content more than 10%. In addition no strength degradation should be accepted after testing. Although the recent criticism of the theory behind extrapolating the low temperature degradation rate from accelerated aging tests [10] these tests are currently the only tests available for predicting of the lifetime of the zirconia-based components.

Surface treatment of zirconia restorations is a common practice, especially when combined with resin-based cements, in order to increase micromechanical retention. The surface treatments commonly used are sandblasting and grinding with or without heat treatment [11-13]. It seems that the induction of surface flaws through surface treatments may potentially deteriorate the longevity of a zirconia restoration. When surface treatments are induced upon zirconia, compressive stresses are introduced into the surface layers. The effect of surface treatments on the mechanical behavior of zirconia, according to a recent study [14] depends on the type and severity of the treatments.

In order to effectively use high strength ceramic materials in a variety of impact related applications, it is necessary to develop knowledge related to their mechanical behavior and failure modes under multi-axial dynamic loading conditions. Dynamic and static mechanical analyses are techniques employed to rheologically characterize a biomaterial [15]. Static mechanical analysis is the deformation generated in a material when constant stress is applied. As soon as the stress is removed the material enters the recovery phase. Static tests provide important information about properties like shear modulus, Young's modulus as well as the residual deformation of the specimen after shear and bending stresses are applied. The Young's modulus determines the stress-strain ratio, when the material is still in the region of elastic deformation [15]. The shear modulus also is defined as the resistance of a material in shear forces. These two parameters define a third one, the Poisson's ratio represents the specimen's resistance to elongation and shear. These are non-catastrophic deformation test giving information about shear modulus, Young's modulus and Poisson's ratio of the material [16]. In dynamic mechanical analysis, a sinusoidal stress is applied and the response of the material is measured a function of frequency. Dynamic tests are also used to evaluate rheological properties of materials, like storage modulus (G_1), loss modulus (G_2) and loss tangent ($\tan\delta$) [16]. The storage modulus is defined as the stress in phase with strain and measures the energy stored and

recovered per cycle. The loss modulus is the stress out of phase with strain in a sinusoidal deformation. In a perfect elastic behavior G_2 should approach zero and G_1 should be stable. On a molecular basis, it represents the absence or presence of any molecular or atomic adjustments, during deformation, which could result in energy dissipation. The loss tangent is a dimensionless parameter that measures the energy lost to energy stored in a cyclic deformation. Actually this parameter is used to determine macroscopic physical properties such as the damping of free vibrations as well as the attenuation of wave propagation [17].

The aim of this in vitro research was to estimate the effect of Chevalier's aging environment and of sandblasting on zirconia ceramic's behavior during static and dynamic loading. According to the null hypothesis of this study, Chevalier's aging environment and sandblasting when imposed separately and in combination upon TZP ceramic, should not cause any strength degradation during static and dynamic loading.

EXPERIMENTAL

In this study specimens were fabricated from blocks of yttria stabilized zirconia oxide with the aid of CAD/CAM technology (Zeno[®] Zr, Wieland, Phorzheim, Germany). The zirconia blocks according to the manufacturer consisted (in mole) of 94% zirconium oxide ($ZrO_2 + HfO_2$), 5% yttrium oxide (Y_2O_3), <1% aluminum oxide (Al_2O_3) and <1% other oxides. The specimens were fabricated with certain geometry for the dynamic and static mechanical analysis (length 18 mm and diameter 1.3 mm) [18] (Figure 1).

The apparatus used in this study was first described by Lakes [19]. This apparatus is capable of torsional and bending tests upon cylindrical specimens. The specimen has on the one end fixed a high intensity, samarium cobalt magnet generating torque of $2,33 \times 10^{-2}$ Nm/amp. The specimen with the magnet is placed in the center of a Helmholtz coil, whereas the torque generated is controlled by the electric current in the coil. Onto the

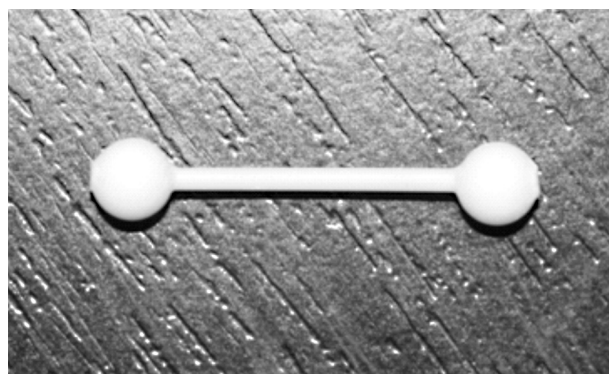


Figure 1. Specimen geometry as produced with CAD/CAM technology.

magnet, a thin cylindrical mirror (8.2 mm diameter and 0,635 mm thick) is cemented. A low power helium neon laser beam is fixed in front of the specimen, so as to reflect the spot on a calibrated chart at a distance of 944cm. Each deformation of the specimen is measured as the deflection of the laser spot on the chart. The magnet exerts an axial tensile stress to the specimen through its weight. There was no constraint on the specimen for either extension or torsion. The method used generates an axial deformation in the specimen, which is considered negligible for the torsional loads used. In addition the method is totally insensitive to axial deformations [20].

The specimens were divided into four groups according to the surface or aging treatment received (Table 1). Except for the control group, one group of specimens was sandblasted with Al_2O_3 particles according to the manufacturer's recommendations. In particular Al_2O_3 110 μ m particles were used under 2 bars of pressure for 10 seconds, from a distance of 50mm. After the sandblasting the specimens were ultrasonically cleaned (CP104 Standard CEIA Italy) for 10 minutes with no heating. The third group of specimens was aged according to Chevalier's recommendation [9]. In order to simulate Chevalier's aging environment a steam autoclave KavoKlave 2100 was used in continuous circles at 122° under pressure of 2 bars for 5 hours without drop of pressure and temperature (KavoDental, Biberach/Riss, Germany). The forth group was sandblasted and aged. All groups underwent static and dynamic loading.

