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Translucent zirconia in the ceramic scenario for monolithic restorations: A Flexural Strength and Translucency comparison test.

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Short Title: Flexural Strength and Contrast Ratio comparison for translucent Zirconia.

Key Words: Translucent Zirconia, Y-TZP, Lithium Disilicate, Contrast Ratio, Flexural Strength, Translucency, Monolithic restoration, Grain Size.

Abstract

Objective: To compare three different compositions of Yttria-Tetragonal Zirconia Polycrystal (Y-TZP) ceramic and a lithium disilicate ceramic in terms of flexural strength and translucency.

Methods: Three zirconia materials of different composition and translucency, Aadva ST [ST], Aadva EI [EI] and Aadva NT [NT](GC Tech, Leuven, Belgium) were cut with a slow speed diamond saw into beams and tabs in order to obtain, after sintering, dimensions of 1.2x4.0x15.0mm and 15.0x15.0x1.0mm respectively. Blocks of IPS e.max CAD LT were cut and crystallized in the same shapes and dimensions and used as a reference group [LD]. Beams (n=15) were tested in a universal testing machine for three-point bending strength. Critical fracture load was recorded in N, flexural strength (σ in MPa), Weibull modulus (m) and Weibull characteristic strength (σ^0 in MPa) were then calculated. Tabs (n=10) were measured with a spectrophotometer equipped with an integrating sphere. Contrast Ratios were calculated as CR=Yb/Yw. SEM of thermally etched samples coupled with lineal line analysis (n=6) was used to measure the tested zirconia grain size. Data were statistically analyzed.

Results: Differences in translucency, flexural strength and grain size were found to be statistically significant. CR increased and flexural strength decreased in the following order (most translucent/weakest) LD-NT-EI-ST (least translucent/strongest). The average grain size was different for the three zirconia samples with (largest) NT>ST>EI (smallest).

Conclusions: All the three zirconia groups were stronger than, but not as translucent as, the LD group. Clinical indications for Zirconia Aadva NT should be limited up to three-unit span bridge.

Introduction

Yttria-Tetragonal Zirconia Polycrystal (Y-TZP) is considered one of the most versatile bioengineering ceramics due to its mechanical, optical and physical proprieties.[1-3]

High hardness and fracture toughness are the main reasons for the adoption of Y-TZP in dentistry as a material indicated for fabrication of fixed partial denture frameworks, monolithic crowns and bridges, implant abutments or screw-retained prostheses.[4] As an advantage in fixed prosthodontics, the Y-TZP structure is responsible for characteristic optical properties like favourable colour and translucency.

Translucency is considered one of the most important factors in matching the appearance of natural teeth with restorative materials and has been defined as the relative amount of light transmission.[5,6]

At clinically indicated thicknesses, the material does not offer a complete barrier to light transmission through the structure, unlike the metal in porcelain fused to metal restorations.[7]

Nevertheless, the absence of a glass matrix in the dense sintering polycrystalline zirconia results in lower translucency compared with other ceramic materials and tooth structures.[8] The ability of light to pass thorough zirconia structure is related to several factors: particle and grain size[9-11] density,[11] and crystal structure.[12-14]

The sintering temperature influences the grain size and density; the smaller the particle and higher the temperature the denser the structure with a larger grain size that influences the translucency.[10] The use of different quality and quantity of dopants and stabilizers has been reported to affect the structure of grain and crystals with consequent influence on both optical and mechanical properties.[14-17]

The need for “high translucency” zirconia is related to the possibility of aesthetic improvement for monolithic restorations. Monolithic zirconia restorations could moreover represent an advantage in terms of simplification of procedure, cost reduction and could overcome the problem of veneer chipping.[18]

New compositions of Y-TZP with claimed different optical and mechanical properties for dental CAD/CAM machining systems were recently introduced to the market. The aim of this study was to compare translucency, as measured by Contrast Ratio, with mechanical properties in terms of flexural strength (σ), Weibull modulus (m) and Weibull characteristic strength (σ^0) for three different Y-TZP samples and compare these to a (high translucency) lithium disilicate glass ceramic used as a control.

The tested null hypotheses were that (1) no statistically significant differences existed in terms of flexural strength and translucency between the tested materials and (2) there was no relationship between strength and translucency in the three materials.

Materials and Methods

CAD/CAM pre-sintered disks of zirconia (Aadva, GC Tech, Leuven, Belgium) characterized by different translucencies and composition (Table 1) were selected for the study; these were Aadva ST (standard translucency – ST group), Aadva EI (Enamel Intensive – EI group) and Aadva NT (natural translucent – NT group).

