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Bi-colored zirconia as dental restoration ceramics

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Abstract

Machinable zirconia blocks with gradient colors are fundamental for fabrication of full-contour esthetic dental restorations. The aim of this study was to process and evaluate bi-colored zirconia ceramics as a pilot dental material by using well-established techniques. Two commercially available partially stabilized zirconia granules, one undoped and one doped with 0.202 wt% Fe_2O_3, resulted in white and yellow colors after sintering, respectively. Bi-colored zirconia was fabricated by two-step dry pressing of both zirconia granules one above the other to form green bodies, followed by cold isostatic pressing (CIP) and, a two-step pressureless sintering finally at 1450 °C. The dilatometer results showed that the Fe_2O_3 doped zirconia sintered slightly rapid, but the difference of shrinkage between two powders was $< 1\%$. Sintered bars achieved full density, 6.018 g/cm^3 (~99%TD), without cracks in the ~1 mm color gradient zone. The microstructures were characterized by scanning electron microscopy (SEM) and careful observation of both surface and interior provided no obvious structural difference of either grains or pores among the three distinct regions, comprising white, yellow and color gradient zone. Vickers hardness of bi-colored zirconia was ~13.1 GPa, with no obvious difference in the three regions. The four-point bending strength of the bi-colored zirconia bars was 745.5 ± 159.6 MPa, which appeared noticeably lower than that of the single-colored references being above 1000 MPa. Fractographic analysis revealed that in most of the cases (60%) the fracture was initiated at the color gradient zone, where large voids with high coordination numbers, agglomerates with critical size and concentration of irregular grains with porous surfaces were observed. Above all, bi-colored zirconia ceramics prepared by the improved technique could meet the basic requirements of dental materials. The ways of minimizing the defects within bi-colored blocks should be developed for the production of esthetic zirconia ceramics of high strength and reliability.

Keywords: A. Pressing; C. Color; D. ZrO_2; E. Biomedical application

1. Introduction

Imitation of natural teeth is the purpose of the artificial dental restorations. It is for the esthetics that ceramics were introduced into prosthetic dentistry. The initial application of ceramic materials was to mask the metal-core color of a crown by fusing porcelain layer by layer onside. This kind of restorations is called as Porcelain Fused to Metal (PFM) crowns [1]. PFM crowns with an opaque metal inside cannot produce the important translucent effect as natural teeth [2]. Additionally, the release of metal ions may cause gingiva discoloration, which would decrease the visual aesthetics further [3]. Besides promoting high cosmetic requirements, other beneficial properties of ceramics are the biocompatibility and chemical stability. Subsequently, the metal cores were replaced by ceramic cores and the strongest dental ceramic commercially available is 3 mol% yttria stabilized tetragonal zirconia polycrystals (3Y-TZP), shortened as zirconia below. This zirconia has become the most popular choice for the dental core and multiple units framework [4].

The porcelain veneered zirconia all-ceramic restorations once were considered as the most promising combination of strength and esthetics. However, the frequently observed chipping of veneering porcelains reduced this anticipation [5–7]. The chipping of veneers was mainly caused by poor

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bonding between porcelain–zirconia [8] and has not yet been solved, although improvements have been reported [9–11]. It was also observed that the veneered layer-construction decreased the strength significantly compared to the monolithic construction [12]. This behavior is expected as the strength of multilayer materials depends on the weakest component; in the veneered crowns being the brittle porcelain. The bending strength of porcelain is only around 100 MPa and porcelain has a very low toughness causing fracture under high biting load [13]. The success of traditional layer-by-layer veneering procedure is dependent on labor technicians with high technical skill and the procedure is very time-consuming. Therefore, full-contour dental zirconia restorations without a need of veneering are highly desired nowadays [14].

Color is one of the first properties to be consider for the esthetic appearance of an artificial tooth [15]. Most of the available commercial zirconia ceramics give white color upon sintering, which disagrees from most natural teeth. Although series of coloring liquids are available for dental zirconia ceramics, the final restorations will be colored homogeneously. They cannot reproduce the gradient color of the natural teeth, which is the integral effect of translucent enamel and yellow dentin [16]. In this case, the requirement of veneers for esthetic purpose can hardly be eliminated.

