Int. J. Appl. Ceram. Technol., 11 [3] 513–523 (2014)
DOI:10.1111/ijac.12118

International Journal of
Applied Ceramic TECHNOLOGY
Ceramic Product Development and Commercialization

Degradation and Mechanical Properties of Zirconia 3-Unit Fixed Dental Prostheses Machined on a CAD/CAM System

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The manufacturing of ceramics fixed dental prostheses (FDPs) is not the same as that of standardized samples and, therefore, the surface finishing are not necessarily the same in both cases. Standardized samples and FDPs were sintered, polished, hydrothermally aged in saliva, and submitted to mechanical tests. The results showed a difference in bending strength between machined (911.19 MPa) and polished FDPs (573.84 MPa). ANOVA statistical analysis did not show a difference in flexural strength between nondegraded (911.19 MPa) and degraded (871.94 MPa) FDPs. No differences were found in the bending strength and toughness of standardized samples and FDPs.

Introduction

Some works reported premature failure of femoral head ceramic prostheses inside the human body.[1] Biomedical zirconia was introduced to solve the problem of alumina brittleness and the potential failure of femoral ceramic prostheses,[2] but its use in dental implants has not been thoroughly investigated.

The ceramic systems used in dentistry must have adequate translucency to achieve good dental esthetics. Compared with alumina, zirconia-based ceramic used in fixed dental prostheses (FDPs) provide an adequate level of opacity, better esthetics, higher flexural

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strength, fracture toughness, and lower modulus of elasticity and hardness.[3] However, a limitation of zirconia application in FDP has been observed clinically: the degradation of zirconia when exposed to the oral environment, which may lead to a long-term decrease in strength and the possibility of failure[4–6] Although Lande & Evans[7] found no significant degradation, other researchers[8,9] found the opposite.

Degradation of the mechanical properties of zirconia is known as "hydrothermal aging" and occurs through spontaneous and progressive transformation of the metastable tetragonal phase to a monoclinic phase. This behavior has been studied at relatively low temperatures (65 to 400°C) in the presence of water vapor.[6,10,11] Factors that facilitate this transformation include particle size and mechanical processing such as grinding and polishing, which may weaken the material.[10] In an attempt to solve this problem, several studies[5–7,11] have been performed to analyze the fracture mechanisms and improve the performance of zirconia prostheses. The identification of fracture mechanisms, crack initiation site, crack speed, direction of crack growth, and the analysis of the influence of thermal gradients during processing, residual stresses and subcritical crack growth can help the development of new materials.

The zirconia-based ceramic composition and content of monoclinic phase influence hydrothermal degradation. During the FDPs machining process, many cracks and grooves are introduced in the surfaces of the part, which may contribute to hydrothermal degradation.

Matsui *et al.*[12] proposed a reaction between water molecules and the grain boundaries of YTZP (Yttria Tetragonal Zirconia Polycrystalline), which would result in the formation of nanometric $Y(OH)_3$ crystallites. The yttria available for formation of this oxide, according to the author, would be present at grain boundaries. After dissolution of yttria in the grain boundaries of the surface, some regions are replaced by a destabilized tetragonal phase that acts as a nucleation site for tetragonal-monoclinic transformation. The spontaneous transformation of tetragonal to monoclinic grains results in volumetric expansion (3–4%) of the crystal structure and the appearance of a residual stress in the neighboring grains with the initiation of microcracks. When the material is subjected to loading, the most superficial tetragonal grains are transformed to monoclinic phase and form microcracks. The interaction of microcracks generated by mechanical stress with those generated by water penetration leads to a gradual degradation of the material and its ultimate failure.

Available zirconia for dental prostheses machined by a CAD-CAM system is commercially supplied as presintered blocks whose mechanical properties are determined after sintering using standardized specimens. When the FDPs and standardized specimens for mechanical test are prepared from the same blocks, it is possible to observe that the later have a better surface finishing than FDPs. These surface morphological features may influence the mechanical properties and the hydrothermal degradation, as well the development of residual stresses in a thin surface layer.

The use of zirconia for dental prostheses is in its early phase, and the issue of hydrothermal aging needs additional studies. The purpose of the this study was to quantitate the mechanical properties of previously degraded CAD-CAM dental prostheses and to compare their mechanical properties (compression, bending and fatigue) with those of undegraded samples. In the present work, we studied the fracture toughness, analyzed the stress and determined the effect of artificial saliva on the degradation of FDP of zirconia stabilized with yttria. The FDPs were machined by CAD-CAM, and the mechanical properties were compared with those of standardized samples.