Disks were cut by a slow speed diamond saw (IsoMet Low Speed Saw, Buehler, Lake Bluff, IL, USA), cutting dimensions of the specimens were calculated to compensate the shrinkage induced by dense-sintering.

All the specimens were sintered in a sintering furnace (Sirona InFire HTC Speed, Sirona Dental, Bensheim, Germany) following the manufacturer's instructions. Briefly, the furnace temperature rose at 5-6°C per minutes until 900°C, it was then held at 900°C for 30 minutes, before increasing very slowly to 1500°C over 4.5 hours, 2 hours at 1500°C, decrease until 1000°C in one hour, then to room temperature very slowly.

Lithium Disilicate blocks for CEREC® (IPS e-max CAD LT, Ivoclar Vivadent AG, Schaan, Liechtenstein) were used as a control material (LD group). With the use of a proprietary device, blocks were perpendicularly cut in order to obtain the desired shape. Specimens were submitted to crystallization firing in a ceramic furnace (Vacumat® 6000M, Vita Zahnfabrik, Bad Säckingen, Germany) following the manufacturer's instructions.

Flexural Strength - 3Point Bending Test

Beam-shaped specimens (n = 15 per group) were prepared and wet-finished with 600 grit paper until dimensions of 15 ± 0.2 mm length, 4 ± 0.2 mm width, and 1.2 ± 0.2 mm height were obtained. Specimens were then wet-polished with 1,200 and 2,400 grit paper. According to ISO 6872:2015, a 45° edge chamfer was made at each major edge.[19]

Tests were performed in a universal testing machine (Triax 50, Controls, Milano, Italy) with a cross-head speed of 1 mm/minute and the span was set at 13.0 mm. Specimens were tested dry at room temperature. The fracture load was recorded in N, and the flexural strength (σ) was calculated in MPa by using the following equation:

$\sigma = Pl / 2wb^2$

where P in the fracture load in N, l is the span in mm, w is the specimen width in mm, and b is the specimen height in mm.

The Weibull characteristic strength (σ^0) and the Weibull modulus (m) were calculated according to the following equation:

$P_f = 1 - \exp [- (\sigma / \sigma^0)^m]$

where P_f is the probability of failure between 0 and 1, σ is the flexural strength in MPa, σ^0 is the Weibull characteristic strength in MPa, and m is the Weibull modulus.

The flexural strength (σ) data were statistically analyzed. One Way ANOVA was applied, followed by the Tukey test for post-hoc comparisons, whereas the level of significance was set at $\alpha = 0.05$.

Translucency measurement – Contrast Ratio (CR)

For optical evaluation, tab shaped specimens (n = 10 per group) with final dimension of 15 ± 0.5 mm in length, 15 ± 0.5 mm in width, and 1.0 ± 0.1 mm thick were obtained and wet-polished with 1,200, 2,400 and 4,000 grit paper.

The measurements were performed with a spectrophotometer (PSD1000, OceanOptics, Dunedin, FL, USA), equipped with an integrating sphere (ISP-REF, OceanOptics) with a 10-mm opening. The spectrophotometer was connected to a computer running color measurement software (OOILab 1.0, OceanOptics). D65 illumination and 10° standard observation angle were selected.

Data were recorded in CIEXYZ colorimetric systems. A quantitative measurement of translucency was made by comparing the reflectance of light "Y" in CIEXYZ colorimetric system (ratio of the intensity of reflected radiant flux to that of the incident radiant flux) through the test specimen over a backing with a high reflectance (White backing – Y_w) to that of low reflectance or high absorbance (Black backing – Y_b). [20] Contrast Ratio was calculated with the following equation:

$CR = Y_b/Y_w$.

It is important to note that by this method a high translucency would produce a low CR. Translucency data were statistically analyzed. One Way ANOVA was applied, followed by the Tukey test for post-hoc comparisons, whereas the level of significance was set at $\alpha = 0.05$.

The Pearson correlation test was applied to the obtained data for CR and Flexural Strength (σ).

SEM Evaluation

An extra specimen per group was produced for microscopic ceramic microstructural evaluation.

Zirconia specimens were thermally etched in air in order to show grain boundaries. Thermal etching was performed in sintering furnace, the firing temperature was set 150°C below the sintering temperature and maintained for 20 minutes.[21]

A LD specimen was etched for 60 seconds with 4.9% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent AG, Schaan, Liechtenstein), and cleaned under running water.

