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The influence of yttria-stabilised zirconia and cerium oxide on the microstructural morphology and properties of a mica glass-ceramic for restorative dental materials

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ABSTRACT

The addition of yttria-stabilized zirconia and cerium oxide to this mica glass ceramic was found to increase mechanical properties and decrease chemical solubility. They were also found to be able to control translucency. X-ray diffraction showed no significant change in phase formation with phlogopite-Ca mica, fluorapatite and tetragonal zirconia the main phases present with their addition. Scanning electron microscopy showed that the additives did affect the grain morphology significantly and this was the controlling factor in the observed changes in strength, hardness, and solubility. The microstructures consisted of mainly plate-like and interlocking crystals. The largest increased in strength and hardness and the largest decreased in chemical solubility can be attributed to the largest change in grain morphology by the addition of both the YSZ and CeO_2. The values of hardness, biaxial flexural strength and chemical solubility were 3.5–6.2 GPa, 105–120 MPa and 142–732 μg/cm^2, respectively making them acceptable for dental materials according to ISO 6872:2015. The addition of YSZ increased the opacity, whilst the CeO_2 improved translucency and influenced the color to a yellowish to yellow-brownish shade close to Thais' teeth.

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Mica-based glass-ceramic; dental ceramics; yttria stabilized zirconia; machinable; iso 6872:2015

1. Introduction

All ceramics for dental restoration must have excellent mechanical properties and a color and translucency close to that of natural teeth. The original color of glass-ceramics is white, so it must be adjusted to match that of natural teeth. Mica glass-ceramic is used as a restorative dental material because it has good biocompatibility and machinability [1,2], which allows fabrication by computer numerical control (CNC) in conjunction with computer-aided design/computer-aided manufacturing (CAD/CAM) systems [3]. Despite these admirable properties it cannot be used for a variety of applications in dentistry because of its relatively low strength. Phlogopite mica glass-ceramic is known under the commercial trademark MACOR [4]. There are several strategies to improve the strength of these mica glass-ceramics including the use of zirconia (ZrO_2) [5]. The addition of yttria-stabilized zirconia (YSZ) results in better mechanical properties, such as hardness, elastic modulus, brittleness index, chemical solubility [6–8] as well as a higher degree of machinability [9]. However, ZrO_2 has the drawback of low translucency and a milky-white color [10]. Therefore, the addition of coloring agents to match a patient's original teeth is essential. Pigment additives or metal ions such as iron (Fe), cerium (Ce) and praseodymium (Pr) are used for brownish, yellowish-brown and dark yellowish colors, respectively [11]. Moreover, zircon-based pigments have been extensively applied in the industry because they are color stable under high firing temperatures [12,13]. Examples are praseodymium zircon yellow (Pr-$ZrSiO_4$), ferrum zircon red (Fe-$ZrSiO_4$), and vanadium zircon blue (V-$ZrSiO_4$) [14,15]. The addition of CeO_2 increases the yellow hues [16], providing a promising candidate for denture replacement materials [17]. The purpose of this study is to investigate the effect of YSZ and CeO_2 addition on the optical properties, chemical composition, microstructural morphology, mechanical properties, and chemical solubility of the mica glass-ceramics.

2. Material and methods

2.1. Preparation of glass-ceramics

Mica glass-ceramics were prepared from a glass frit feedstock of 37%SiO_2, 15.9%MgO, 13.6%$SrCO_3$, 11.9%Al_2O_3, 6.5%$CaCO_3$, 3.0%P_2O_5, and 0.6%CaF_2 as. The glass frit was ground to less than 45 μm. Cerium oxide (< 5 um) and/or YSZ (10–15 nm) were added according to the ratios in Table 1. All chemicals were

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Table 1. Percentage weight of components of the glass-ceramics.