Materials and Methods

Commercial zirconia blocks for CAD-CAM systems are available presintered with low hardness. In the present work, two presintered commercial zirconia blocks from different manufacturer are used:

1. blocks Vita InCeram 2000 YZ-Cubes® (Vita-Zahnfabrik®, Bad Sackingen, Germany) composed of yttria stabilized tetragonal ZrO_2 (3 Y_2O_3 mol%).
2. blocks ProtMat ZrHP® (ProtMat Advanced Materials®, Volta Redonda, RJ, Brazil), composed of yttria stabilized tetragonal ZrO_2 (3 Y_2O_3 mol%).

Sample Preparation

Blocks of Vita 2000 YZ-Cubes® and ProtMat ZrHP® were purchased from the Brazilian market. The ZrHP® blocks were made with zirconia powder from Tosoh® (Shunan-shi, Yamaguchi, Japan). No information is available of Vita® blocks. Standardized samples with dimensions $43 \times 3 \times 4$ mm were cut from both blocks, with submitted to subsequent grinding, surface polishing, and sintering in a furnace without atmosphere control. The standardized samples were sintered in air for 2 h at 1530°C. The heating rate was 5°C/min, and the cooling rate was 10°C/min down to 400°C, and then the furnace was switched off. The total heat treatment time was 8.5 h.

The flexural strength of standardized samples was measured using a bending test.

The coefficient of thermal expansion (CTE) was determined with a Netszch 420 dilatometer. The CTE's in air were measured using an alumina standard with a heating rate of 20°C/min.

Zirconia 3-unit Fixed Dental Prosthesis Manufacturing

The CAD-CAM process of FDPs can introduce defects that may not present in standardized samples. For this reason, in this study, the mechanical properties of standardized samples and FDPs three-point bending test were compared.

In this work, 50 samples of 3-unit FDPs were machined with a CAD-CAM system from commercially ZrHP® presintered blocks (Fig. 1). For the FDP preparations, we used a model where the dental second premolar and second molar teeth were used as retainers to support a 3-unit FDP (Fig. 1). Teeth on the model were prepared with a circumferential shoulder of 1.5 mm, the space required for the zirconia substructure (1 mm) and the veneer (0.5 mm). The preparations were molded with silicone plus (Virtual Ivoclar Vivadent®, São Paulo, SP, Brazil). The casting material was removed after 6 min according to the manufacturer's recommendations.

Fig. 1. Dental prosthesis manufactured with a CAD-CAM system.

The 3-unit FDPs models were scanned (Dental Wings®, Montreal, Canada), and the FDPs samples were machined with the CEREC® system (Sirona Dental Systems GmbH, Bensheim, Germany) CAD-CAM system. The dental prostheses were sintered in air for 2 h at 1530°C. The heating rate was 5°C/min. The cooling rate was 10°C/min down to 400°C, and then the furnace was switched off. The total heat treatment time was 8.5 h.

After machining and sintering, the FDPs received a feldspathic ceramic veneering layer Cerabien CZR® (Noritake-Shinmachi, Nagoya, Japan) and were baked according to manufacturer's recommendation (930°C). To increase the strength between the zirconia framework and the CZR, a very fine thin layer of Shade Base Procelain® (SBP, Noritake-Shinmachi) mixed with Noritake Meister liquid was applied. After drying the SBP at the muffle entrance for 5 min, the FDP was baked from 600°C up to 930°C under vacuum and exposed to ambient atmosphere at 930°C for 4 min before cooling.

Hydrothermal Aging

To simulate degradation in the oral environment, one group of FDPs (n = 20) was subjected to accelerated aging. FDPs were immersed in artificial saliva at a pressure of 2 bar for 30 h in an autoclave at 135°C. The chemical composition of the artificial saliva was 0.96 g potassium chloride, 0.67 sodium chloride, 0.04 g magnesium chloride, 0.27 g potassium phosphate, 0.12 g of calcium chloride, 0.01 nipagin, 0.1 g nipasol, 8.0 g carboxyl methyl cellulose, 24 g sorbitol, and 1000 mL of purified water. The pH of the artificial saliva was adjusted to 2.5 by adding hydrochloric acid to distilled water.

After hydrothermal aging, the prostheses were dried and cemented in epoxy resin dental models for mechanical tests (Fig. 2). The cement used was zinc phosphate, and the manipulation was carried out according to the manufacturer's instructions. The same procedure was carried out with the undegraded prostheses.

Standardized Four-point Bending Testing

The flexural strength, σ_f, of each specimen submitted to a four-point bending testing was determined at room temperature according to ASTM C1161 (ASTM

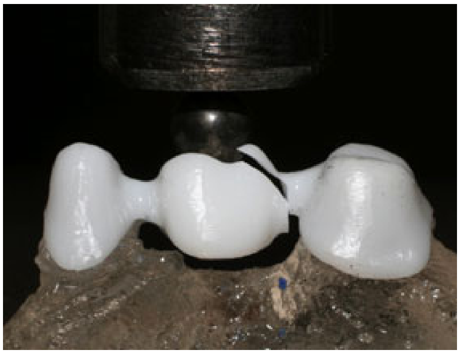

Fig. 2. Dental prosthesis set up for 3-point bending testing.