Specimens were ultrasonically vibrated in a 95% alcohol solution for 3 minutes (CP104, CEIA, Italy), and air dried with an oil-free stream and then secured to SEM (JSM-6060LV, JEOL, Tokyo, Japan) tabs with gold conducting tape. After gold coating in a vacuum sputter coater (SC7620 Sputter Coater, Polaron Range, Quorum Technologies, Newhaven, UK) samples were submitted to SEM observation. The LD surface was observed at x5000 while the zirconia groups were examined under x35000 magnification for crystal morphology evaluation. Grain size measurement for the three zirconia samples was conducted by the lineal intercept method which involved counting the number of interceptions made by a known-length test line [22] on a digitally calibrated SEM image of the sample surface using Image J software; six lines in different orientations were used for each analyzed image, and average grain size calculated as:

$D = 1.56 [C/MN]$

Where D was the average grain size, 1.56 was the proportionality constant due to non-spherical grains, C the total length of test line used, N the number of intercepts and M the magnification of the photomicrograph (=1 in this study as the image was already digitally calibrated). One Way ANOVA was applied, followed by the Tukey test for post-hoc comparisons, whereas the level of significance was set at $\alpha = 0.05$.

Results

The mean of CR, flexural strength (*σ*), Weibull characteristic strength (σ^0), Weibull modulus (*m*), grain size and statistical significances are reported in Table 2.

All the differences between groups were found to be significant for all the tested variables. Translucency and flexural strength acted as inversely related variables; this relationship was linear with a correlation co-efficient of 0.89. Materials resulted in the following order from the most opaque and strongest to the most translucent and weakest: ST > EI > NT > LD (Figure 1 and Figure 2). Weibull graphs are shown in Figure 3.

The SEM evaluation (Figure 4) of the various zirconia ceramic surfaces highlighted the structural differences between groups. The smallest grains were observed for EI, intermediate grain dimensions were reported for ST and larger grains were observed on the NT zirconia surface. The average grain sizes for each material were statistically different to each other.

The control group LD highlights the differences between polycrystalline ceramics and glass ceramics. After the glass matrix dissolution by acid etching, elongated crystals of lithium disilicate were evident. Elongated crystals were randomly oriented and were interspersed with a little amount of small spherical crystals (Figure 4).

Discussion

Flexural strength and translucency between groups showed statistically significant differences and there was a clear inverse relationship between these variables, therefore both null hypotheses have been rejected.

Flexural strength was not related to mean grain size which is perhaps not surprising given there were also differences compositionally between the samples in terms of stabilizers added. Weibull plots showed distinctive shoulders and S shaped curves which may be indicative of residual stress or different populations of flaws being present in the samples. There were no obvious flaws visible in the SEM images.

Translucency is one of the main parameters in matching the appearance of the natural tooth and was identified as pivotal factor in controlling aesthetics and in a critical consideration for material selection.[20]

In the traditional composition of the ST group most of the light passing through the material is intensively scattered and diffusely reflected, leading to an opaque appearance, reaching the limit between a “low translucent” and a “medium translucent” material according to Vichi et al.[8]. Translucency is generally measured with CR. CR is the ratio of the reflectance of a specimen over a black backing to that over a white backing of a known reflectance, and is an estimate of opacity. CR ranges from 0 to 1, with 0 corresponding to transparency (totally translucent) and 1 corresponding to total opacity (absence of translucency). The mean measured values of CR and flexural strength of ST were similar to that reported for other 3Y-TPZ.[23-25]

The difference in CR between the ST and EI (CR 0.05) and between EI and NT (CR 0.04) even if statistically significant should be considered from a clinical point of view. Differences below 0.07 in CR should be considered not visible by the human eye based on the mean translucency perception threshold (TPT) defined by Liu et al.[26] although the authors recognized that there were significant variations depending upon the observer, e.g. a clinician with 10 years of shade-matching experience could have a TPT of 0.04. Accordingly, even if below the 0.07 mean TPT, the increased translucency of EI compared to ST could be perceived by expert clinicians and technicians and it could accordingly be classified as a “medium translucent” material.[8]

It has been reported by Samodurova et al.[27] that the presence of alumina positively influences the nucleation of zirconia and promotes strong grain boundaries. The absence of Al_2O_3 and the small grains generated after dense sintering (Figure 4) of EI could induce an increased ability for the light to pass through the material in contrast to the larger grain sizes seen for ST, which contains Al_2O_3 and may justify the decreased flexural strength obtained by the Al_2O_3-free composition.