For mimicking the color gradient appearance of a natural tooth, one possible way is to manipulate the distribution of the coloring additives within one single zirconia block. This novel esthetic zirconia block contains several structure layers with different coloring additive arranged in desirable orders. It could be regarded as a kind of Functional Gradient Material (FGM), where the function refers to the esthetic property. Combined with the advanced CAD/CAM technique, the full-contour zirconia restorations can be shaped from such blocks and are expected to exhibit satisfied esthetic effects for direct use in the posterior regions.

The so far established process for producing machinable zirconia blocks is dry pressing of thermal-spray granulated zirconia powders into green bodies followed by partial sintering. These porous blocks can be milled, then sintered to full density and give individually shaped restorations [17]. With the development of pre-colored zirconia powders, it appeared possible to fabricate layered and differently colored esthetic zirconia blocks based on the existing equipment and processes. The aim of this study was to investigate the feasibility of the new process and evaluate the mechanical properties and microscopic features of a bi-colored zirconia pilot dental material.

2. Materials and methods

2.1. Sample preparation

Two commercial 3Y-TZP powders manufactured by Tosoh Cooperation (Tokyo, Japan) were selected, namely, TZ-3YSB-E (Lot SY301041B) and TZ-YELLOW-SBE (Lot S300033B). They will yield ceramics with two different colors after sintering. The former gives after sintering a white body and the latter one becomes yellow, due to doping with a small amount of Fe_2O_3 (0.202 wt%). Both of them are provided as thermal-sprayed granules aimed for direct dry pressing. Bi-colored 3Y-TZP discs were made by uniaxial dry pressing of the same amount of each powder in a stainless steel die one layer above another under 2 MPa pressure for 1 min, followed by cold isostatic pressing (CIP) under 200 MPa. Pressureless sintering was performed in air at 900 °C to make a partly solidified bulk that could be easily shaped by milling. Rectangular bars were cut from this bi-colored zirconia disc and then fully sintered at 1450 °C for 2 h. A color gradient zone was observed in the middle of the bars. All the experimental bars were grinded and polished to a fineness of 1 μm using a series of diamond polishing papers. The final dimensions of the test bars were $20 \times 4 \times 1.2$ mm^3 with edge chamfers suitable for four-point bending test according to ISO 6872:2008(E) [18]. The bars were ultrasonically cleaned in acetone solution and deionizer water for 5 min, respectively. Fig. 1 shows a photograph of the 20 mm long test bar of the bi-colored type. Two reference groups were made from pure white or yellow zirconia powders, respectively, through the same way as the bi-colored group but just with one dry pressing. Ten bars in each group were prepared for bending test.

2.2. Characterizations

The sintering shrinkage behavior of the two different zirconia powders was investigated by using a contact-mode dilatometer (Bähr-Thermoanalyse, Hüllhorst, Germany). The powders were sintered at a heating rate of 3 °C/min up to 1450 °C and hold for 2 h. The density of the sintered samples was determined in water by Archimedes' method. The theoretical density of 3Y-TZP is calculated to be 6.10 g/cm^3.

The microstructure of the bi-colored zirconia samples was investigated by scanning electron microscopy (SEM, JEOL JSM-7000F, Tokyo, Japan). For achieving a better contrast, the samples were thermally etched at 1250 °C for 1 h before being coated with a thin carbon layer to avoid charging effects. For evaluating the interior microstructure, one sample was cross-section polished (CP) by an ion beam polisher (JEOL SM-09010, Tokyo, Japan) at the color gradient zone. The grain sizes in three different regions (white, yellow and the color gradient zone) were measured by the lineal intercept method. The reported mean grain size was calculated according to ISO 13356:2008 [19].

Fig. 1. A photo showing a 20 mm long bi-colored zirconia test bar prepared for four-point bending test.

Three bi-colored zirconia bars were randomly chosen to determine the Vickers hardness (Zwick Roell Indentec ZHV30, Leominster, United Kingdom). For each bar, five indentations at 30 kg on the surface of each of the three distinct regions were performed. Hardness value was calculated using the following expression:

$$HV30 = \frac{1854P}{d^2} \quad (1)$$

where, $HV30$ is the Vickers hardness, P is the applied load (kg), and d is the arithmetic mean of the two diagonal lengths (mm).