C1161-02 Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature). A custom four-point flexure fixture having an outer span (L) of 20 mm and an inner span of 10 mm was used. The width (w) and thickness (t) of each specimen were measured to determine the flexural strength according to the following equation:

$$\sigma_f = \frac{3PL}{4wt^2}, \tag{1}$$

where P is the maximum load recorded during testing.

An indentation hardness test (Vickers) was used to measure the apparent fracture toughness (K_{IC}) and the hardness of ten sintered samples. Polished specimens to a 1 μm diamond finish were indented at a load of P = 98N for 15 s.

To analyze the reliability of the products, the results of fracture toughness were subjected to Weibull statistics testing. The Weibull parameters m and σ_f were determined using correction factors according to the number of samples used. Test procedures for determining the Weibull modulus are specified in DIN EN 843-5 and DIN 51 110. If the measurements data show a high variation, the calculated Weibull modulus will be low; this reveals that flaws are clustered inconsistently and the measured strength will be generally weak and variable. Dental prostheses and other products made from ceramic of low Weibull modulus will exhibit low reliability and their strengths will be broadly distributed.

Bending and Compressive Testing

The standard way of calculation of the mechanical properties used in the present work consisted in carrying out a set of four-point bending tests of specimens cut out from presintered zirconia blocks. In case of FDPs, it is not possible to cut out standardized specimens with the required dimensions because the prostheses are too small. When the ceramic samples are smaller than the normalized ones, the material parameters measured in the mechanical test will exhibit higher strength values than those which the ceramic material really has. In the present work, we proposed a possible solution to this problem, which is not to cut the specimens from the FDPs, but to measure the zirconia mechanical properties directly from the whole FDP. Consequently, in the present work, we use the FDPs for bending test. Fuis and Navrat[13] proposed a similar solution, which used ceramic heads of total hip joint endoprosthesis for compression tests.

The mechanical bending tests of CAD-CAM FDPs were performed before (n = 20) and after degradation (n = 20). The group of undegraded samples was divided in two subgroups where half of the samples were polished with rubber (n = 10) before sintering and half received no surface polishing after machining and sintering (n = 10). The purpose of FDPs polishing surface was to study the influence of surface finishing on FDP strength under bending loading.

The three-point bending tests were carried out in an EMIC® (SJ Pinhais, PR, Brazil) mechanical testing machine (model DL10000) with a 5000 N load cell. In the prosthesis bending testing, a steel ball 5 mm in diameter, placed on the cusp, was used to increase the contact surface (Fig. 2). All loads were applied axially at the center of the pontic.

For calculations of the flexural strength, the FDPs were considered as a beam supported by two pins equidistant from the loading pins. The flexural strength (σ_T) was calculated using the equation

$$\sigma_T = M_f \cdot y/I, \tag{2}$$

where M_f is the maximum bending moment, y is the distance between the neutral line and the starting point of fracture, and I is the moment of inertia of this section. The bending moment M_f is calculated (equation b) by the product of a reaction force on the support pin (Q) by the lever arm (x), which is the distance between the support pin and the line of application of the force:

$$M_f = Q \cdot x \tag{3}$$

Assuming that the prosthesis is symmetrical with respect to the pontic, the reactions are equal in both support pins corresponding to half of the applied force. The modulus of rupture is calculated by the equation

$$\sigma_{T\max} = \frac{K16M_f}{\pi d^3} \tag{4}$$

where d is the diameter of the connector in the fracture section and the tensile stress (σ_T) is maximum when $y = d/2$. The value of K was obtained from the curves of Fig. 3.

After the bending test, the FDPs were cut and a new sample was designated as "unitary prosthesis." These new samples were bonded to the resin model for a second series mechanical testing and subjected to compression tests.

The mean values of all mechanical testing were compared by one-way ANOVA, followed by a *post hoc* Tukey test with the value of statistical significance set at the 0.05 level.

Fatigue Tests

FDPs prepared in the same conditions and dimensions of the substructures used in the three-point bending test were subjected to fatigue tests using a MTS 810 Materials Testing System® (MTS Inc., Eden Prairie, MN). Degraded (n = 10) and undegraded (n = 10) prostheses were used. Considering that there is no standard recommendation for FDP preparation or for FDP fatigue tests, an adaptation of the procedure from the ISO 14801 (ISO 14801: Dentistry – Fatigue test for endosseous dental implants) was used. The fatigue machine applied a 5 Hz cyclic load. A compressive sine wave at peak load of 550 N and minimum load of 400 N was used. The maximum load (550 N) was 80% of the mean of maximum force supported by the FDP, as determined in the 4-point bending tests.

A 5 mm diameter sphere was placed on the prosthesis cusp to increase the contact surface. This procedure increases the force distribution and reduces the stress concentration.

