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Comparative study of three yttria-stabilized zirconia formulations in colored vs natural shades

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Abstract

Recently the attention in the field of fixed dental restorations has focused on structural ceramics. Yttria-stabilized tetragonal zirconia (Y-TZP) is proposed as an alternative to conventional ceramic-metal prostheses, since it merges excellent aesthetic quality with outstanding toughness. Before Y-TZP integral structures spread in dental practice, much needs to be studied as regards the mechanical response, the influence of color and of the manufacturing process, the occurrence of aging phenomena. This work aims at a comparative study of three Y-TZP commercial formulations in five shades; the variations introduced by polishing and their recovery after annealing are also addressed. Flexural strength and microhardness are investigated, basing on international standards. Significant differences between the groups and parts' reliability are evaluated through statistical data processing and Weibull analysis. Results show low flexural strength (500-800MPa), at least 45% inferior to technical specifications, and low Weibull modulus. Hardness is instead higher than expected (1500-1700 HV1). The main finding of the research is that the effect of color on mechanical properties is significant in many cases, hence esthetical requirements must be merged with mechanical ones. Y-TZP shows an extreme variability with manufacturing conditions, so nominal characteristics should be assumed with caution and higher reliability is still required.

Keywords: Y-TZP, dental prostheses, biomaterials, flexural strength, zirconia

INTRODUCTION

Over the past 20 years, increased demand by patients for dental restorations similar to natural teeth has led to the development of all-ceramic prostheses as an alternative to metal-ceramic ones [1]. Structural ceramics offer important advantages both in terms of aesthetics and biocompatibility, with respect to the traditional metal structures covered with ceramic dentine and enamel [1,2]. All-ceramic restorations are not affected by discoloration, show an extremely natural translucence and can be obtained in specific material formulations to match patients' natural tooth color [1-3]. Next to the above advantages, a major drawback of all-ceramic prostheses consists of the inherent brittleness of such materials [1].

Zirconium oxide represents a unique solution due to the possibility to exploit singular toughening mechanisms [3-6]. Zirconia shows three allotropic crystalline structures: it is monoclinic at room temperature up to 1443 K, beyond this temperature it becomes tetragonal and then cubic over 2643 K. While cooling, tetragonal to monoclinic transformation is accompanied by a volume expansion of about 3 to 4% [1]. This can be usefully exploited if the tetragonal form is retained at room temperature in a metastable state [5]. Under an external load, tetragonal stabilized zirconia exhibits a stress-induced martensitic phase transformation that generates compressive stresses at crack tip [1]. Crack propagation is thus obstructed and the material shows outstanding toughness (6-8MPa $m^{0.5}$) [4,7-9]. An efficient means to stabilize the tetragonal phase at room temperature is the addition yttria (Y_2O_3), typically in 3 mol%, obtaining yttrium cation-doped tetragonal zirconia polycrystals (3Y-TZP) [4].

Since the late eighties Y-TZP has been extensively used in orthopaedics, for example for the replacement of femoral heads, and many studies documented its complete biocompatibility and very low risk of citotoxicity, mutagenicity or flogistic reactions, even if compared to Titanium [6]. A sudden stop to Y-TZP applications was then due to an important number of hip prostheses' failures occurred in 2001, which scared the whole orthopaedic community [4,10]. The reason was ascribed to the well-known phenomenon of zirconia LTD, an ageing process fostered by the presence of water that causes surface degradation, microcracking and strength decay [4,10]. Actually further investigations lead to reconsider the above failures as a circumscribed event, limited in time and number, and likely to be dependent on the production process [10]. Today the effect of LTD on clinical failures and, more generally, on the variations of mechanical properties is still to be fully understood [4,10].