Bi-colored zirconia bars and single-colored bars ($n=10$ per group) were selected for the four-point bending test. Test was carried out in a universal testing machine at a cross-head speed of 0.5 mm/min (Shimadzu AG-IC, Tokyo, Japan). The inner and outer spans of the attachment were 8 mm and 16 mm, respectively. The flexural strength was calculated using the following equation:

$$\sigma = \frac{3PL}{4wb^2} \quad (2)$$

Where, P is the fracture load, L is the outer spans, w and b is the width and thickness of the bar, respectively.

The failure patterns were classified into three categories according to the different fractured positions. A fracture occurring at the color gradient zone in bi-colored samples or the medium zone in single-colored samples was denoted as M, and those occurring in the white and yellow regions were denoted as W and Y, respectively. The fractured surfaces were further investigated by SEM to define the origins.

2.3. Statistical analysis

Grain size, hardness and four-point bending strength were analyzed by a one-way analysis of variance (ANOVA) with Tukey's Multiple Comparison Test to a significance level of $P<0.05$. SPSS 13.0 (SPSS Inc., Chicago, IL, USA) was used to statistically analyze the data.

3. Results and discussion

The selection of the intrinsic components of an FGM is important for the achieved properties and the success of application. Interior residual stresses between the layers deteriorate their bonding and reduce overall strength of the FGM. The interior residual stresses can be introduced by any anisotropic or dissimilar thermal expansion coefficient (TEC) or different sintering shrinkage between the layers. According to the powder manufacture, the two zirconia components in the bi-colored zirconia have similar TEC of $10.5 \times 10^{-6}\,K^{-1}$. Fig. 2 shows the relative shrinkage and shrinkage rate recorded during the dilatometer measurement by heating up to 1450 °C. Doping with a small amount of Fe_2O_3 reduces the temperature of both sintering start (Ts) and maximum shrinkage rate (Tc) around 50 °C, while it does not obviously influence the maximum shrinkage rate. It is evident that Fe_2O_3 act as a

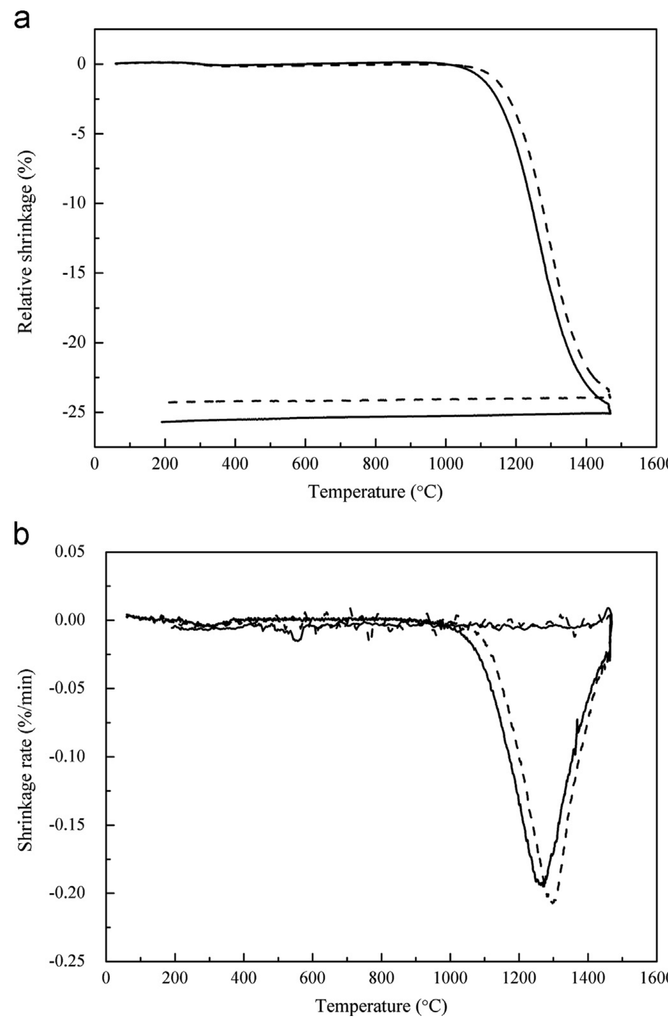

Fig. 2. Dilatometer curves illustrating the sintering properties of the two zirconia powders, TZ-3YSB-E (dash line) and TZ-YELLOW-SBE (solid line), yielding white and yellow color, respectively. The direct measured linear shrinkage versus temperature in (a) and calculated densification rate versus temperature in (b).