Table 1. Groups of zirconia specimens according to surface treatment.

Group	Treatment	No of specimens
I	Control(C)	10
II	Aging(A)	10
III	Sandblasting(S)	10
IV	Sandblasting and aging (SA)	10

Static loading

Torsion

Static loading of the specimens was conducted, with the use of shear and bending stresses. Firstly shear stress was exerted to the specimens, by using torsional load. The magnetic field created inside the Helmholtz coil, forced the magnet to turn creating a torque to the specimen. The rotation of the magnet is given by

$$\varphi = X/2D, \quad (2)$$

where X is the displacement on the chart and D is the distance of the magnet from the chart [19]. Through a commutator electric current is supplied to the Helmholtz coil of magnitude 100, 200, 300, 400 mAmps for 10 seconds each. These measurements were made so as to check the credibility of the procedure.

The shear strain in a circular cylinder in torsion is

$$\gamma = \varphi \cdot R/L \quad (3)$$

where R is the radial distance from the centerline, L is the length of the specimen and φ is the radial deflection of the specimen. In cases of linearly elastic or linearly viscoelastic the distribution of the shear stress is given by [17]

$$\sigma = MR/\pi r^4/2 \quad (4)$$

It should be pointed out that the stresses exerted on the specimens are small, in higher stresses the outer layers of the specimen experience different stress and consequently the intrinsic material non-linearity is underestimated in the results.

In order for the shear moduli to be calculated, 10sec of torque is applied to the specimen and the angular displacement is recorded on the chart.

The shear modulus is given by $G = \sigma/\gamma$ (5)

$$G = 2TL/\pi R^4\varphi \quad (6)$$

In the above equation T is the torque applied given by

$$T = Mc \quad (7)$$

where M is the torque of the magnet $M=2.33 \times 10^{-2}$ Nm/amp and c is the electric current [21].

The strain of the specimens is within the linear elastic behavior of the material, because after the release of the stress no permanent deformation remains.

Bending

After the shear stress is applied, with the same device bending stresses are exerted on the specimen. The only difference is the orientation of the Helmholtz coil. The coils are orientated in a position of 90° degrees compared to the former position used for the shear test. The stress applied for 10 sec is a cantilever bending stress. Again the deflection of the specimen is recorded as the angular deformation of the laser reflection on the chart. According to the following equation (Eq.8) by knowing the shear stress and the Young's modulus of a material the Poisson's ratio can be also calculated [22].

$$G = E / 2(\nu + 1) \quad (8)$$

The magnetic field created by the current between the coils, causes the specimen to bend. The bending angle (θ) is calculated from Equation 9

$$\theta = PL/EI \quad (9)$$

where P is the bending force

$$P = Tc, \quad (10)$$

L is the length of the specimen, E is the Young's modulus and I is the inertia of the cylindrical specimen given by

$$I = \pi d^4 / 64 \quad (11)$$

By combining Equation 10, 11 and 9 the Young's modulus is calculated by

$$E = 64TLc / \pi \theta d^4 \quad (12)$$

Dynamic loading

Vibration

Vibration is a dynamic experiment where both the stress exerted on the specimen and the strain created, vary sinusoidally, as long as the viscoelastic behavior of the specimen is linear. With this experiment the storage modulus G_1 , the loss modulus G_2 and the complex modulus G^* are calculated. The storage modulus is the part of the complex modulus in phase with the strain. The loss modulus is referred as the part of the complex modulus 90° out of phase and expresses the loss of energy [16]. The loss modulus in most cases of stiff solids is very small compared to the storage modulus. Another parameter calculated is the loss tangent ($\tan\delta$), which is an expression of the phase angle between stress and strain sinusoids.

The dynamic experiment is conducted with the same device used in the torsion test. The only differentiation is the alternating electric current supplied to the Helmholtz coil. The poles of the magnetic field change constantly applying a steady-state torsional vibration to the specimen by frequencies ranging from 1 to 180 Hz. A maximum displacement or amplitude is measured. The resonance frequency v_0 corresponding to this amplitude is also measured. In addition the frequencies corresponding to the half of the maximum amplitude v_1 , v_2 and the resonance full width are calculated. These parameters are integrated in the calculation of $\tan\delta$ [20].

$$\tan\delta = (1/\sqrt{3}) \cdot (\Delta v / v_0) \quad (14)$$

The storage modulus is calculated from [20]

$$v_0 = (1/2\pi) \sqrt{G_1 \pi r^4 / 2LI} \quad (15)$$

where L is the specimen's length r is the radius and I is the moment of inertia of the magnet and the specimen together. The inertia of the magnet is much greater compared to the specimen's, for this reason $I \approx I_{\text{magnet}}$.

Surface characterization

In this experiment for surface characterization a scanning electron microscopy (Quanta 2000 FEI)

combined with X-ray energy dispersion spectrometer EDAX was used. To prevent any charge accumulation all specimens were carbon-coated (200Å).

The percentage of crystals transformed as a result of sandblasting, aging, mechanical testing was determined using X-ray diffraction analysis. The data were collected with a diffractometer (Phillips PW1710) using Ni-filtered Cu-K α radiation. All specimens were scanned with diffraction spectra ranging between $3-53^\circ$ 2θ at a scan speed of $1.2^\circ/\text{min}$. The Garvie-Nicholson [23] equation was used to calculate the percentage of monoclinic crystal phase of each study group [23]:

$$X_m = \frac{Im(\bar{1}11) + Im(111)}{Im(\bar{1}11) + Im(111) + It(101)} \quad (16)$$

Statistical analysis

A pilot study was conducted in order to estimate the number of specimens needed for statistical significance (power analysis, G*Power Software). For each group 10 specimens of zirconia were finally tested as shown in Table 1.