Materials	Glass frit (wt%)	YSZ (wt%)	CeO_2 (wt%)
GC	100	-	-
GCC	99	-	1
GCY	95	5	-
GCCY	94	5	1

homogeneously mixed, in a rolling mill for 2 h. They were melted in a furnace at 1450°C while another furnace was used to heat a carbon mold to the annealing temperature at 582°C for 90 min. The melted glass was poured into a 16 mm diameter by 115 mm long cavity in the heated carbon mold followed by annealing to relieve thermal stresses in the glass rod. The glass rod was then heat-treated in two stages; nucleation at 643°C for 10 min followed by crystallization at 897°C for 10 min both with a 55°C/min heating rate, to transform the glass to glass-ceramics. The heat treatment temperatures were those used in a previous work on these materials [18] and were calculated according to Marotta's regime [19].

2.2. Characterization

Phase formation was examined at room temperature by X-ray diffraction (XRD; X'pert PW 3040/00, Phillips, Netherlands) which each sample, sliced from the cast rod, scanned from 5° 2θ to 80° 2θ with a step size of 0.05° 2θ and a count time of 2 s at 30 kV and 40 mA. All specimens were progressively ground from 600 to 2500 grit with silicon carbide abrasive paper and then polished with 0.1 μm alumina paste and etched by 10% hydrofluoric acid solution for 15 sec. The etched specimens were cleaned and sputtered with gold. The percent crystallinity and aspect ratio of the crystalline glass-ceramics were observed by scanning electron microscope (SEM, JSM-7800 F, JEOL Ltd, Japan) and calculated by the ImageJ/Fiji software [20] by area. Biaxial flexural testing was performed using a piston on three balls method according to ISO 6872: 2015 [21], in this case, the samples were covered with thin plastic to ensure that the load was evenly distributed over all loading surfaces. The hardness was tested by Vickers hardness testing (FM-700, Future-Tech Corp) [22] with a load of 0.3 kg and loading time of 15 sec. Fracture toughness was calculated using the full Charles and Evans equation [23] (equation 1) [24].

$$K_{IC} = 0.016HV(a)^{\frac{1}{2}}\left(\frac{c}{a}\right)^{-\frac{3}{2}} \quad (1)$$

Where H_V is the Vickers hardness (GPa), a is the half diagonal of the indentation (mm) and c is the half distance between the opposition crack tips (mm). In general ceramics, where no data for Young's modulus exists, this equation can be used to determine fracture toughness values at the 20–30% confidence level but when values for Young's modulus are known, better toughness data can be obtained by using the full Charles and Evans equation [24].

The optical properties of contrast ratio (CR) and translucency parameter (TP) were measured on 1 mm thick specimens of the glass-ceramics using colourimeter spectroscopy (XE, Hunter Lab Ultra Scan, Virginia, USA). The specimens were placed over ideal white and black backgrounds [25,26]. The color of the specimens was measured according to the CIE L*a*b* color scale over white and a black background to obtain L*, a*, and b* values for calculation of the translucency parameter. The color difference of the specimens between before and after the addition of YSZ and CeO_2 was measured and calculated as $\sqrt{\left(L_B^* - L_W^*\right)^2 + \left(a_B^* - a_W^*\right)^2 + \left(b_B^* - b_W^*\right)^2}$ where the subscript *B* refers to the color parameters on the black backing and the subscript *W* refers to those on the white [27]. Contrast ratios were determined from the ratio of the reflectance of light from the glass-ceramic on a black surface (Yb) to the reflectance from the glass-ceramic on a white surface (Yw), (Yb/Yw) [28]. The value of contrast ratio ranges from 0 to 1, where 0 means totally transparent and 1 means totally opaque whereas the translucency parameter value is the color difference of the same specimen with variation from 0 to 100. In terms of dental ceramic applications, the translucency is one of the first factors in regulating esthetics and is critical in the selection of materials by dentists [29].

The chemical solubility was measured according to ISO 6872:2015 [21]. Samples of more than 30 cm^2 surface area were separated and polished progressively with silicon carbide abrasive papers from 350 to 2500 grit. They were cleaned by ultrasonics and dried at 150 ± 5°C for 4 h, then weighed. They were exposed to 4% acetic acid solution for 16 hours at 80°C, thoroughly washed, dried at 150 ± 5°C for 4 h and reweighed. The difference in weight was used to describe the chemical solubility in $\mu g/cm^2$.