sintering aid. This observation agrees with previous findings revealing that the effect upon sintering increases with the concentration of Fe_2O_3 [20]. The eutectic liquid temperature of the ternary ZrO_2–Y_2O_3–Fe_2O_3 system is lower than that of the corresponding binary ZrO_2–Y_2O_3 system. However, the activity of the dopant has not been clarified fully and another idea is the impurity drag mechanism [21]. In present study, the amount of Fe_2O_3 doped into zirconia for coloring was very small (0.202 wt%) and the final difference of relative shrinkage was less than 1%. This difference of linear shrinkage is not expected to lead to high residual stresses. In other words, these two commercial zirconia powders are suitable candidates for fabricating the bi-colored zirconia ceramics.

The mean density of bi-colored 3Y-TZP was 6.018 g/cm^3 (corresponding to a relative density of 98.7%), which is above the critical standard 6.0 g/cm^3 for surgical zirconia ceramics [19]. A light yellow region (~1 mm width) was visually observed at the color gradient zone of the bi-colored zirconia bars. The formation of this narrow zone might be attributed to

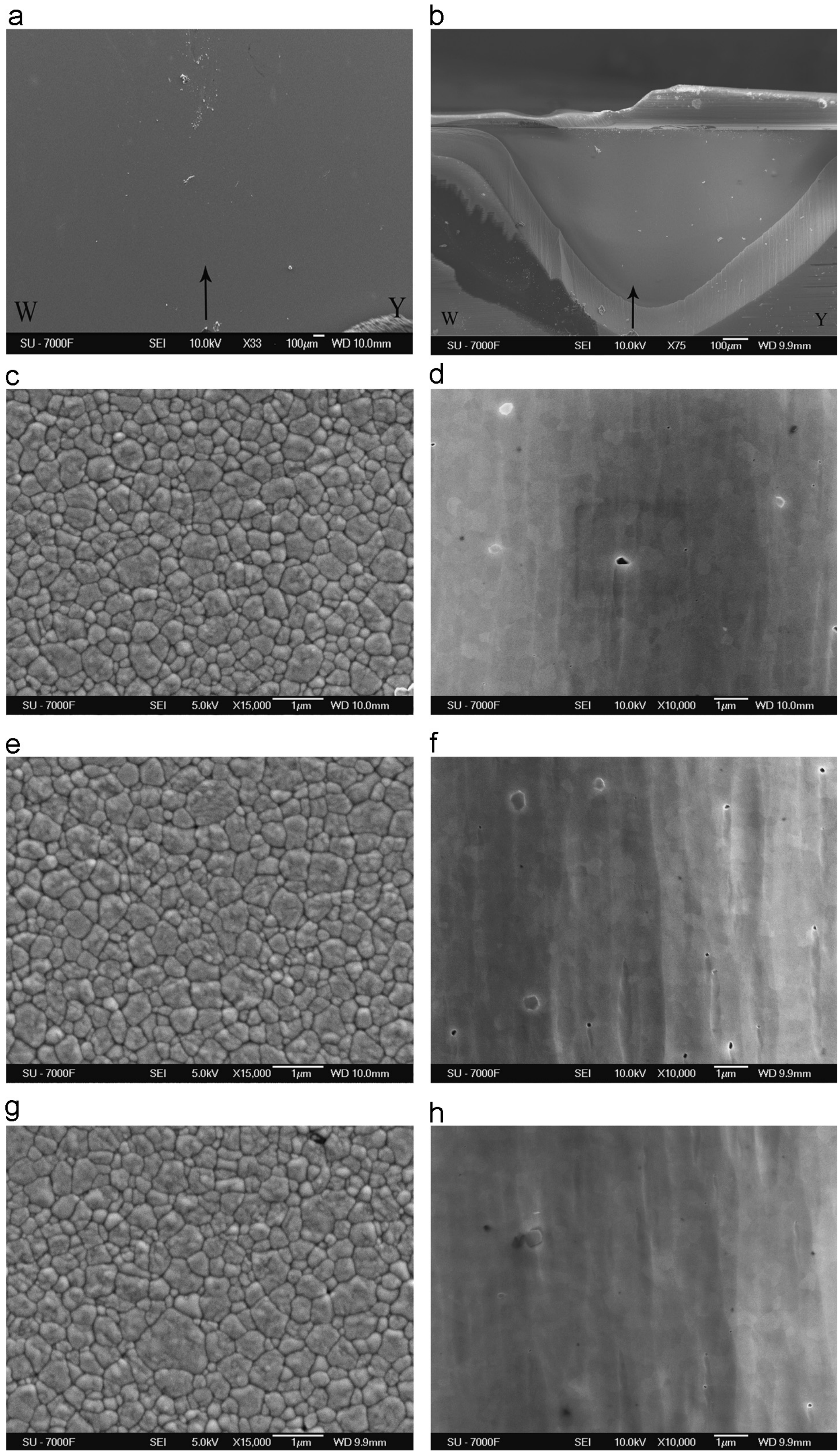

Fig. 3. SEM micrographs showing the microstructure of the bi-colored zirconia ceramics. Low magnification images of the polished surface (a) and cross-section (b) revealing the overall homogeneity of the sample. High magnification images of the polished surface and cross-section taken on the white region (c,d), color gradient zone (e,f) and yellow region (g,h), respectively.

the mixing of the white and yellow powders inside. A flat, but not completely smooth surface was formed after first dry pressing under 2 MPa pressure that was not sufficiently high to deform or to destroy the powder granules [22]. During the second pressing, the concave regions on the interface were filled by another powder and a mixture layer was then generated. The observed color gradient zone also indicates that the diffusion of Fe^{3+} may occur during sintering and contributes to the formation of a gradient light yellow color.