The recorded differences for mean flexural strength between EI and ST do not influence the clinical indications of EI according to ISO 6872:2015 (Table 3). Both the ST and EI zirconia compositions fulfill the highest requirements (ISO Class 6) and are accordingly indicated for up to four or more unit FPDs.[19]

Further investigations will be necessary to evaluate the long term stability of the tetragonal phase (t-ZrO_2) and the influence of phase stability on mechanical and optical properties. The exposed surface of zirconia is susceptible to a phase change from t-ZrO_2 to monoclinic (m-ZrO_2). This aging phenomenon, called low temperature degradation (LTD), may affect the mechanical properties of the material; the presence of alumina was reported to have a preventative role in in zirconia LTD[16,13] and accordingly there may be differences between EI and ST following LTD.

The formulation of NT differed from that of ST or EI with an absence of Al_2O_3 and moreover an increased level of Y_2O_3 from 5% to 9% in weight (corresponding respectively to 3% and 5.5% mole) used as a stabilizer. The increment of yttria induced, during dense sintering, the development of a certain amount of cubic (c-ZrO_2) and tetragonal (t-ZrO_2) zirconia grains (Figure 3).

An increment of yttria as stabilizer from 3% to 8% mole has been associated with an increment of cubic phase in zirconia structure and to an increment in translucency;[14] at this level there is a change in the zirconia from partially stabilized (PSZ) to fully stabilized (FSZ) with several structural implications.[17,12,28] The GC Aadva NT, however, with a yttria content of 5.5% mol does not achieve this and should be considered a PSZ even if contains both t-ZrO_2 and c-ZrO_2.

The level of translucency reached by NT was significantly higher if compared to the other two zirconia but moreover significantly lower compared to LD. NT has a positive difference in CR of 0.09 with ST and the same negative difference with LD; both of these differences are above the TPT. Together with EI, NT could be classified as “medium translucent” material. Their use as monolithic material in aesthetic areas should not to be recommended because they are still less translucent that natural tissues. The reported CR for Enamel and Dentine was about 0.45 and 0.65 respectively.[8]

Accordingly, in order to obtain a tooth like appearance, a veneering process for all the tested materials is highly recommended. These findings are in general agreement with several studies involving other “translucent zirconia” that reported a significant lower level of translucency when compared with lithium disilicate.[13,29-31] Furthermore unlike the lithium disilicate, the tested zirconia had their natural white colour. It has been widely reported by several authors that the use of coloring liquids or pre-coloured material with an increased chroma had a significant negative influence for CR and light transmittance.[31-34] Further studies should be performed to clarify the influence of different shades of coloring liquids on the translucency of the tested zirconia materials.

The significant lower strength achieved by NT compared to ST and EI, has been correlated with the presence of c-ZrO_2 crystals. Lower flexural strength has been reported also for other “translucent zirconia” containing cubic phase, such as the FSZs.[12] Due to the higher stability induced by yttria, it has been reported however that the zirconia surface was less susceptible to LTD.[15] These consideration should be evaluated for NT with further investigations.

The clinical indications for NT are limited up to three unit FPDs, corresponding to ISO Class 5 (Table 3). The LD samples with a significant lower mean flexural strength compared to the other tested materials, fulfill the requirements of Class 3 of Class 4 materials; clinical indications are limited up to three unit FPDs not involving molar region.[19]

The well known relationship that correlates mechanical properties, translucency and material thickness should be carefully evaluated by clinicians during material selection. Lowering the thickness of the restoration would allow the material to be more translucent [35] but minimal indicated thickness should always be respected in order to avoid the risk of material fracture failure. Precise indications for minimal thickness should be provided by the manufacturer with respect to the wide range of available materials in fixed prosthodontic.

In order to achieve excellent aesthetics, material thickness should not be excessive because increased thickness is related to lower translucency. For this reason, achieving the optimal natural appearance of a human tooth with a monolithic restoration that guarantees adequate mechanical and optical properties, requires further investigation. Zirconia due to its versatility as a bioengineered ceramic could be easily influenced by the use of different dopants and stabilizers, interesting results as been recently reported by Zhang et al.[16] by the experimental introduction of 0.2% mole La_2O_3 in conventional Al_2O_3-doped 3Y-TZP, which resulted in a translucency close to that of lithium disilicate, absence of LTD and excellent mechanical properties. These findings, even if encouraging, need further investigation in order to validate the use of different dopants in dentistry.

Conclusions

Within the limitation of this *in-vitro* study, the following conclusions could be drawn:

There was an inverse relationship between strength and translucency for the materials tested.

Addition of Al_2O_3 and increasing yttria content strongly influence mechanical and optical properties of Y-TZP ceramics which will affect their clinical indications.