In the '90s, the use of Y-TZP was introduced in dentistry for endodontic dowels and implant abutments [11-13] and quickly spread as the most promising solution for cores in fixed prostheses [11,14,15,16]. 3Y-TZP is now adopted for the fabrication of dental crowns and fixed partial dentures [4]. The wide interest in Y-TZP for dental restorations based on its outstanding aesthetic and mechanical properties seems not to be shaded by concerns as to ageing problems [10,17]. Besides the above doubts on the exact consequences and occurrence of LTD [1], in the field of dental restorations the risk of ageing appears to be less critical than in orthopaedics, since zirconia dental prostheses are covered with veneering and luting materials that offer a shield from the oral environment [17]. In effect, many clinical studies attest the success of zirconia dental prostheses after mid-term follow-up [18,19]. Long-term clinical results are still under development, since these restorations are relatively recent [19]. Traditional metal-ceramic fixed partial dentures proved their reliability over a period of 20 to 30 years [20].

As to laboratory characterization, Y-TZP showed excellent biaxial flexural strength (1000-1200 MPa) and high fracture toughness with respect to other restorative ceramics [21,22]; in other studies uniaxial flexural strength was about 850 MPa with strong variations dependent on the production process, grain size, porosity [5]. Also, it should be considered that flexural strength and fracture toughness are conflicting characteristics and that stronger parts can be less reliable [17]. Many other studies address the mechanical response of Y-TZP [23,24,25], but most tests were conducted on benchmarks representative of the geometry of frameworks or abutments, instead of on standardized specimens. Thus, the results can be considered as a combination of the material's properties and the geometrical distribution of stresses.

All-ceramic restorations offer the chance of full integration of CAD/CAM approaches to dental technology, with great advantages in terms of process repetitiveness, reliability, accuracy and control. Y-TZP parts can be manufactured either by soft machining of presintered blanks, followed by sintering at high temperature, or by hard machining of fully sintered blocks [4,26]. The second solution does not require shrinkage calculation, but milling of fully sintered parts can cause tetragonal to monoclinic transformation on the surface, leading to compressive stresses and higher hardness, but also microcracks, surface damage and quicker LTD [4,26,27,28]. Soft machining of presintered parts is usually preferred. Presintered parts after milling can be colored to different shades to match the patient's complexion, they are then sintered and covered with feldspathic porcelain to obtain the final appearance [26]. Different shades proved to affect strength and, most significantly, microhardness in some studies [29-31], but the tests are still very few and not conclusive. After sintering, dental practitioners often resort to surface grinding or polishing to obtain dimensional adjustments (i.e. improve the marginal fit) or smoother surfaces. The eventual phase transformations due to the finishing step are still to be ascertained, some studies indicate an increase in the monoclinic phase percentage connected with a hardness increase and toughness decay, but literature data are controversial [1,32-34]. A sandblasting step is indicated by some authors as useful in recovering the negative effects of grinding [17], as well as annealing at 900–1000 °C for 1h, which induces the reverse transformation accompanied by the relaxation of compressive stresses and a decrease in strength [4].

To sum up, Y-TZP is certainly a promising material in the field of dental restorations and new approaches are being developed to enhance its characteristics [2]. Many studies attest that the material's properties are strongly dependent on the production process and that comprehensive reliable experimental figures are still required to deal with the described complex panorama [4,6,10,17]. Moreover, very little has been studied as to the effect of coloring, which can likely affect at least surface properties. The present work proposes a comparative study of Y-TZP, consistent with international standards and based on statistical data processing, aimed at contrasting different commercial formulations and colors and evaluating the variations introduced by polishing and their recovery after annealing.

MATERIALS AND METHODS

Three commercial formulations of Y-TZP are considered, from three producers, indicated with B, K and S. The nominal compositions as well as physical and mechanical characteristic from the datasheets are listed in Table I.

The soft machining solution is chosen on the basis of its wider spread in the dental community and higher robustness, related by many authors[4,26,27,28]. The materials are supplied to the dental technician as pre-sintered blocks to be milled, colored and sintered. To study the effect of color on mechanical properties, the experimental plan includes four different shades plus the natural white for each of the three materials: as-received white, marked with W, and four of the most common colors A1, B2, C3, D4 (Vita Scale[30]). The whole production process, illustrated in Figure 1, can be divided into two phases. In the first, operated by the producer, Y-TZP powders are compacted and pre-sintered at about 1000°C obtaining a semi-manufactured block whose strength is suitable for milling through numerical control equipment. The blocks are then milled by the dental technician to the desired shape, oversized to compensate for shrinkage during the final sintering that is expected to be around 21%. Shades different from the natural white are obtained by dipping in a dye solution and parts are finally sintered. The four dye solutions are analyzed by infrared spectroscopy, thermal analysis and ICP technique to investigate the chromophore elements.