Fig. 3 shows the SEM micrographs of the bi-colored 3Y-TZP samples. The optical color gradient zone cannot be distinguished under SEM either on the surface or on the cross-section, see Fig. 3a and b. Higher magnification micrographs of the surface and cross-section of the white, color gradient and yellow regions are shown pair-wise in Fig. 3c–d, e–f and g–h, respectively. Three regions with different colors exhibited very similar homogeneous microstructure consisting of crystalline grains with very similar morphologies both on the surface and in the interior. No significant difference of grain size existed among the three regions, as revealed by the data summarized in Table 1. It indicates that the addition of a small amount of Fe_2O_3 does not cause noticeable grain growth, in agreement with the previous findings [23,24]. A slight increase of mean grain size with the addition of higher amount of Fe_2O_3 was revealed, as reported in the referred studies. Similarly, limited grain growth was found when zirconia was doped by additional cerium and bismuth [25].

The hardness of bi-layered zirconia was around 13.1 GPa. No significant difference was found among the three regions, as revealed by the data summarized in Table 1. This hardness is in accordance with that reported data of the pure and colored zirconia [26,27]. The hardness is not expected to change significantly even with higher addition of Fe_2O_3 [24]. A slightly lower hardness was noted, however, at the color gradient zone. That might reflect some hidden interior defects, such as granule packing porosity. Presence of micron-sized pores might not affect bending strength, whereas any defect of ten microns or larger will be detrimental. The bending test may reveal a weaker strength if there are critical processing defects in color gradient zone.

Table 1
Grain size and hardness of the three regions of the bi-colored zirconia bars.

	Grain size (*SD*) in nm	Hardness (*SD*) in Hv
White region	326 (33)^a	1339 (4.2)^A
Color gradient zone	354 (37)^a	1332 (9.9)^A
Yellow region	355 (38)^a	1337 (3.6)^A

SD: standard deviation. Means (*SD*) with the same superscript letter in the same column, eg. a or A, are not significantly different, since the *P* value is actually greater than the significance level ($P<0.05$).