The NT zirconia has a significant higher translucency than the other zirconia materials tested but a lower flexural strength that limits its clinical indication up to three unit FPDs (ISO 6872:2015).

Table and Figure

Table 1: Composition of tested Aadva Zirconia Disks.

Components	ST	EI	NT
ZrO_2 wt%	94.8	95	91
Y_2O_3 (wt%) [mole]	(5) [3%]	(5) [3%]	(9) [5.5%]
Al_2O_3 wt%	0.2	trace	trace
Crystal structures	Tetragonal	Tetragonal	Tetragonal & Cubic

Table 2: Results and statistical significance, different letters indicate different statistical significance.

Translucency - CR			**Flexural Strength - 3PBT**				**Grain Size**	
Groups	Mean	SD	Mean σ (MPa)	SD	m	σ^0 (MPa)	Mean (nm)	SD
Aadva ST	0.74^{d}	0.01	1215^{a}	190	7.1	1296	445^{b}	34
Aadva EI	0.69^{c}	0.01	983^{b}	182	5.0	1059	284^{c}	11
Aadva NT	0.65^{b}	0.01	539^{c}	66	10.1	566	558^{a}	38
IPS e.max LT	0.56^{a}	0.02	377^{d}	39	9.81	395		

Table 3: Clinical recommendation proposed by ISO 6872:2015 for dental ceramics.

Class Recommended clinical indications Flexural strength minimum (mean) MPa		
1	(a) Esthetic ceramic for coverage of a metal or a ceramic substructure.	50
	(b) Esthetic-ceramic: single-unit anterior prostheses, veneers, inlays, or onlays.	
2	(a) Esthetic-ceramic: adhesively cemented, single-unit, anterior or posterior prostheses.	100
	(b) Adhesively cemented, substructure ceramic for single-unit anterior or posterior prostheses.	
3	Esthetic-ceramic: non-adhesively cemented, single-unit, anterior or posterior prostheses.	300
4	a) Substructure ceramic for non-adhesively cemented, single-unit, anterior or posterior prostheses.	
	b) Substructure ceramic for three-unit prostheses not involving molar restoration.	
5	Substructure ceramic for three-unit prostheses involving molar restoration.	500
6	Substructure ceramic for prostheses involving four or more units.	800

Figure 1: Box Plot for Flexural Strength data.