Table I. Technical specifications of the materials studied

		B	K	S
CHEMICAL COMPOSITION	[wt%]			
ZrO_2		94.438	95	94.74-95.05
Y_2O_3		5.290	5.15±0.20	4.95-5.26
Al_2O_3		0.256	0.25±0.10	0.15-0.35
SiO_2		0.002	≤ 0.02	max 0.02
Fe_2O_3		0.002	≤0.01	max 0.01
Na_2O_3		0.012	≤0.04	max 0.04
DENSITY	[g/cm^3]	6.07	6.05	6.05
FLEXURAL STRENGHT	[MPa]	1156	1400	1200
HARDNESS (HV10)		1250	1250	1250

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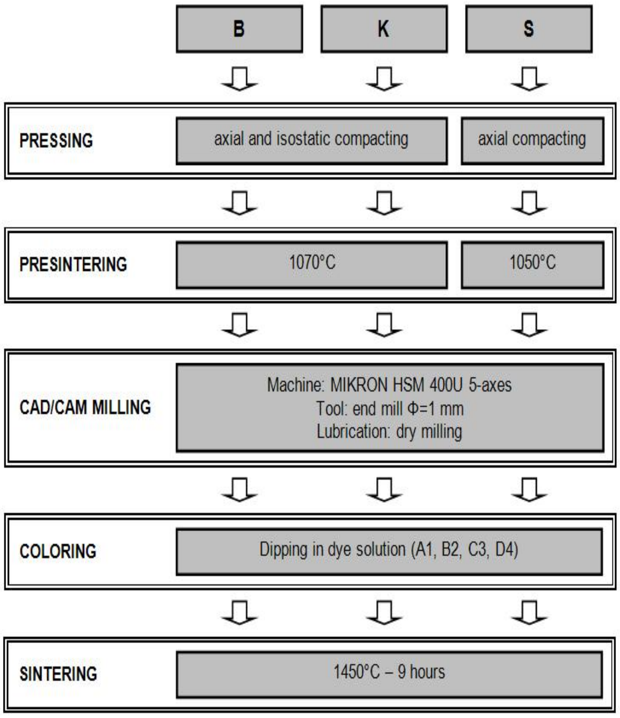

Figure 1. Specimens' production process

Specimens are produced consistent with ASTM C1161 (standard test method for flexural strength of advanced ceramics at ambient temperature), five for each typology, 75 specimens on the whole. The specimens are identified as follows: *formulation/color_ progressive specimen number* (i.e. B/A1_01 is the B Y-TZP, color A1, number 01).

The main dimensions and loading configuration for the three-point bend tests are listed in Table II. Instron 3345 testing machine is used, employing a load cell with a capacity of 5kN. HV1 Vickers hardness tests (load 9,807 N) are carried out on the specimens following ISO 14705 (Fine ceramics, advanced ceramics, advanced technical ceramics - Test method for hardness of monolithic ceramics at room temperature). Five indentations are measured on two samples for each group, for an amount of 10 values for each specimen typology. HV1 is measured in two steps:

- first the specimens are wet polished using 2500 grit SiC papers and hardness tests are carried out immediately afterwards;
- specimens are then annealed in air at 1200°C for 12 minutes and hardness tests are repeated, ensuring samples traceability.

Fine polishing is chosen to avoid the risk of creating macroscopic defects on specimen surface that could initiate failure[17] and evaluate only the effect of eventual phase transformation due to surface finish.