However the reliability of the method was also estimated by repeating the torsion, bending and vibration experiment for each specimen of the pilot study. The Intraclass Correlation Coefficient (ICC) was found >0.9 , which indicates that the method is reliable and only one measurement for each specimen is necessary. The mean values, the minimum and maximum and standard deviation for each parameter were measured. Normality tests were conducted and also Levene's test homogeneity of variances. The effect of sandblasting, aging and both on the parameters studied were calculated using One-Way Analysis and Tuckey's b tests multiple comparisons tests. For all statistical tests PASW 18 software was used.

RESULTS

Static and dynamic measurements

Every specimen underwent static and dynamic loading. The test itself had no obvious effect on the mechanical properties on zirconia. This was verified by measuring all the above parameters twice for each specimen of the pilot study. In addition the tests were conducted with the same order, shear, bending, vibration and vice versa. The mean values and standard deviations

Table 2. Mean values and standard deviations of static and dynamic measurements of tested groups.

Group	Shear Modulus (E+10 N/m ²)	Young's Modulus (E+10 N/m ²)	Poisson's Ratio	Storage Modulus (E+10 N/m ²)	Loss Modulus (E+08 N/m ²)	$\tan\delta$
C	7.176 (± 0.422)	1.871 (± 0.086)	0.304 (± 0.043)	7.389 (± 0.464)	5.401 (± 1.493)	0.007 (± 0.002)
A	7.629 (± 0.408)	1.963 (± 0.142)	0.287 (± 0.063)	7.864 (± 0.477)	5.546 (± 1.798)	0.007 (± 0.002)
S	7.745 (± 0.308)	2.021 (± 0.101)	0.305 (± 0.055)	7.900 (± 0.296)	6.235 (± 1.804)	0.008 (± 0.002)
SA	7.969 (± 0.191)	2.130 (± 0.111)	0.340 (± 0.062)	8.156 (± 0.191)	5.854 (± 1.311)	0.007 (± 0.002)

of these parameters for each study group are presented in Table 2. From the results of the test, it seems that there is a gradual increase of the shear modulus, the Young's modulus and storage modulus reaching their highest values for the SA specimens. These findings are graphically presented in Figure 2.

One-way Anova analysis (Table 3) revealed a statistical significant change of the shear modulus, the

Young's modulus and storage modulus when zirconia underwent surface treatment and aging. From the results it seems that the aging process according to Chevalier's recommendations had a strengthening effect on zirconia ceramic. The same effect is established on tested zirconia ceramic after the performed sandblasting. When the two processes were combined the strengthening effect was more pronounced. For the shear modulus and the storage modulus all groups differ statistically from the control group but not from each other.

Surface characterization

Scanning electron microscopy

As no major differences are observed between the non-tested (Fig. 3a) and the tested specimen (Fig. 3b), it seems that the test itself had no impact on the surface morphology of zirconia specimens. From the rest microphotographs it is obvious that the surface of the sandblasted (Fig. 3c) as well as the aged and sandblasted specimens (Fig. 3e) is characterized by imperfections and irregularities.

XRD analysis

Figure 4 demonstrates the XRD patterns from which the presence of monoclinic crystal phase in all experimental groups can be observed. The control group contains no monoclinic crystals.

The aging environment suggested by Chevalier [9] resulted in an increase of monoclinic phase fraction up to 4%. A similar behavior was observed after sandblasting zirconia with Al_2O_3 . The monoclinic fraction reached 5% in this study group. However when both treatments were combined the percentage of monoclinic phase reached 8%, two times more than the percentage of the aged group (Fig. 5).

DISCUSSION

In this *in vitro* study a Y-TZP ceramic underwent static and dynamic loading after aging and surface treatment. According to the null hypothesis, the aging process itself must not deteriorate the material mechanically. In addition sandblasting alone or in combination with aging must not affect the response of zirconia during static or dynamic loading. According to the findings of this research, the null hypothesis is rejected, as a gradual increase of three parameters, the shear modulus, the Young's modulus and the storage modulus, after aging, sandblasting and both in comparison with the control group is demonstrated.

According to Sindel et al. [24] CAD/CAM machining of zirconia induces compressive stresses on the surface of the material. After machining of the