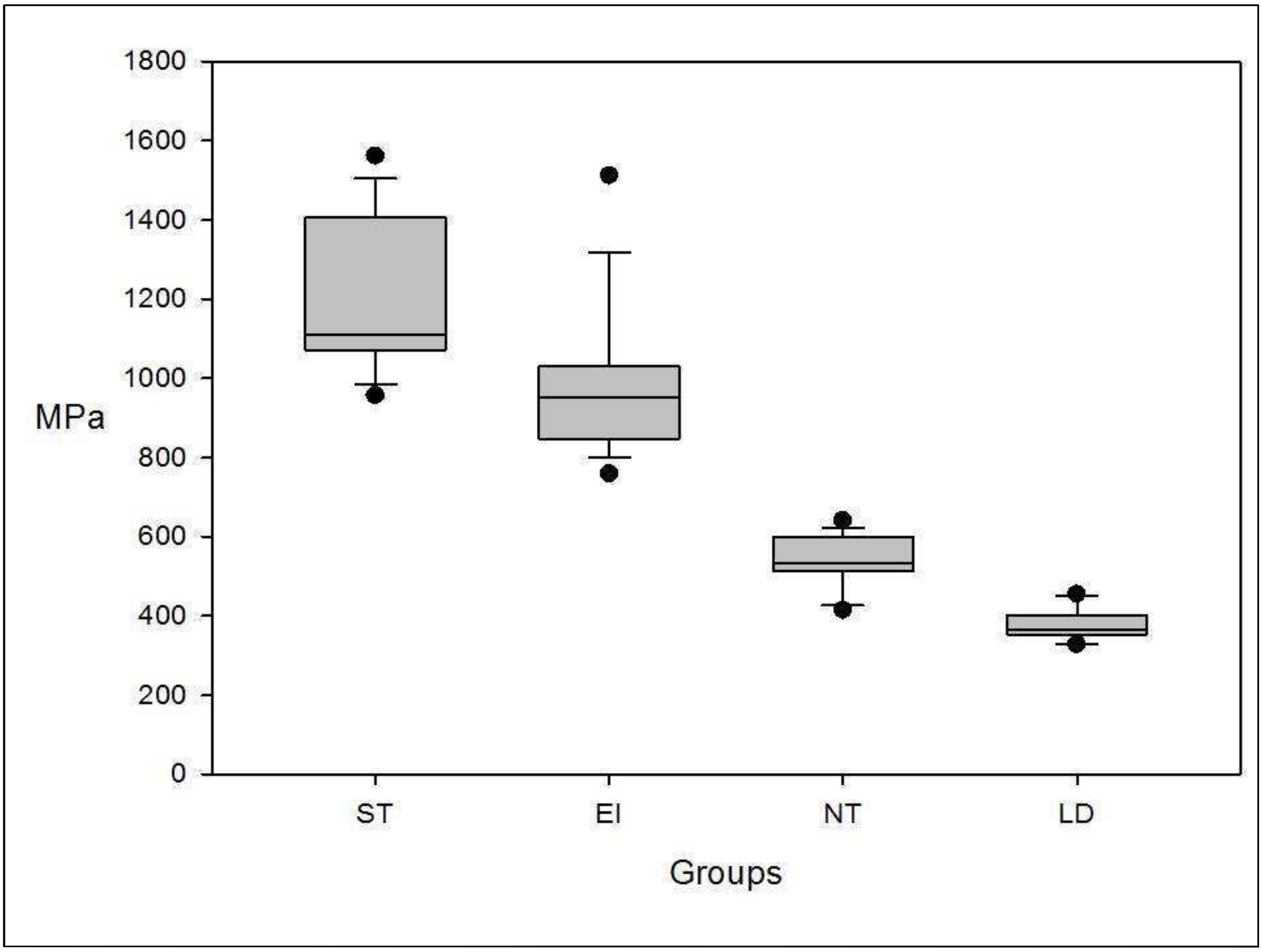
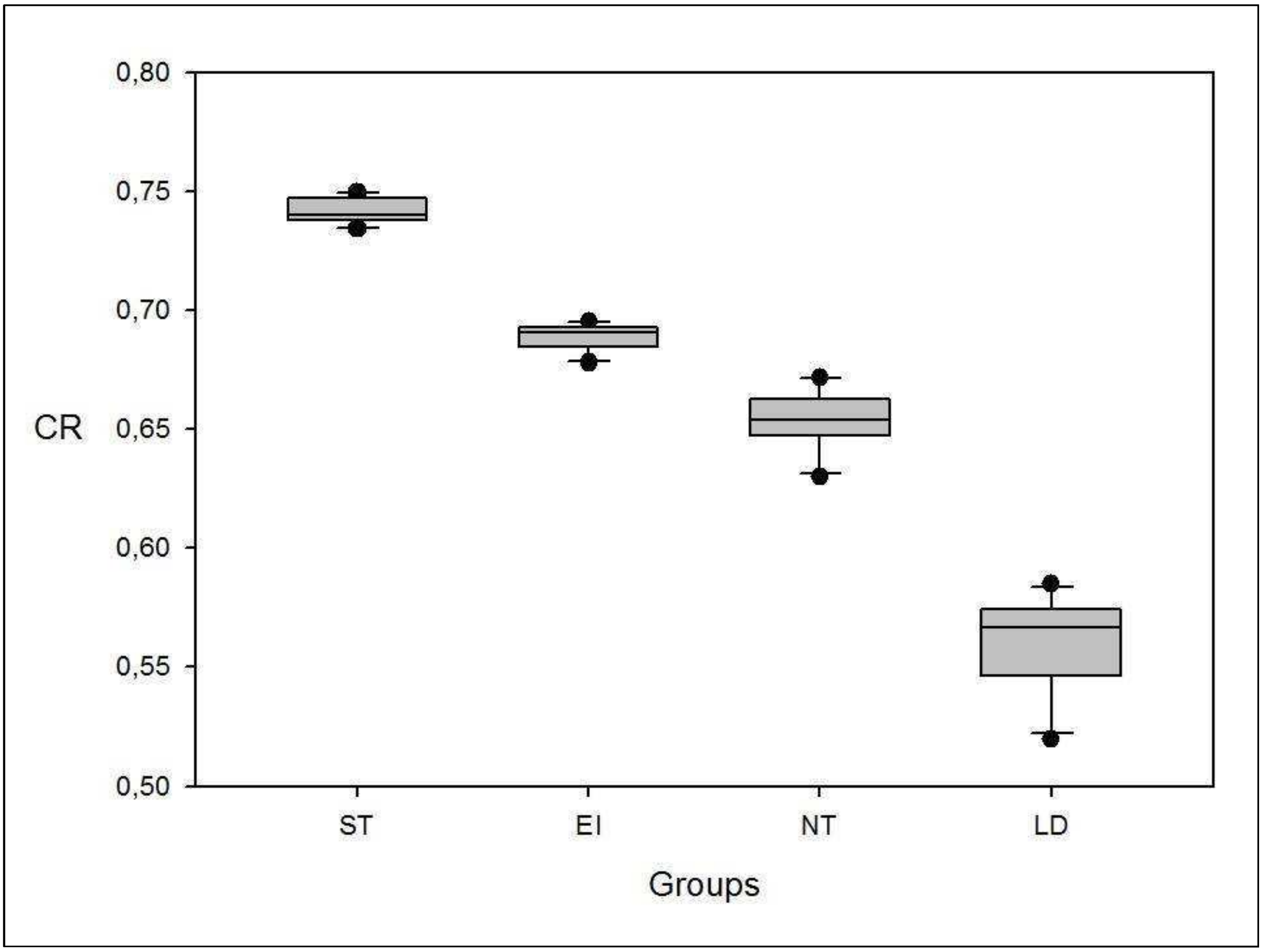