Table II. Specimens' dimensions and loading configuration (ASTM C1161).

specimen size	
width	4 mm
thickness	3 mm
length	45 mm
support span	40 mm
load span	20 mm
crosshead speed	0.5 mm/min

Flexural strength and hardness results are processed through statistical tools. The t-test for independent samples is carried out on the data, to evaluate the presence of significant differences between the different formulations and amongst the different shades for each producer. The null hypothesis is that the mean of individual differences of paired observations is zero. The *p*-level reported with a t-test represents the probability of error involved in rejecting the null hypothesis, i.e. accepting the existence of a difference between two groups of specimens. A level of significance of 0.05 is chosen. As to hardness measurements, the t-test for dependent samples is carried out as well comparing the results after polishing with those after annealing, to evaluate the influence of surface finishing on parts' properties and the eventual recovery.

The variability of flexural strength is characterized using the Weibull analysis, which is suitable for brittle materials as strength is determined by the most critical defect, so the Weibull modulus quantifies parts' reliability[1].

After testing, the rupture surface of a specimen for each of the three WHITE groups is observed using the scanning electron microscope (SEM) to investigate failure mechanisms. The three representative samples are chosen on the basis of flexural strength values close to the average recorded for that group.

RESULTS

Dye analysis

The four dyes are water solutions (90%) containing PEG, identified by infrared spectroscopy. Thermal analysis in air reveals that PEG burns completely at 600 °C, the remaining ash is analyzed by ICP mass spectrometry and EDS analysis obtaining the results in Table III. The main chromophore element in Fe in all dyes.

Table III. Chemical composition of dyes' ash.

Weight %	**A1**	**B2**	**C3**	**D4**
Fe	4.30	6.04	5.00	2.22
Cr	0.30	0.24	0.26	0.05
Cu	0.004	-	0.03	0.007
Mn	-	-	0.27	0.05
Ca	1.06	0.60	0.28	0.07
Al	0.16	1.38	0.19	0.012
Si	1.00	0.39	0.81	0.25

Flexural strength

Results of the bending tests are shown in Figure 2, where mean values are indicated on error boxes whose height is twice the standard deviation (SD). The three formulations are represented in three different colors and grouped as to the five shades, so that all comparisons are can be done between the groups. Mean flexural strength varies in the range 500 to 790 MPa for all the groups, definitely lower than the nominal values in the datasheets and in some cases nearly 50% of the expected strength. Also, experimental figures are significantly low with respect to literature data[1,5,17]. Some groups show very little scattering, whereas others prove much lower reliability.

Differences between the groups can be studied through the t-test, comparing the different shades of the same producer as well as groups of the same color between different formulations. Tables IV and V list the p-values obtained in the two analyses. Significant differences, related to p-values inferior than 0.05 are underlined.

The test proves a relevant difference between B and S formulations on three of the five shades (Table IV). For W and A1 colors the formulations are undifferentiated. Neither K or S specimens show significant differences in flexural strength between the five shades, whereas for B formulation three comparisons are statistically different: D4 compared to both W and A1, as well as A1 compared to B2 (Table V). Qualitative trends can also be observed in Figure 2. For B formulation color improves or does not worsen parts' strength, to an extent that in some cases is statistically significant. In the case of K and S, instead, most shades are weaker than the natural white specimens, even if the variations are below the level of significance.

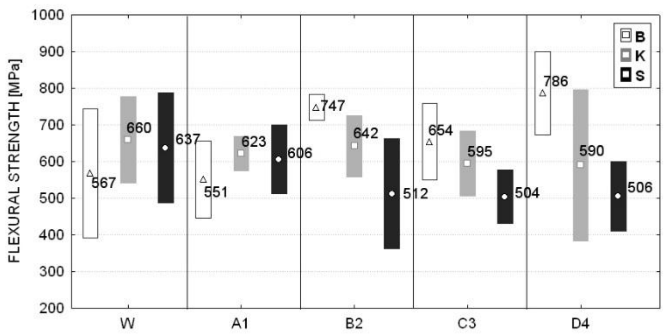

Figure 2. Flexural strength results: mean values and ±SD boxes.