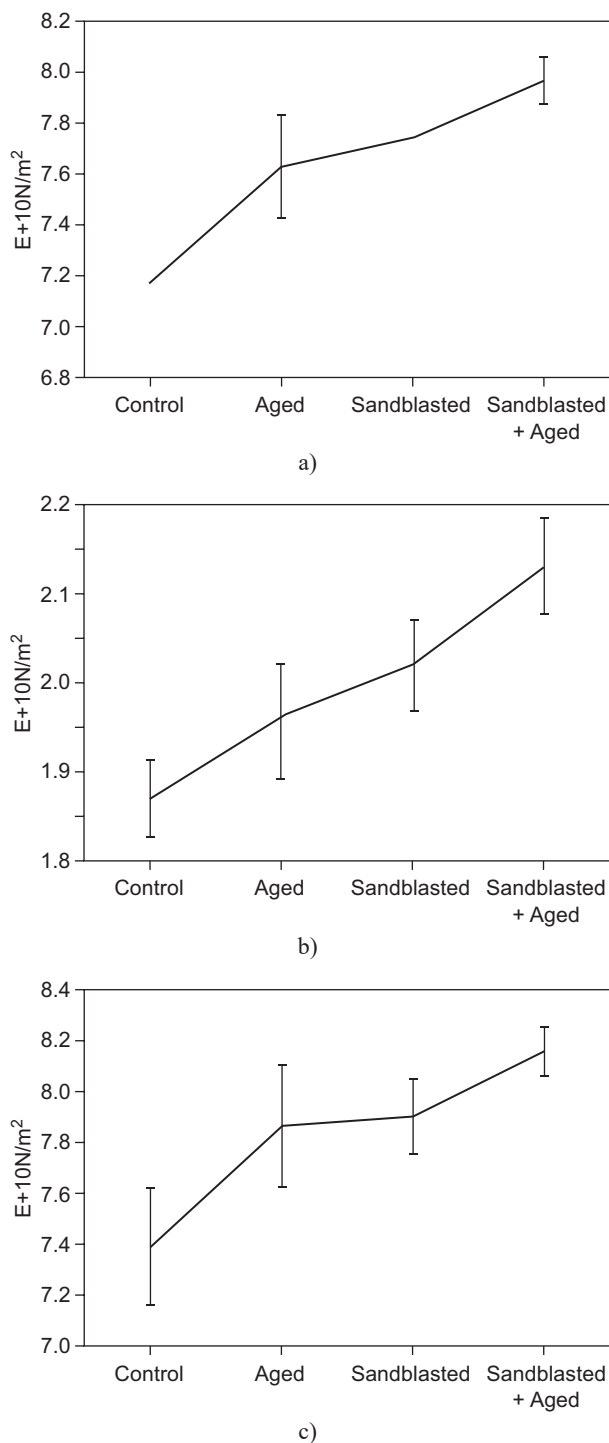
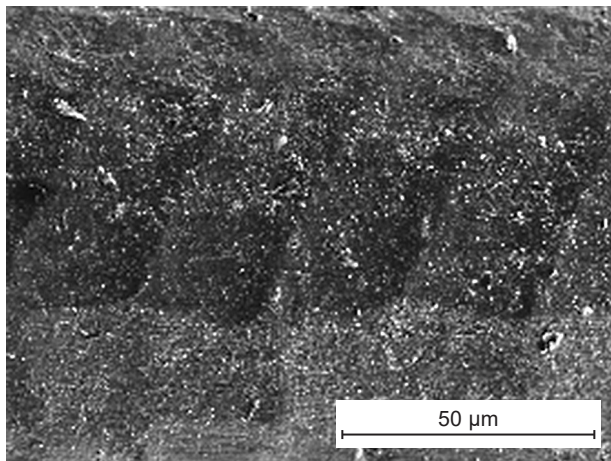


Figure 2. Graphs presenting the increase of Shear, Young's and Storage Modulus of control, aged, sandblasted and combined specimens.

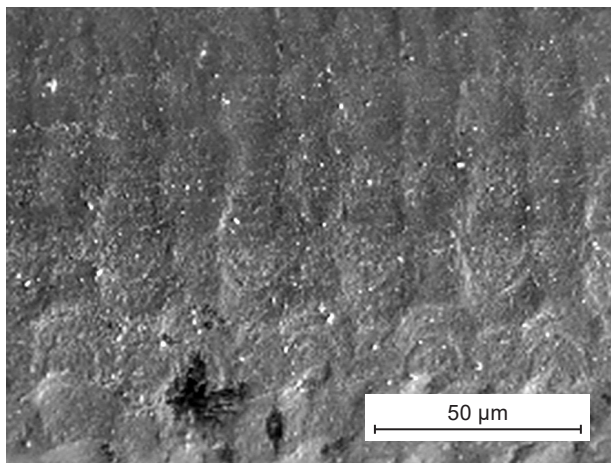
material, the sintering process that follows partially heals microflaws and cracks created during machining [25]. In addition, the microflaws initiate tetragonal to monoclinic transformation, whereas sintering facilitates the reverse transformation [13] demonstrated that CAD/

CAM machining may enhance the short term low – temperature-degradation resistance. In particular after prolonged aging in Chevalier’s aging environment the amount of monoclinic phase increased dramatically in comparison to the amount of the other study groups that underwent sandblasting and grinding. After 10 hours of aging, the percentage of monoclinic phase detected was above the limit of 10% set by Chevalier [9]. In the present study after CAD/CAM machining and sintering an XRD analysis followed for specimens from the control group. According to the results there was no obvious effect of CAD/CAM machining and sintering on the specimens and the percentage of monoclinic phase was 0%.

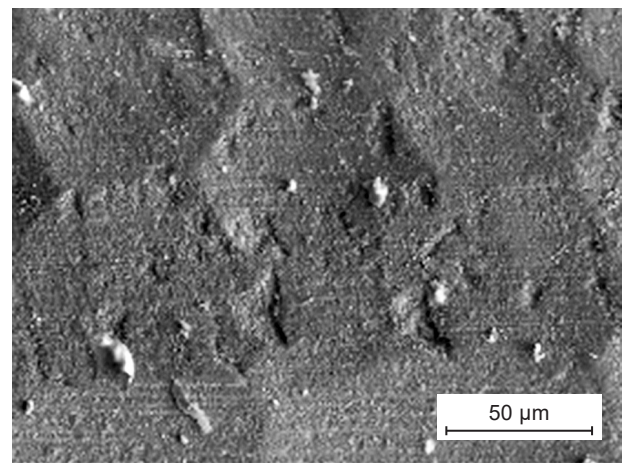
Aging of zirconia ceramics has been described by many researchers without however been able to render their results to clinical performance. Chevalier [9] managed to set conditions that can simulate an accelerated aging process for TZP ceramics. In the current study when these conditions were imposed upon the specimens, an increase of mechanical properties was observed. In particular after 5 hours of aging the