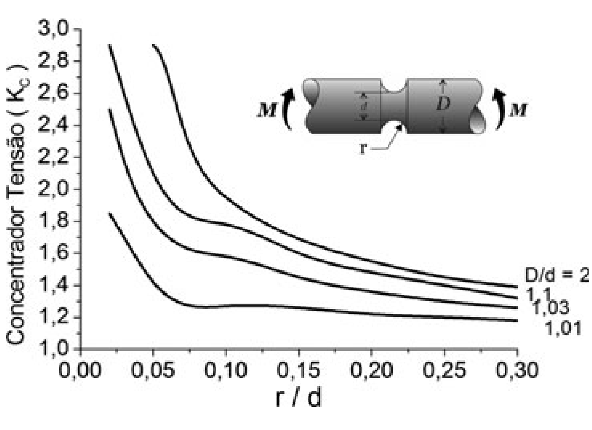

Fig. 3. Generalized diagram indicating the relationship between radii of curvature and stress concentration factor Kc.

Characterization of Zirconia Blocks and FDPs

The crystalline phases present in the presintered and sintered samples were identified by X-ray diffraction using a Cu-K line and a speed of 0.050 2s/count. For grain size measurement, the surfaces of sintered samples were ground, polished, thermally etched at 1400°C for 15 min, and observed by scanning electron microscopy (SEM-JEOL® model LSM-5800LV, Japan). An intercept procedure according to ASTM E112 and Image Pro® software was used to measure the grain size.

To examine the fracture surface morphology before and after the mechanical tests, some samples were coated with gold using a sputter-coating machine. The surfaces were characterized by SEM.

Results and Discussion

Physical and Mechanical Properties of Zirconia Blocks

Table I presents a summary of physical and mechanical properties of zirconia blocks determined according to standard procedures.

The data in Table I show that the mechanical properties of the two commercial blocks are similar. Values close to maximum density show that the sintering conditions suggested by the manufacturers are adequate for an almost complete elimination of the pores, leading to good mechanical properties of the products.

Figure 4 shows the failure probability of zirconia standardized samples for both zirconia blocks. Weibull modules are close to 10, and the flexural strength is 900 MPa.

Table I. Mechanical Properties of Zirconia Blocks

Properties	ProtMat ZrHP	VITA InCeram 2000 YZ
Density after sintering (g/cm^3)	6.05 ± 0.08	6.05 ± 0.06
E modulus (GPa) resonance method	205 ± 10	210 ± 11
Flexural Strength at 4 points (MPa)	920 ± 108	900 ± 117
Fracture toughness K_{IC} (MPa·m$^{1/2}$). Vickers	6.15 ± 0.25	5.9 ± 0.18
Grain size (nm)	400–500	380–500
Sintering temperature (°C)	1530	1530
Thermal expansion coefficient (α) $\times 10^{-6}/^{\circ}$C	10.5 ± 0.2	10.5 ± 0.2
Vickers hardness (HV_{2000})	1320 ± 70	1200 ± 68

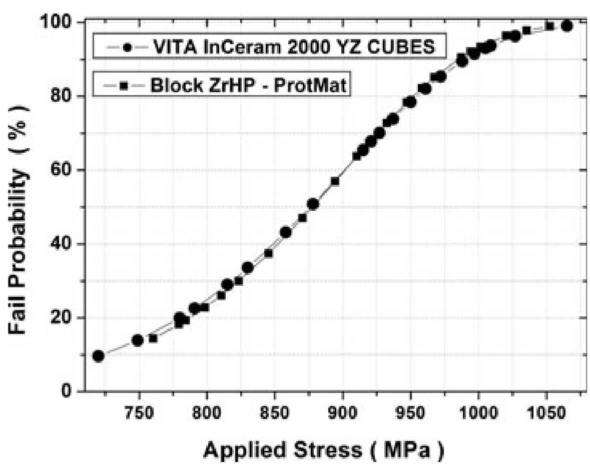

Fig. 4. Diagram of Weibull probability of failure of the sintered ZrO_2. Data from standard specimens as ASTM C1161.

Phase Composition: X-ray Diffraction Analysis

X-ray diffraction patterns were obtained from the surface of zirconia samples before and after sintering for 2 h at 1530°C. Figure 5 shows the X-ray diffractograms.

Tetragonal and monocyclic phases were identified both in the presintered and sintered standardized samples cut from ProtMat Zr HP blocks. The standardized samples before sintering showed 85.8% of the tetragonal phase and 10.5% of the monocyclic phase. The content of 10.5% of the monoclinic phase in the presintered samples is certainly related to the cutting process of the samples, which promoted phase transformation from tetragonal to monoclinic and induced residual stress. After sintering, the standardized samples showed 98.2% of the tetragonal phase and 1.8% of the monoclinic phase.