Figure 2: Box Plot for Translucency data.



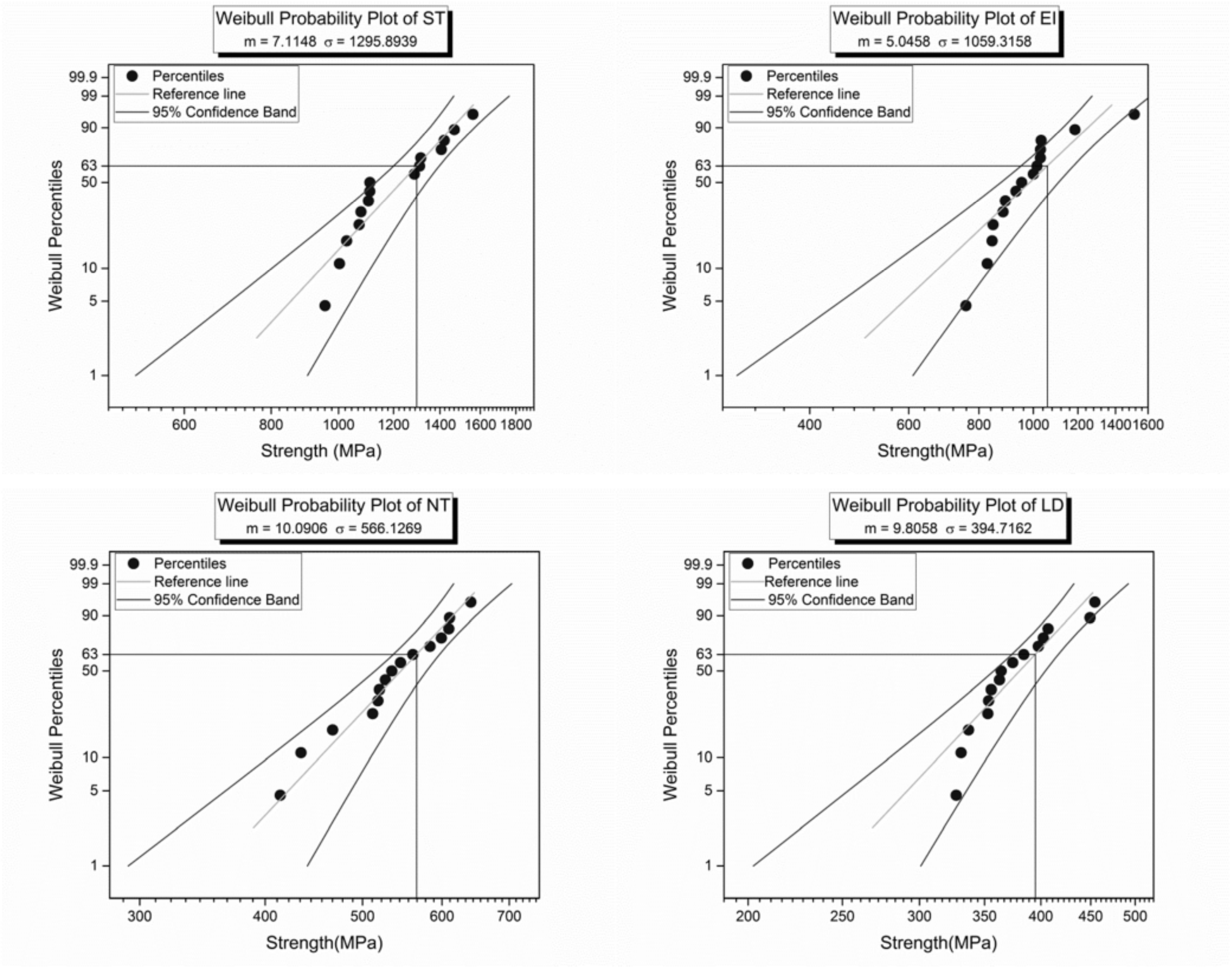
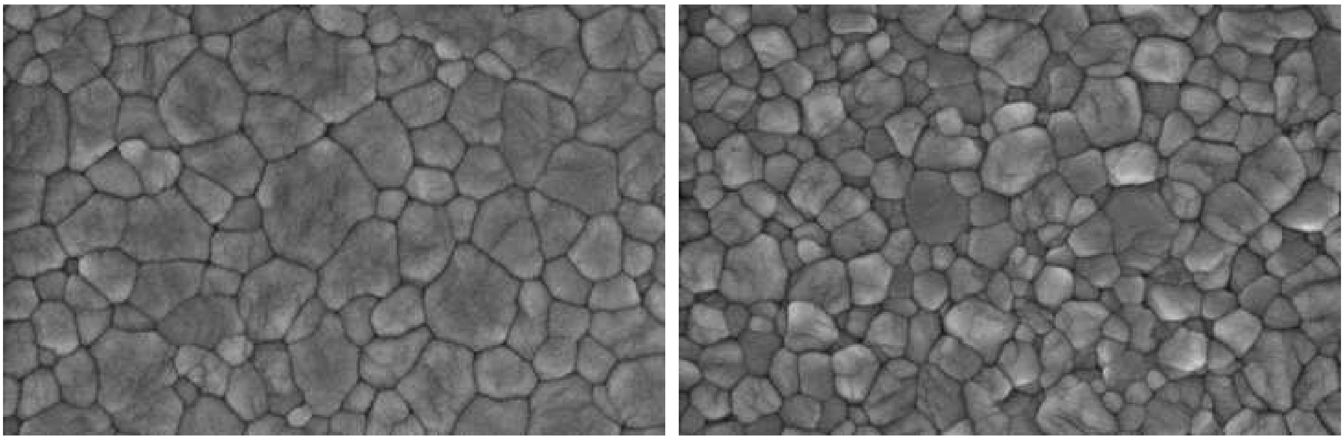
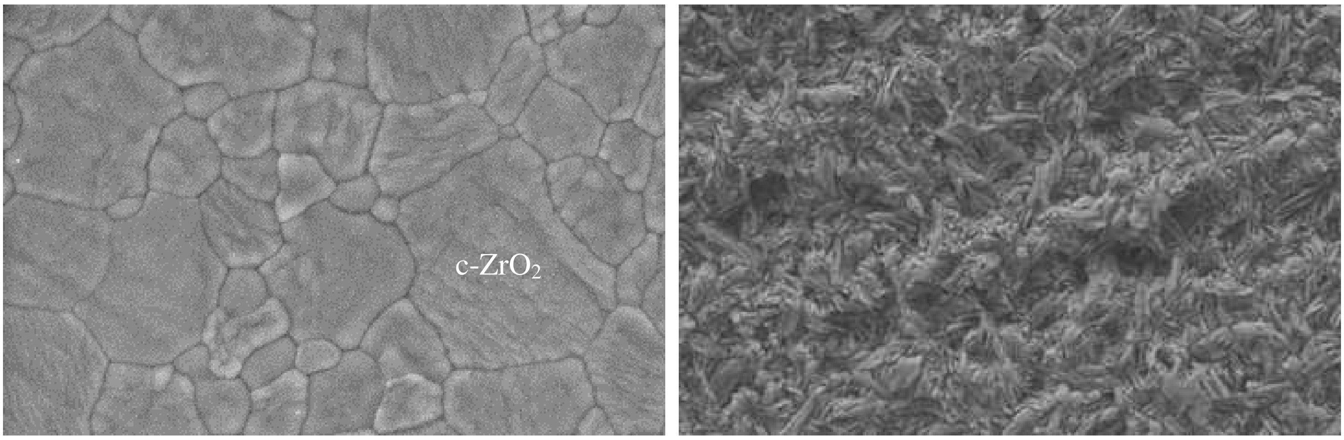


Figure 3: Weibull distributions for each of the groups



Aadva ST x35000

Aadva EI x35000



Aadva NT x35000

IPS e.max CAD LT x5000

Figure 4: SEM evaluation of ceramic structure.

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