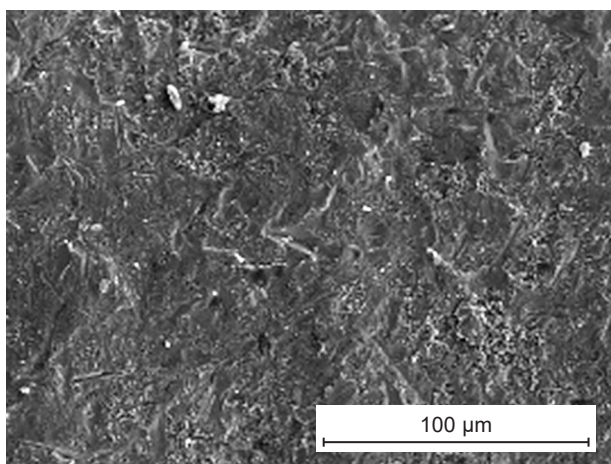
a)



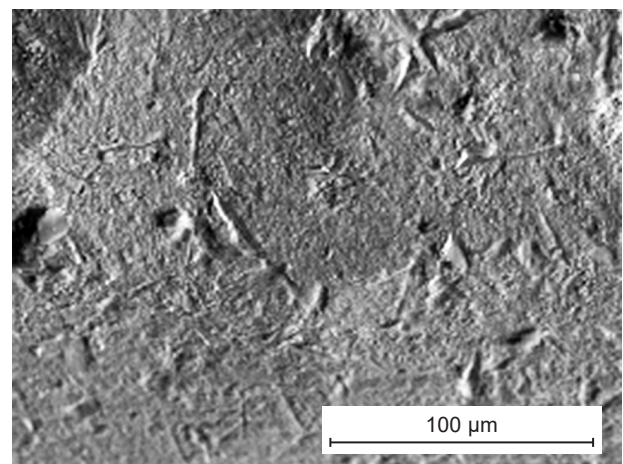
b)



d)



c)



e)

Figure 3. Representative SEM microphotographs of zirconia specimens: a) as-received specimen, b) control specimen after mechanical testing, c) sandblasted specimen after mechanical testing, d) aged specimen after mechanical testing, e) Sandblasted+aged specimen after mechanical testing.

shear modulus, the Young's modulus and the storage modulus, as measured through torsion, bending and vibration, increased compared to the control group. In addition according to the XRD analysis the percentage of monoclinic crystal phase was 4%. These results meet Chevalier's [9] recommendations that the percentage of monoclinic phase, after 5 hours of aging in this environment, must be below the safety percentage of 10%. According to Kim et al. [13] even after 8 hours of aging in 122 °C under 2 bars of pressure the amount of monoclinic phase was found below 10%. However as reported in the previous study, by increasing the hours of aging, a completely different XRD pattern was obtained, demonstrating a great increase of monoclinic phase that reached 55% after 20 hours of aging.

The mean values of three parameters, the shear modulus, the Young's modulus and storage modulus increased after 5 hours of aging. There is limited information about these properties of Y-TZP ceramics after aging. There are researches that have been mostly concentrated on the study of internal friction, as a measure of stiffness of zirconia ceramics under high temperature [26,27]. Internal friction seems to increase with increasing temperature until a maximum at about 500K or 227°C. After this critical point a major decrease of the parameter follows. At this critical temperature between 200 and 300°C, according to Swabb [28], aging of zirconia ceramics is most pronounced. Another parameter studied by these researchers is the Young's modulus. These studies as well as that of Chowdhury et al. [29] demonstrate a gradual decrease of Young's modulus with increasing temperature. This finding opposes the results of the present study that show an increase of the modulus after aging. However it must

be noted that in the study of Chowdhury et al. [29] the modulus decreased in specimens with concentration of monoclinic crystals over 10%. The specimens that underwent aging and bending test in the present study had a percentage of monoclinic phase of not more than 4%. Increase of strength with temperature was also noted by Kim et al. [13], with the critical point at 125°C and 12% monoclinic crystal phase.

Sandblasting of zirconia restorations is a common practice after annealing. The purpose of sandblasting is to enhance the bond strength with resin cements and veneering ceramics [30]. However it seems that sandblasting reduces the stability of tetragonal crystal phase [31]. According to Deville et al. [32] the effect of sandblasting on zirconia depends on the type of particles used, the size of the particles, the distance of the source of particles, the pressure used for sandblasting, as well as the duration of the procedure. Sandblasting induces compressive stresses on the surface of zirconia ceramics, creating microcracks increasing hydrothermal degradation resistance [13,34]. This is true when the microcracks of the material are restricted in a zone of plastic deformation. Inside this zone the crystal phase turnover inhibits the growth of microcracks enhancing the resistance of the material. There are plenty of studies about the effect of sandblasting on zirconia ceramics [11-13, 33-37] presenting controversial findings concerning the effect on zirconia's mechanical strength after sandblasting. Kim et al. [13] performed XRD analysis for every surface treatment they conducted on TZP ceramics. The grit-blasted surfaces consisted of negligible amount of monoclinic crystals. Karacoka & Yilmaz [12] noted that specimens that underwent sandblasting showed an increase of their flexural strength. Sato et al. [34] also

Table 3. One-Way Anova of static and dynamic measurements of tested groups (^ap < 0.001, ^bp = 0.001).