The sintering temperature (1530°C) was above the monoclinic to tetragonal transformation temperature, which is about 1170°C, and the metastable tetragonal phase remained after cooling to room temperature due to the presence of yttria. When zirconia is cut before fully sintering, a 20% shrinkage must be expected. The small content of monoclinic phase (1.8%) identified in the sintered samples is probably related to the residual thermal stresses developed during the cooling process and also by spontaneous transformation into the surface of the zirconia phase. The result is in agreement with the literature.[14,15]

FDPs Three-point Bending Testing

Table II shows the mean maximum force (*N*), flexural strength (MPa), and standard deviation for the 3-unit FDP. Statistical analysis was performed using One-way ANOVA (Bonferroni *post hoc*).

After bending tests, the fractured surface of each FDP was analyzed with a stereomicroscope Zeiss for measurement dimensions of the diameter (D) and length (L) of the connector in the fractured region (Table II). The measurements of the dimensions of the prosthesis were used to calculate the maximum fracture stress. Figures 6 and 8 show the local measures and schematic representation of the force applied to the reaction of support.

Table II shows that machined CAD-CAM FDPs (740.90 MPa) have lower flexural strength than standardized samples (900–920 MPa).

The mean flexural strengths of the CAD-CAM machined undegraded prostheses (911.2 ± 184.1 MPa) were slightly higher than the degraded group (871.9 ± 149.9 MPa), but the statistical analysis of 1-way ANOVA (Bonferroni, Tukey test and Scheffe'

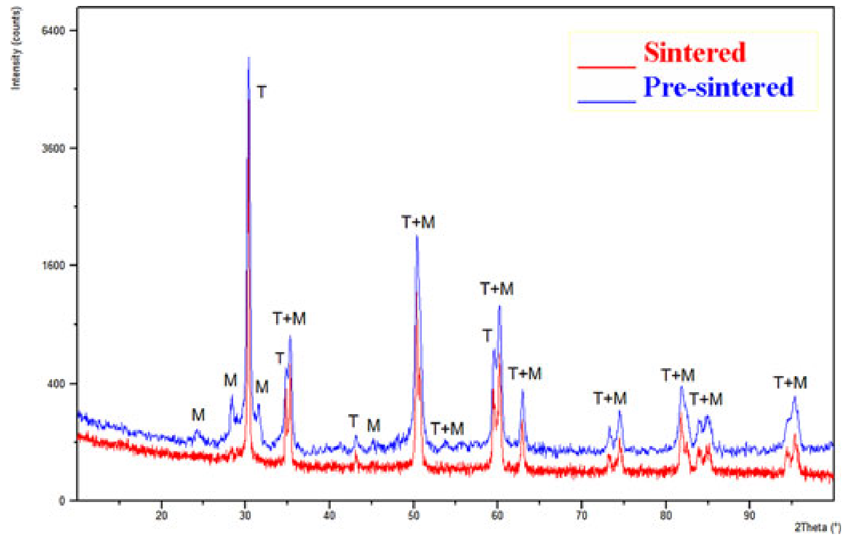

Fig. 5. X-ray diffraction patterns obtained from zirconia blocks before and after sintering at 1530°C. (T = tetragonal; M = monoclinic)

Table II. Mechanical Properties of 3-Unit FDP Bending Test and Percentual of Monoclinic Phase. Maximum Force (*F*), Connector Dental Prosthesis Dimensions (*D* and *L*), and Strength Under Three-Point Bending Test

FDPs	*F* (*N*)	*D* (mm)	*L* (mm)	Stress (MPa)
Machined	740.90 ± 98.25	4.11 ± 0.48	10.07 ± 1.00	911.2 ± 184.1
Degraded	790.08 ± 150.18	4.14 ± 0.65	9.43 ± 1.46	871.9 ± 149.9
Polished	592.66 ± 101.36	4.36 ± 0.12	9.68 ± 0.34	573.8 ± 139,1

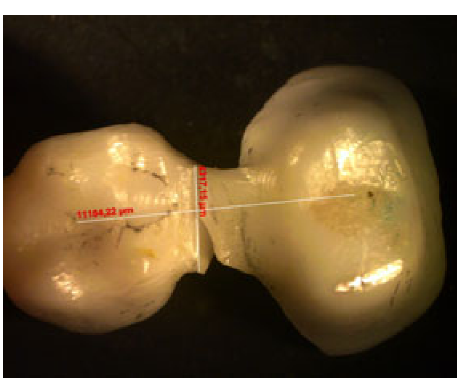

Fig. 6. Optical image shows the position of the dimension measurements of dental prosthesis after mechanical testing.

Test) of flexural strength values did not show a significant difference between undegraded (machined) and degraded. Two sample independent *t*-Test, at the 0.05 level, shows that the bending strength of machined (undegraded) and degraded group are not significantly different than the test difference ($P = 0.57$).