Table IV. Results of t-test on flexural strength comparing different formulations on the same shade.

p value	W	A1	B2	C3	D4
B *vs* **K**	0.357	0.201	**<u>0.032</u>**	0.365	0.100
B *vs* **S**	0.517	0.410	**<u>0.009</u>**	**<u>0.030</u>**	**<u>0.003</u>**
K *vs* **S**	0.799	0.726	0.128	0.114	0.431

Values below the level of significance of 95% are underlined.

Table V. Results of t-test on flexural strength comparing different shades of the same formulation.

p value	B	K	S
W *vs* **A1**	0.868	0.536	0.703
W *vs* **B2**	0.055	0.792	0.223
W *vs* **C3**	0.370	0.357	0.111
W *vs* **D4**	**<u>0.048</u>**	0.530	0.136
A1 *vs* **B2**	**<u>0.004</u>**	0.666	0.268
A1 *vs* **C3**	0.159	0.553	0.090
A1 *vs* **D4**	**<u>0.009</u>**	0.736	0.132
B2 *vs* **C3**	0.096	0.413	0.913
B2 *vs* **D4**	0.485	0.614	0.935
C3 *vs* **D4**	0.092	0.961	0.976

Values below the level of significance of 95% are underlined.

The variability of flexural strength values is analyzed using the Weibull distribution function, expressed in equation (1)

$$P_i = 1 - \exp\left(-\left(\frac{\sigma_i}{\bar{\sigma}}\right)^m\right) \quad (1)$$

where P_i is the failure probability, σ_i the fracture strength, $\bar{\sigma}$ the mean strength and m is the Weibull modulus. In Figure 3 $ln(ln(1/(1-P_i)))$ is plotted against $ln(\sigma_i)$ for the three Y-TZP formulations. The five shades are grouped together on the basis of the results of separate initial analyses. The three plots can be well fitted by lines whose slope is the Weibull modulus, listed in Table VI together with the coefficient of determination R^2 of the linear regressions. Weibull modulus is quite low with respect to literature studies[1,5,17], which indicates poor reliability.

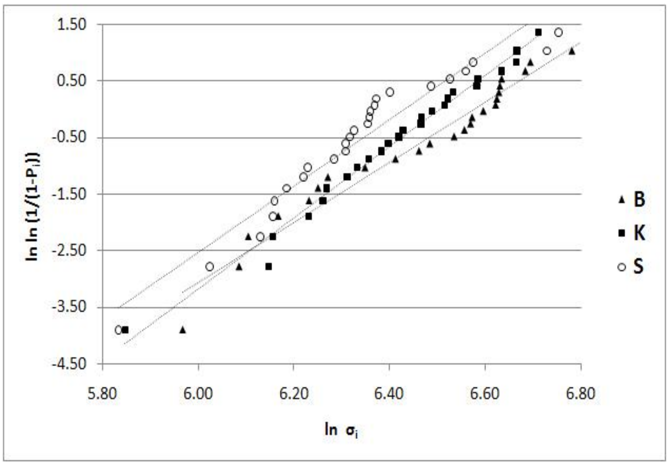

Figure 3. Weibull plots for the three Y-TZP formulations.

Table VI. Weibull modulus for the three Y-TZP formulations and determination coefficient of the Weibull plots.

	m	R^2
B	5.31	0.96
K	6.31	0.99
S	5.88	0.93

Hardness

The results of hardness measurements after polishing and after the subsequent annealing are listed in Table VII and diagrammed in Figure 4, where data relative to the two processing conditions are placed beside as solid and dashed bars.

Hardness is in the range 1500-1700 HV1, much higher than the values stated in the technical specifications and in accordance with literature data[1]. Standard deviation is lower than 50HV1 for all the groups except one. All the indentations are free from cracks, at least within the resolution of an optical microscope, which indicates the efficacy of the transformation toughening mechanism[4].

Table VII. HV1 measured after polishing and after annealing on the 15 specimen groups.