		Sum of squares	df	Mean square	F	Sig.
Shear modulus	Between groups	3.343	3	1.114	9.376	0.0000a
	Within groups	4.278	36	0.119		
	Total	7.621	39			
Young's modulus	Between groups	0.353	3	0.118	9.366	0.000a
	Within groups	0.452	36	0.013		
	Total	0.805	39			
Poisson	Between groups	0.015	3	0.005	1.576	0.212
	Within groups	0.114	36	0.003		
	Total	0.129	39			
Storage modulus	Between groups	3.067	3	1.022	7.208	0.001b
	Within groups	5.105	36	0.142		
	Total	8.172	39			
Loss modulus	Between groups	4.090	3	1.363	0.523	0.669
	Within groups	93.898	36	2.608		
	Total	97.988	39			
tanδ	Between groups	0.000	3	0.000	0.348	0.791
	Within groups	0.000	36	0.000		
	Total	0.000	39			

^ap < 0.001, ^bp = 0.001).

concluded that sandblasting enhances zirconia's flexural strength. Wang et al. [33] used two different particle sizes of Al_2O_3 , 50 μm and 110 μm for sandblasting. Using particles of 50 μm led to an increase of mechanical strength. However sandblasting with particles of 110 μm had the opposite effect. It should be noted that in the present study, after sandblasting, according to the XRD analysis the percentage of monoclinic phase was 5%. In the studies mentioned earlier, the percentage of monoclinic phase after sandblasting ranged from 7.36% up until 17.1%. Comparing the above literature, it seems that in the present study the sandblasting procedure was gentler because of the duration and the distance of the source from the specimen.

According to the results of the present study, all the sandblasted specimens demonstrated an increase of shear modulus, Young's modulus and storage modulus compared to the control group. After sandblasting of the cylindrical specimens, the radius of each specimen was measured and compared to the one measured before the

surface treatment. By this way, it was verified that the increase of the parameters measured is not due to radius reduction. There are no available data in the literature about the effect of sandblasting on the parameters measured in this study. However the results of this study are in accordance with part of the existing literature, demonstrating an increase of stiffness for zirconia ceramics after sandblasting, along with an increase in the percentage of the monoclinic phase [11,12,14,34]. There are also studies supporting that sandblasting enhances a strength degradation of zirconia, by accelerating the crack growth beyond the zone of plastic deformation [35]. Kosmac et al. [14] have reported that when the microflaws created by surface treatment do not outrange the zone of plastic deformation the phase transition is in favor of the material's strength. Whereas if the cracks created are greater than this zone, the increase of monoclinic phase has a detrimental effect on zirconia's strength.

In this *in vitro* research, the combination of sandblasting and aging of zirconia and their effect upon static and dynamic loading was studied. Therefore after sandblasting, a group of specimens underwent aging according to Chevalier's recommendations [9]. The combination of treatments on zirconia ceramics according to the literature seems to deteriorate the mechanical strength of the material [11]. This phenomenon can be attributed to two different mechanisms. According to the first one, sandblasting induces compressive stresses on the surface of the material. These compressive stresses are caused by sandblasting in the zone of plastic deformation of the surface. When temperature and pressure act upon this surface, these stresses are released [32] and the mechanical performance is decreased. The second mechanism is the reverse phase transformation, from monoclinic to tetragonal. Sato et al. [34] have noted that by increasing the monoclinic content, the flexural strength also increases and vice versa. In this study the specimens were firstly sandblasted and then aged. Even after both treatments an increase of the shear modulus, Young's modulus and storage modulus was observed. This increase of stiffness was accompanied by an increase of the monoclinic content up to 8%. In this case the percentage is still lower than the 10% that Chevalier [9] considers as a safety limit for zirconia ceramics. These results seem to oppose the data from the literature. However according to Guazzato et al [11] and Deville et al. [32] the inversion of the crystal transformation and the strength deterioration greatly depend on the length of cracks and microflaws created during sandblasting. Deville et al. [32] identified a greater crystal change from monoclinic to tetragonal around the longest crack paths. Chevalier et al. [38] have studied this phenomenon, by creating surface flaws on zirconia specimens using Vicker's indentation test. Again the phenomenon was concentrated around the longer crack paths. It is obvious that residual stresses affect greatly the performance of

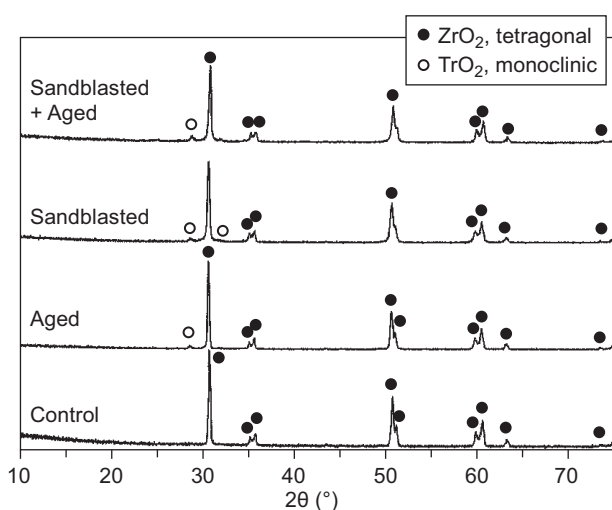


Figure 4. XRD patterns of control, aged, sandblasted and sandblasted + aged specimens after mechanical testing.