Figure 3 shows that very small radii of curvature should be avoided in order not to increase the stress concentration factor *K*. Applying the bending moment specific to the different measures of L/2 and the load applied to the fracture, we can calculate the value of applied stress required to fracture the prosthesis. The maximum stress and maximum diameter of the connector are shown in Table II. The results show that the flexural strength is inversely proportional to the diameter of the connector (Fig. 7). This result may be associated with the increased probability of finding a critical defect as the prosthesis size increases. Clinically, the occlusal contact among teeth and the gingival tissue define the limits of the connector dimensions.

Figure 8 shows the machined and sintered prostheses surface finishing. The machined surface shows flaws,

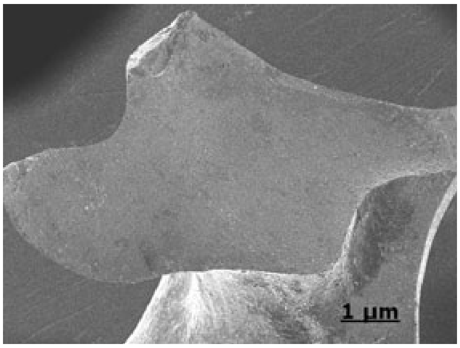

Fig. 7. SEM image after a bending test. Fracture surface of the connector zirconia CAD-CAM prosthesis.

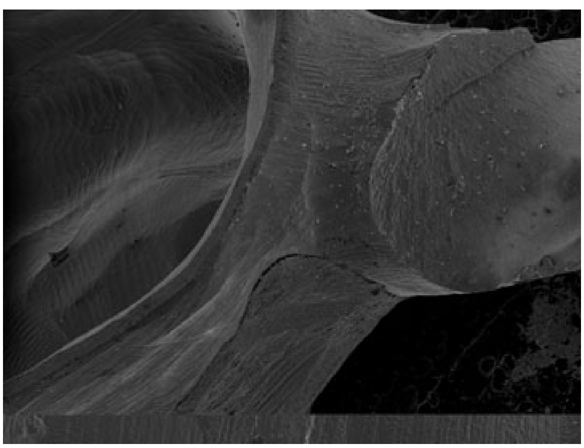

Fig. 8. CAD-CAM machined and sintered dental prosthesis surface finishing.

roughness, and grooves heritage from the grinding at the internal and external surface. As shown in failed FDP, the fracture region has a shape which can induce tensile stress concentration during clinical loading. Thus, it is important to minimize the size and number of grooves in these areas. The quality of surface finishing depends on the wear of cutting tools during the machining process.

Based only on the quality of the surface finish of the FDPs, it was expected that polished FDPs would present higher bending resistance than unpolished FDPs. However, the results of Table II show the opposite. Table II shows that unpolished PDFs have higher flexural strength (911.2 ± 184.1) than polished FDPs (573.8 ± 139.1). Statistical analysis of 1-way ANOVA (Bonferroni, Tukey test and Scheffe' Test) of flexural strength showed a significant difference between undegraded (machined) and polished samples. Two sample independent *t*-Test, at the 0.05 level, shows that the difference between FDPs machined and polished population means is significantly different than the test difference ($P < 0.05$).

With the polishing of the FDPs, the layer of monoclinic phase formed during CAD-CAM machining process increases. Polished FDPs have a higher percentage of monoclinic phase (14.5%) than standardized sintered samples. In this case, the better FDPs surface finishing did not improve their mechanical properties, showing that the compressive stress created by phase transformation is more important than surface finishing.

The literature results show that the influence of grinding on the flexural strength of zirconia ceramics is contradictory.[16–18] The toughness is related to the volume percentage of transformed zirconia, which in turn depends on the metastability of the tetragonal-monoclinic phase transformation, the grinding severity, and the locally developed temperatures. The strength increased after fine grinding with 25 mm grit size diamond wheel, whereas coarser grinding resulted in strength reduction.[18] During zirconia grinding, the tetragonal-monoclinic phase transformation induced compressive stresses and acted as stress concentrator, lowering the mean flexural strength.

The strength of ceramics is often determined by a mechanical test of standardized polished samples because the surface grooves introduced during machining and finishing operations influence the sample mechanical behavior, and it is well known that the ceramic strength can be increased by modifying machining procedures and reducing surface grooves or by transformation toughening. Crack tip shielding by transformation toughening has been shown to be a practical method of improving the mechanical reliability of ceramic materials in a number of important applications. For zirconia, transformation toughening occurs as a result of a stress-induced phase transformation in a ceramic matrix. Toughening is postulated to occur mainly as a result of an expansion in volume of tetragonal zirconia particles as they are transformed to the monoclinic form of zirconia. After the transformation, residual stresses surround each grain. These stresses act across the projected fracture plane and have to be nullified for the crack to propagate through the transformed zone.