The results of the four-point bending test are summarized in Table 2. The mean four-point bending strength of bi-colored zirconia was over 700 MPa, which is higher than that of the other reported dental ceramics [4]. It indicates that the bi-colored zirconia ceramics could meet the requirement of mechanical strength for dental restorations, because the normal biting force, even in the posterior region, is around 400–500 N [28]. Compared to both the white and the single-colored zirconia bars tested in present study, however, the hidden problems of bi-colored zirconia are exposed. The observed strength of bi-colored group is significant lower than that of the other two reference groups, being >1000 MPa. In addition, the frequent fracture was observed to occur in the middle of bi-colored bars at the color gradient zone. It suggests that some critical defects might be introduced during the applied two steps dry pressing procedure of different granules. This is analogous to the loss of hardness in the color gradient zone.

By further fractographic analysis of all failed bi-colored zirconia bars, the fracture origins were found at the tension side, as expected. The SEM micrographs taken on one characteristic test sample containing almost all kinds of defects is presented in Fig. 4. The fracture surface of the white part and of the yellow part is shown in Fig. 4a and b, respectively. The critical fracture origin could be easily identified by tracking the twist hackle markings. These markings are anticipated as the result of local shear stress or structural inhomogeneity [29]. Voids, especially the inter-granular voids with high coordination numbers, are found within the fracture origin. They are a consequence of inhomogeneous granule packing during dry pressing and cannot be completely healed or removed by subsequent CIP-ing and sintering. This packing problem is commonly encountered even in commercial products, but seems to become more detrimental at the interface zone between two combined layers. Besides granule packing defects, other defects were also found in the color gradient zone. Several irregular larger grains with porous surfaces that aggravate structural homogeneity were seen at fracture origins,

Table 2
Results of four-point bending test of three experimental groups and the distribution of fractured patterns ($n=10$ per group).

Group	Four-point bending strength in MPa (*SD*)	Distribution of fractured patterns (%)		
		W	M	Y
Bi-colored zirconia	745.5 (159.6)^a	10	60	30
White zirconia	1049.7 (193.8)^b	90	10	0
Yellow zirconia	1115.6 (243.1)^b	0	40	60

SD: standard deviation. Means (*SD*) with the same superscript letter in the same column, eg. a or b, are not significantly different, since the *P* value is actually greater than the significance level ($P<0.05$). The abbreviation W, M and Y indicates that the fracture occurred in the white, medium or yellow region of the test samples, respectively.