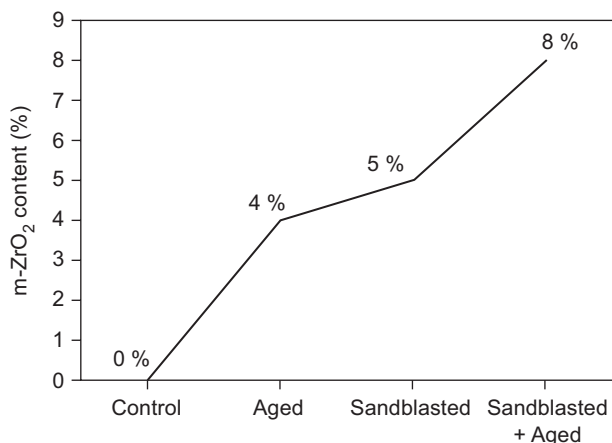


Figure 5. Graph showing the gradual increase of the monoclinic phase (%) after the different treatments applied.

zirconia ceramics. According to Johnson et al. [39] the amount of residual stresses created upon a surface depend on the severity of the surface treatment. Due to the gentle sandblasting, probably the length of the cracks created was not longer than that of the plastic deformation zone. All the amount of energy provided by temperature and pressure was used for crystal phase transformation in favor of the monoclinic content. In order for the cracks to propagate and to deteriorate the material mechanically, it is possible that an extra amount of energy should be provided. It should be noted that in every specimen the static and dynamic tests lasted few seconds and also no specimen underwent the test twice. The conclusions drawn may be valid for immediate loading of Y-TZP ceramics but not for long-term cyclic loading. Kim et al. [13] and Zhang & [40] have noted that microcracks created by surface treatment of zirconia, may propagate and cause reduction of mechanical strength under cyclic loading. For this reason further investigation is required about the long-term performance of zirconia under cyclic loading.

CONCLUSIONS

According to the results of this study, the static and dynamic loadings applied do not affect Y-TZP ceramics morphologically or mechanically. The aging environment, recommended from Chevalier [9] caused an increase of the shear modulus, Young's modulus and storage modulus. The same effect on Y-TZP was noted after sandblasting, as well as when both treatments were combined. This increase of stiffness was accompanied by a gradual increase of the monoclinic content. The reasons that aging of zirconia after sandblasting strengthened further the material can be attributed to the increased amount of the monoclinic phase that was below the suggested critical limit of 10% , the lack of the reversion of the *m-t* transformation and the possible presence of processing cracks that did not extend beyond the zone of plastic deformation.

REFERENCES

- Denry L.I. (1996): Recent advances in ceramics for dentistry. *Critical Reviews in Oral Biology & Medicine*, 7, 134-143.
- Garvie R.C., Hannik R.H., Pascoe R.T. (1975): Ceramic steel? *Nature*, 258, 703-704. doi:10.1038/258703a0
- Piconi C., Maccauro G. (1999): Zirconia as a ceramic biomaterial. *Biomaterials*, 20, 1-25. doi: 10.1016/S0142-9612(98)00010-6
- Koutayas S.O., Vagkopoulou T., Pelekanos S., Koidis P., Strub JR. (2009): Zirconia in dentistry: part 2. Evidence-based clinical breakthrough. *European Journal of Esthetic Dentistry*, 4, 348-380.
- Subbarao E.C. (1981): Zirconia an overview. *Advanced Ceramics*, 3, 1-24.
- Hannik R.J.H., Kelly P.M., Mudle B.C. (2000): Transformation toughening in Zirconia-containing ceramics. *Journal of the American Ceramic Society*, 83, 461-487. doi: 10.1111/j.1151-2916.2000.tb01221.x
- Sato T., Shimada M. (1985): Transformation of Ceria-doped tetragonal Zirconia polycrystals by annealing in water. *American Ceramic Society Bulletin*, 64, 1382-1384.
- Kobayashi K., Kuwajima H., Masaki T. (1981): Phase change and mechanical properties of ZrO_2 - Y_2O_3 solid electrolyte after ageing. *Solid State Ionics*, 3, 489-492. doi: 10.1016/0167-2738(81)90138-7
- Chevalier J. (1999): Low temperature aging of Y-TZP ceramics. *Journal of the American Ceramic Society*, 82, 2150-2154. doi: 10.1111/j.1151-2916.1999.tb02055.x
- Lughi V., Sergo V. (2010): Low temperature degradation -aging- of zirconia: A critical review of the relevant aspects in dentistry. *Dental Materials*, 26, 807-820. doi: 10.1016/j.dental.2010.04.006
- Guazzato M., Quach L., Albakry M., Swain M.V. (2005): Influence of surface and heat treatments on the flexural strength of Y-TZP dental ceramic. *Journal of Dentistry*, 33, 9-18. doi: 10.1016/j.jdent.2004.07.001
- Karakoca S., Yilmaz H. (2009): Influence of surface treatments on surface roughness, phase transformation, and biaxial flexural strength of Y-TZP ceramics. *Journal of Biomedical Materials Research Part B: Applied Biomaterials*, 91, 930-933. doi: 10.1002/jbm.b.31477
- Kim J.W., Covell N.S., Guess P.C., Rekow E.D., Zhang Y. (2010): Concerns of hydrothermal degradation in CAD/CAM Zirconia. *Journal of Dental Research*, 89, 91-95.
- Kosmac T., Oblak C., Jevnikar P., Funduk N., Marion L. (1999): The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. *Dental Materials*, 15, 426-433. doi: 10.1016/S0109-5641(99)00070-6
- Jones D.S. (1999): Dynamic mechanical analysis of polymeric systems of pharmaceutical and biomedical significance. *International Journal of Pharmaceutics*, 179, 167-178. doi: 10.1016/S0378-5173(98)00337-8
- Papadogiannis D., Lakes B.D., Palaghias G., Papadogiannis Y. (2009): Creep and dynamic viscoelastic behavior of endodontic fiber-reinforced composite posts. *Journal of Prosthodontic Research*, 53, 185-192. doi: 10.1016/j.jpor.2009.07.001
- Ferry D.J. (1980). *Viscoelastic properties of polymers*. 3rd ed. John Wiley and Sons.
- Papadogiannis Y., Boyer D.B., Lakes R.S. (1984): Creep of conventional and microfilled dental composites. *Journal of Biomedical Materials Research*, 18, 15-24. doi: 10.1002/jbm.820180104
- Lakes R.S. (1983): Size effects and micromechanics of a porous solid. *Journal of Materials Science*, 18, 2572-2580. doi: 10.1007/BF00547573
- Papadogiannis Y., Boyer D.B., Lakes R.S. (1987): Creep of amalgam at low stresses. *Journal of Dental Research*, 66, 1569-1575.
- Gere J.M., Timoshenko S.P. (1990). Torsion. In: Craig R. (ed): *Mechanics of materials*. 3rd ed. PWS-KENT Publishing Company. pp. 159-219.
- Darvell B. (2002). *Materials science for dentistry*. 7th ed. B.W. Darvell.
- Garvie R.C., Nicholson P.S. (1972): Phase analysis in Zirconia systems. *Journal of the American Ceramic Society*, 55, 302-305. doi:10.1111/j.1151-2916.1972.tb11290.x