The hypothesis that the CAD-CAM process changes the mechanical properties of zirconia is not confirmed. The mechanical properties of the standardized samples (ZrHP=920 ± 178 and Vita=900 ± 172 MPa) are not statistically different from those of machined and sintered FDPs (911.2 ± 184.2 MPa). Means comparison using Bonferroni Test, Scheffe' Test, and Tukey Test at the 0.05 level showed that the mechanical properties of the machined CAD-CAM FDPs and standard samples not significantly different ($P = 0.96$).

During CAD-CAM machining, grooves are created on the surface which reduce the flexural resistance. At the same time, cutting induces a phase transformation. As the X-ray diffraction result showed, after CAD-CAM machining, sintered FDP has a thin layer of monocyclic phase (3.5%), which has a compressive residual stresses. Thus, the reduction in mechanical strength due to surface grooves is minimized by the formation of the monoclinic phase. This could be the explanation for the fact that the flexural strength of machined FDP and standardized specimens was similar. Moreover, Swain and Hannink[19] showed that zirconia hand grinding induced the tetragonal to monoclinic transformation. They demonstrated that machine grinding increases the local temperature, which can exceed the monoclinic to tetragonal transformation temperature. As a result, the deep defects introduced by grinding are no longer counteracted by the transformation-induced compressive stresses and act as stress concentrators, lowering the mean flexural strength of the ceramic. The authors did not examine the strength response; however, because the strengthening mechanism of zirconia is mainly related to the tetragonal to monoclinic transformation, a larger mean flexural strength is anticipated when a larger amount of monoclinic phase is present on the surface of the ceramic.

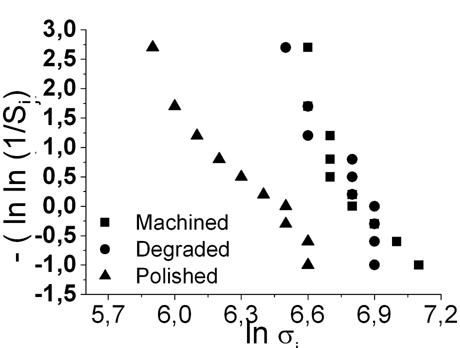

Fig. 9. Distribution of Weibull in the bending test.

Figure 9 shows the fracture toughness, statistical evaluation of the probability of failure, and Weibull modules (m) of FDPs. The m values for degraded, polished, and machined FDPs were $m = 5.3$, $m = 3.9$, and $m = 8.64$, respectively. The results show that FDPs has a higher scatter in the values of fracture toughness than standardized samples. This variation may be explained by differences in D, L, and surface defects introduced during the machining process. To verify the quality of fit of the equation to experimental data, a correlation coefficient (R^2) was calculated. The values were close to 1, indicating that the results are adequately described by the equation.

The statistical analysis (Tukey, Scheffe, Bonferroni and ANOVA) of values obtained in bending tests showed no statistical difference in flexural strength between degraded and undegraded machined FDPs.

Statistical analysis revealed that there is a significant difference in bending fracture toughness between the machined (without polishing) and polished FDPs. The strength reduction after polishing may be associated with the superficial monoclinic phase transformation induced during hand polishing. The higher standard deviation of the fracture stress for polished prostheses is attributed to the fact that during hand polishing is difficult to control the compressive force and obtain the same surface finishing quality in all samples. The present study suggests that dental prosthesis preparation changes the mechanical properties. Fine polishing may improve surface finishing but induces a compressively stressed layer and therefore decreases the mean flexural strength. Sandblasting may be useful to increase the strength of dental Y-TZP, as reported by Xu *et al.*[18]

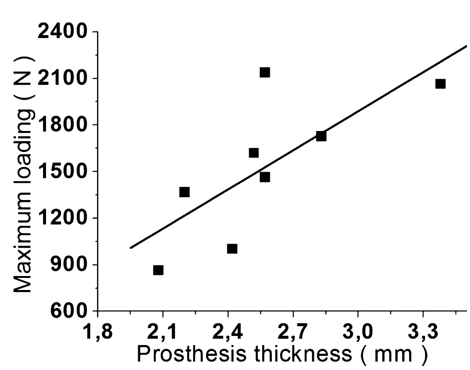

Fig. 10. Maximum applied force of dental prosthesis under compression testing.

Compression Testing

To investigate the influence of the thickness of the prosthesis on compression resistance, we used unitary prostheses. Figure 10 shows the maximum loads in compression fracture of the unitary prostheses. The thickness of the prostheses was measured by optical microscopy after the compression test. It can be seen in Fig. 10 that the maximum force (N) increases as the prosthesis thickness increases. The behavior among degraded and undegraded samples is the same. It can be said that the compression strength is directly proportional to the thickness of the sample. The linear regression analysis of experimental data gives us the equation: ($F = 722.09x - 370.91$, $R^2 = 0.504$). Based on this result, the minimum recommended thickness for this zirconia-based ceramic material for a loading of 500 N is 1.2 mm.