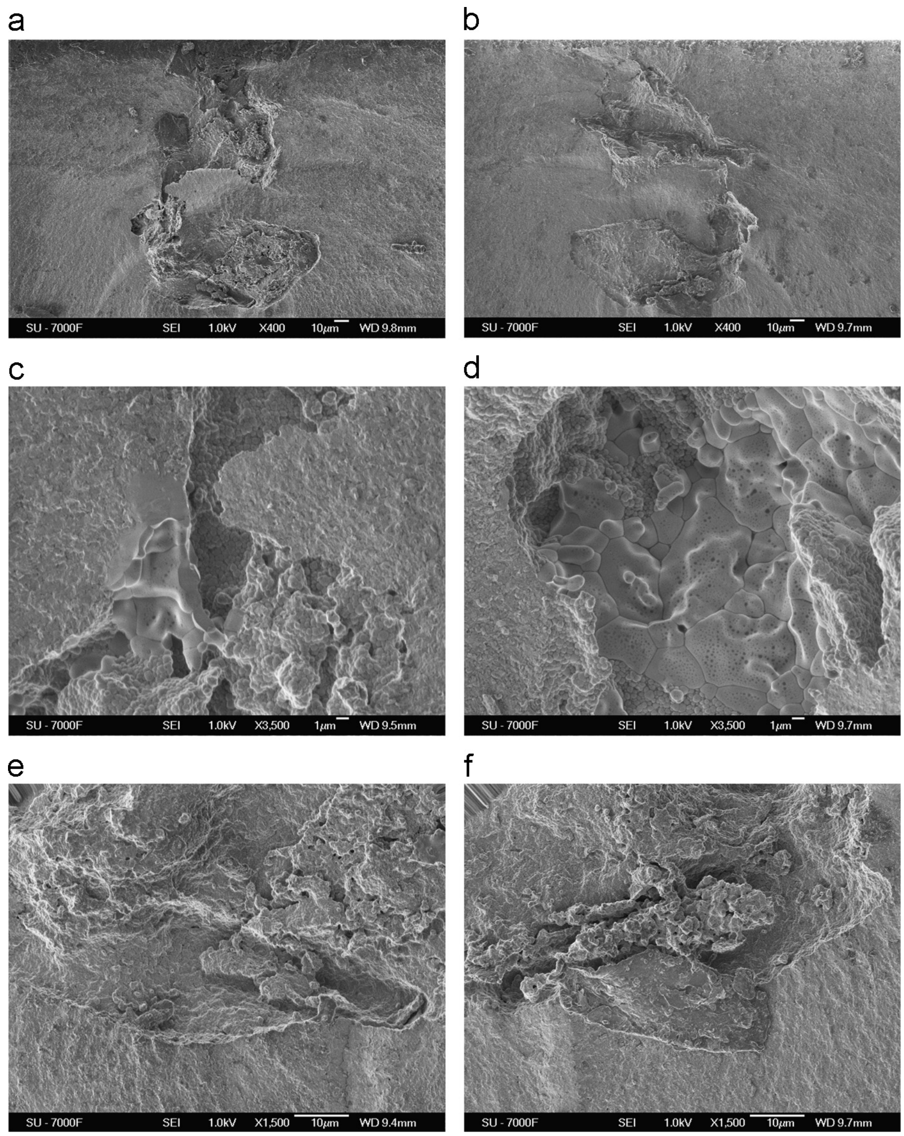

Fig. 4. SEM micrographs revealing the microstructure of a pair of fracture surfaces taken on a bi-colored sample that failed at the color gradient zone during bending test. The twist hackle markings point to the fracture origin in (a) and (b). Irregular grains with porous surfaces (c,d), large voids and critical agglomerates (e,f) are seen as common defects.

cf. Fig. 4c and d. This phenomenon might be ascribed to a local enrichment of the Fe-dopant (Fe_2O_3), as increase of Fe^{3+} could trigger abnormal grain growth [30]. If Fe^{3+} segregates and diffuses at grain boundaries during sintering it may result in small pores on the grain surfaces by pore-boundary separation, in a similar way as seen when zirconia was doped by Ce^{3+} and Bi^{3+} [25]. Any agglomerate or defect exceeding the critical size for dental ceramics, >34 μm, may be equally responsible for a crucial failure in clinic [31]. Agglomerate about 40 μm in size caused a serious chipping-off failure that exposed further packing voids, see Fig. 4e and f. Above all, it is enough with one critical flaw size or the concentration of several connected flaws with similar size together that result in the initiation of fracture and crack propagation through the bulk. In a worst case scenario in clinic applications, the defect is located at a crucial position with high stress concentration, like the thin margin, a connector or upon the surface directly exposed to the oral environment. Unexpected or even catstrophic failures of dental restorations might occur and strongly limit their expected life-time [31]. Therefore, special care is needed to control the homogeneous distribution of coloring additives and to ensure the granulated powders to be free of hard or large agglomerates exceeding critical size. Further, the used immature two-step dry-pressing procedure in this study needs to be improved to minimize the introduced packing defects. Clinical application of this technique to fabricate esthetic zirconia blocks with gradient colors should be carried out in the near future.

4. Conclusions

Through two-step dry-pressing two different zirconia powders having similar compositions, sintering properties and TEC, followed by CIP-ing and pressureless sintering, fully dense bi-colored zirconia ceramics with a gradient color zone were fabricated. The feasibility of this technique for dental application was confirmed since the density, hardness and four-point bending strength were up to the requirements. The color gradient zone in the bi-colored zirconia was a relative weak region mainly due to the critical inhomogeneous microstructure, which probably related with granule packing defects and local enrichment of Fe_2O_3. Therefore, there is still a large opportunity for further improvement of this technique.

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