24. Sindel J., Petschelt A., Grellner F., Dierken C., Greil P. (1998): Evaluation of subsurface damage in CAD/CAM machined dental ceramics. *Journal of Materials Science: Materials in Medicine*, 9, 291-295. doi: 10.1023/A: 1008812929476
25. Raigrodski A.J. (2004): Contemporary all ceramic fixed partial dentures: a review. *Dental Clinics of North America*, 48, 531-544.
26. Lakki A., Herzog R., Weller M., Reetz C., Görke O., Kilo M., Borchardt G. (2000): Mechanical loss, creep, diffusion and ionic conductivity of ZrO_2 -8 mol% Y_2O_3 polycrystals. *Journal of the European Ceramic Society*, 20, 285-296. doi: 10.1016/S0955-2219(99)00162-4
27. Roebben G., Basu B., Vleugels J., Van der Biest O. (2003): Transformation induced damping behaviour of Y-TZP ceramics. *Journal of the European Ceramic Society*, 23, 481-489. doi: 10.1016/S0955-2219(02)00101-2
28. Swabb J.J. (1991): Low temperature degradation of Y-TZP materials. *Journal of Materials Science*, 26, 6706-6714. doi: 10.1016/0955-2219(95)00035-S
29. Chowdhury S., Vohra Y.K., Lemons J.E., Ueno M., Ikeda J. (2006): Accelerating aging of Zirconia femoral head implants: change of surface structure and mechanical properties. *Journal of Biomedical Materials Research Part B: Applied Biomaterials*, 81B, 486-492. doi: 10.1002/jbm.b.30688
30. Kern M., Wegner S.M. (1998): Bonding to Zirconia ceramic: adhesion methods and their durability. *Dental Materials*, 81, 487-491. doi: 10.1016/S0109-5641(98)00011-6
31. Vagopoulou T., Koutayas S.O., Koidis P., Strub J.R. (2009): Zirconia in dentistry: Part 1. Discovering the nature of an upcoming bioceramic. *European Journal of Esthetic Dentistry*, 4, 2-23.
32. Deville S., Chevalier J., Gremillard L. (2006): Influence of surface finish and residual stresses on the aging sensitivity of biomedical grade Zirconia. *Biomaterials*, 27, 2186-2192. doi: 10.1016/j.biomaterials.2005.11.021
33. Wang H., Aboushelib M.N., Feilzer A.J. (2008): Strength influencing variables on CAD/CAM Zirconia frameworks. *Dental Materials Research*, 24, 633-638. doi: 10.1016/j.dental.2007.06.030
34. Sato H., Yamada K., Pezzotti G., Ban S. (2008): Mechanical properties of dental Zirconia ceramics changed with sand-blasting and heat treatment. *Dental Materials Journal*, 27, 408-414. doi: 10.4012/dmj.27.408
35. Zhang Y., Lawn B.R., Rekow E.D., Thompson V.P. (2004): Effect of sandblasting on the long term performance of dental ceramics. *Journal of Biomedical Materials Research Part B: Applied Biomaterials*, 71, 381-386. doi: 10.1002/jbm.b.30097
36. Papanagiotou H., Morgano S., Giordano A., Pober R. (2006): In vitro evaluation of low-temperature aging effects and finishing procedures on the flexural strength and structural stability of Y-TZP dental ceramics. *Journal of Prosthetic Dentistry*, 96, 154-164. doi: 10.1016/j.prosdent.2006.08.004
37. Curtis A.R., Wright A.J., Fleming G.J.P. (2006): The influence of surface modification techniques on the performance of a Y-TZP dental ceramic. *Journal of Dentistry*, 34, 195-206. doi: 10.1016/j.jdent.2005.06.006
38. Chevalier J., Ollangon C., Fantozzi G. (1996): Study of the residual stress field around Vickers indentation in a 3Y-TZP. *Journal of Materials Science*, 31, 2711-2717. doi: 10.1007/BF00687305
39. Johnson W.D., Evans A.G., Marshall D.B., James M.R. (1986): Residual stresses in machined ceramic surfaces. *Journal of the American Ceramic Society*, 69, 44-47. doi: 10.1111/j.1151-2916.1986.tb04691.x
40. Zhang Y., Lawn B.R. (2005): Fatigue sensitivity of Y-TZP to microscale sharp-contact flaws. *Journal of Biomedical Materials Research Part B: Applied Biomaterials*, 72, 388-392. doi: 10.1002/jbm.b.30174