	B		K		S	
	polished	*annealed*	*polished*	*annealed*	*polished*	*annealed*
	mean (SD)	mean (SD)	mean (SD)	mean (SD)	mean (SD)	mean (SD)
W	1634 (33.4)	1600 (33.8)	1656 (31.4)	1632 (23.2)	1605 (41.6)	1616 (39.0)
A1	1627 (47.6)	1586 (43.5)	1660 (26.8)	1654 (26.8)	1620 (43.0)	1614 (38.1)
B2	1580 (44.5)	1569 (37.3)	1579 (73.5)	1630 (40.4)	1603 (20.1)	1626 (31.9)
C3	1629 (29.7)	1571 (39.4)	1637 (25.8)	1588 (30.3)	1599 (32.8)	1633 (27.4)
D4	1652 (35.2)	1577 (46.7)	1675 (41.9)	1635 (39.4)	1605 (20.5)	1638 (27.4)

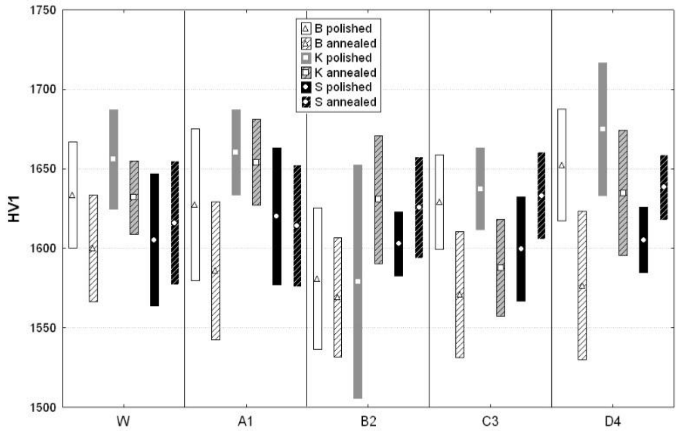

Figure 4. Hardness results: mean values and ±SD boxes. Solid plots are obtained after polishing, dashed ones after annealing.

Differences between the groups can be analyzed though the results of the t-test. Tables VIII and IX show the p-values obtained on HV1 for a each shade comparing different formulations (Table VIII) and for each producer contrasting different shades (Table IX). Hardness is significantly different amongst the three formulation in many cases, mostly for shaded samples. Also, the statistical analysis proves a great influence of color on hardness for B and K specimens after grinding, giving significant differences for many of the studied comparisons. These differences are often flattened after annealing. The specific effect of the annealing thermal treatment can be observed in Table X, where the results of the t-test for dependent samples show that for three of the five shades are hardness is significantly altered by the production process.

Table VIII. Results of t-test on HV1 comparing different formulations on the same shade.

p value	W		A1		B2		C3		D4	
	polished	*annealed*	*polished*	*annealed*	*polished*	*annealed*	*polished*	*annealed*	*polished*	*annealed*
B *vs* K	0.141	**0.024**	0.072	**0.001**	0.945	**0.002**	0.503	0.299	0.210	**0.007**
B *vs* S	0.111	0.339	0.723	0.140	**0.169**	**0.001**	**0.049**	**0.001**	**0.002**	**0.001**
K *vs* S	**0.007**	0.282	**0.021**	**0.014**	0.332	0.757	**0.010**	**0.003**	**0.000**	0.806

Values below the level of significance of 95% are underlined.

Table IX. Results of t-test on HV1 comparing different shades of the same formulation.

p value	B		K		S	
	polished	*annealed*	*polished*	*annealed*	*polished*	*annealed*
W *vs* **A1**	0.740	0.429	0.734	0.063	0.444	0.913
W *vs* **B2**	**0.008**	0.068	**0.007**	0.931	0.871	0.558
W *vs* **C3**	0.743	0.093	0.167	**0.002**	0.729	0.274
W *vs* **D4**	0.236	0.215	0.266	0.843	1.000	0.129
A1 *vs* **B2**	**0.036**	0.366	**0.004**	0.143	0.266	0.477
A1 *vs* **C3**	0.934	0.430	0.066	**0.000**	0.241	0.219
A1 *vs* **D4**	0.199	0.650	0.369	0.216	0.339	0.094
B2 *vs* **C3**	**0.011**	0.918	**0.029**	**0.015**	0.777	0.580
B2 *vs* **D4**	**0.001**	0.696	**0.002**	0.816	0.794	0.300
C3 *vs* **D4**	0.124	0.771	**0.027**	**0.008**	0.635	0.631

Values below the level of significance of 95% are underlined.