Comparing the maximum force applied in 3-unit FDP during three-point bending tests and unit FDP compression tests, it can be stated that the thickness of the prostheses and manufacturing defects are the main factors influencing the bending resistance.

The mechanical strength of polished FDP (573.8 ± 139.1 MPa) is smaller than standardized sample (ProtMat = 920 and Vita = 900 MPa).

Machined samples from the green zirconia blocks create defects and generate residual compressive stresses in the surface due to phase transformation. In the case of yttria stabilized zirconia, the residual compressive stress and phase transformation reduce crack growth. However, the sintering process promotes a new phase transformation, reducing the monoclinic layer at the surface. During cooling after sintering, only a small surface layer remains, forming spontaneously as a result from the lack of hydrostatic compression in this region. This layer can grow with a superficial treatment such as blasting or grinding, generating residual compressive stresses down to 10 to 100 μm.[19]

It can be observed in Fig. 8 that the machined FDP have surface defects which act as stress concentrators. In the case of zirconia FDPs, we have a competition between the subcritical grooves size and the compressive layer heritage from the tetragonal to monoclinic phase transformation. If the effect of surface defects is more important than that of the compressive residual stress layer induced by monoclinic transformation, the grooves act as critical defects and influences the toughness of yttria stabilized zirconia.[19] On the other hand, if the surface grooves have a small size, the influence of the residual stress layer is more important.

The influence of polishing on the aging sensitivity of zirconia is contradictory.[14,19] Rough polishing produces a residual compressive stress layer in the surface, which is beneficial for the hydrothermal aging resistance, while smooth polishing produces preferential transformation nucleation close to machine grooves, due to tensile residual stresses caused by elastic/plastic damage.[14] Fine polishing after grinding may remove the compressive layer of the monoclinic phase from the surface.[19]

Fatigue Tests

The fatigue resistance under a maximum load of 550 N and a minimum load of 400 N was low (806 10^3 cycles). The maximum applied force used in the fatigue tests was about 80% of the compression resistance of the machined prosthesis. Based on the results, it can be concluded that there was no statistical difference in fatigue resistance between the prosthesis subjected to accelerated degradation and undegraded in artificial saliva ($P < 0.05$). These results suggest that degradation occurs only at the sample surface as described before.[7] The surface morphology changes by degradation were not sufficient to reduce the fatigue mechanical strength of the prosthesis.

Surface Fracture Morphology

Lohbauer *et al.*[20] analyzed the fracture surface of dental zirconia. They observed the general direction of crack propagation, as evidenced by arrest lines, hackle, twist hackle, and wake hackle. The fracture clearly started from the incisal tip of the palatal zirconia framework and propagated toward the cervical region in this maxillary FDP. Their results were somewhat different, because most bridge and framework fractures start from the gingival side, often at a connector, and propagate toward the palatal regions due to bending stresses generated by occlusal loading on the pontic. In the present work, the prosthesis fracture under bending loading occured in three regions:

1. fracture in the connector: 36 prostheses in the present work
2. fracture in the retainer : 1 prosthesis in the present work

3. fracture close to the retainer: 3 prostheses in the present work

Fractographic analysis revealed that failure originated in surface defects. Analysis of the fracture surface after the compressive tests showed that the failure was predominant in the prosthesis connector. The fracture crack propagated obliquely to the FDPs length, connecting the gingival embrasure of the connector and the occlusal loading point on the retainer.

Macroscopic analysis of the prostheses after the bending tests revealed that the fracture began in the cervical region of the connector and propagated obliquely toward the occlusal surface. The prosthesis fracture starts by nucleation of microcracks in region with surface defects. The microcrack growth and the propagation were not straight. This behavior can be associated with phase transformation during crack propagation. Compression of the crack tip due to increased volume of neighboring grains induced by the crystal phase transformation, the presence of microstructural defects (pores and voids) and particle boundaries change the direction of crack propagation.

Conclusions

The results of this study show that:

1. The commercial zirconia blocks 2000 YZ CUBES (VITA) and ZrHP (ProtMat) have similar physical and mechanical properties.
2. CAD/CAM machining introduces grooves in the surface of dental prostheses, but does not significantly change the flexural mechanical properties.
3. The resistance to compression of the prostheses is directly proportional to the thickness.
4. Hydrothermal aging does not significantly influence the mechanical properties of dental prostheses under bending, compression, and fatigue tests.

Acknowledgments

This work was carried out with financial support from the Brazilian Agencies CNPq (Process 472449/2004-4, 400603/2004-7 and 500126/2003-6) and FAPERJ (Process E-26/151.970/2004). The authors thank Dr. Umberto de Luca (de Luca Clinic) for collaborating in the development of prosthetic components and CAD/CAM machining.

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