Table X. Results of t-test on HV1 comparing the specimens after polishing and after annealing.

p value	**B**	**K**	**S**
W polished *vs* annealed	0,061	0,091	0,488
A1 polished *vs* annealed	0,156	0,705	0,777
C3 polished *vs* annealed	**0,014**	**0,006**	**0,031**
B2 polished *vs* annealed	0,596	**0,011**	0,133
D4 polished *vs* annealed	**0,000**	0,106	**0,002**

Values below the level of significance of 95% are underlined.

Rupture surfaces

The rupture surfaces of all the observed specimens show a conchoidal fracture, similar to the one exemplified in Figure 5a. At higher magnifications cracks can be observed (Figure 5b).

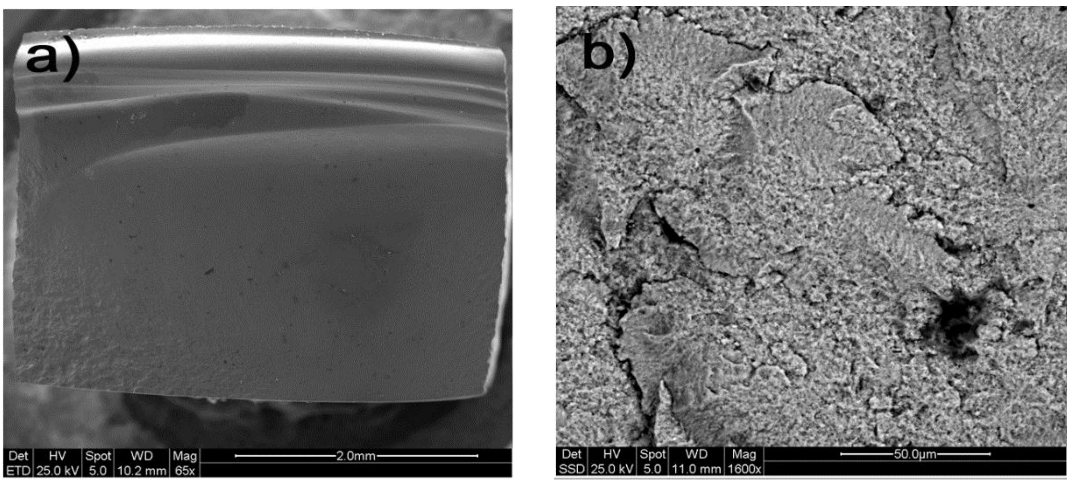

Figure 5. Rupture surface of a S white specimen.

DISCUSSION AND CONCLUSION

A comparative statistically-sound standardized study is proposed regarding flexural strength and microhardness of different commercial Y-TZP formulations and shades. The effect of polishing on surface hardness is also considered.

Flexural strength is proved to be quite low for all the specimens, at least 45% inferior to technical specifications, as well as parts' reliability attested by Weibull modulus. For one of the considered formulations, shade significantly affects strength in several cases. The direct consequence is that nominal characteristics should be assumed with great caution for prostheses' design. Nevertheless, measured flexural strength is higher than alternative ceramic prosthetic materials.

The studied materials result harder than expected, but the values are shade-dependent and in some of the considered cases the influence is not negligible. The effects of color on the mechanical properties are still very little known and considered, either by the producers or the users. Yet, the present study suggests that the property variations due to shade can not be neglected, so esthetical requirements must be merged with mechanical ones.

As a conclusion, Y-TZP is certainly a promising innovation in the field of fixed dental prostheses thanks to its outstanding aesthetical and mechanical properties. However, it shows an extreme variability with the manufacturing conditions that still needs investigation. Reliability of the expected mechanical response is required before widespread clinical application, to prevent failures caused by a poor knowledge.

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