3. Results and discussion

3.1. XRD analysis

The XRD patterns in Figure 1 identify the crystalline phases present in the glass-ceramics. The slight hump from approximately 20° to 35° 2θ suggest the presence of a residual glassy phase. For all samples phlogopite-Ca ($CaMg_6Al_2Si_6O_{20}F_4$: JCPDS: 025–0155), anorthite ($CaAl_2(SiO_4)_2$, JCPDS: 00–002-0523), fluorapatite ($Ca_5(PO_4)_3F$, JCPDS: 00–060-0667), are the major phases present. For the GCY and GCCY samples, there are the extra peaks of tetragonal zirconia (t-ZrO_2, JCPDS: 00–070-7300). Calcium magnesium silicate ($CaMg_3(SiO_4)_3$, JCPDS: 00–002-0455) is more prevalent in the

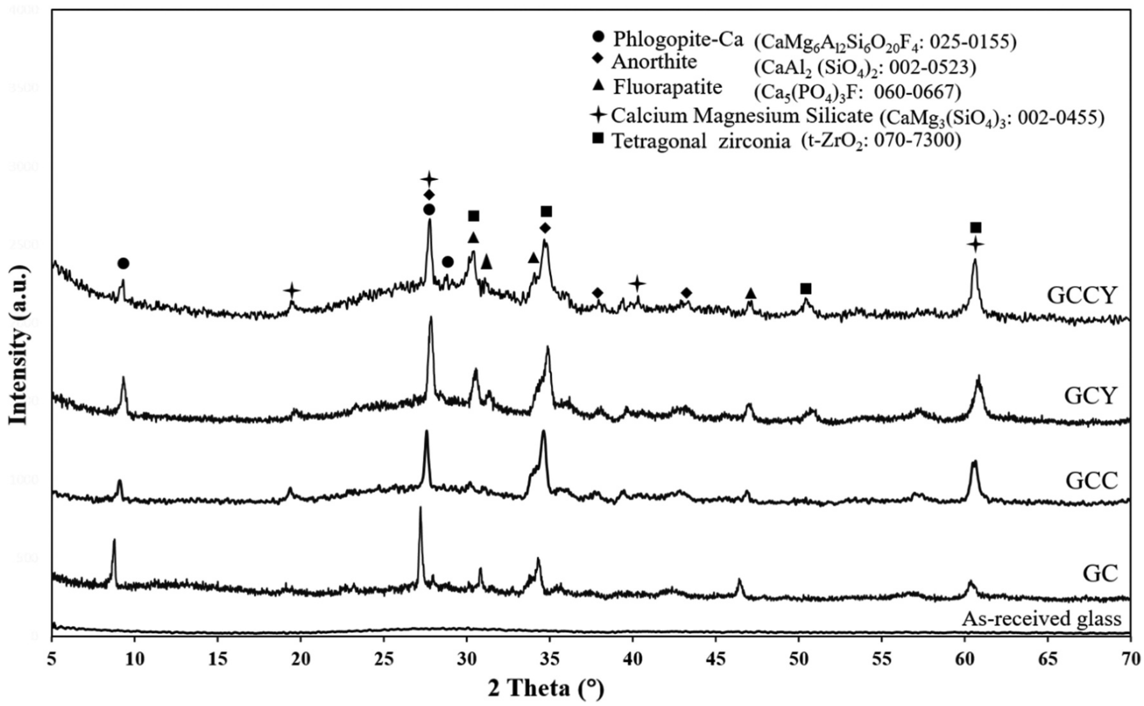

Figure 1. XRD patterns of glass-ceramics crystallized at 897°C.

specimens with CeO_2 added (GCC and GCCY). Ceria performs as a nucleating agent in glass-ceramics [17] and it is a catalyst for $CaMg_3(SiO_4)_3$. In the case of adding YSZ into the glass frits, the appearance of peaks for tetragonal zirconia (t-ZrO_2) are noted. There is no significant difference, other than this, to the phases formed in all specimens.

3.2. Microstructural analysis

The crystalline structure of the original glass-ceramic is shown in Figure 2 and the metrics for morphology are given in Table 2. The addition of the 1% ceria had little effect on the level of crystallinity in these glass-ceramics with only about a 4% increase. The effect on morphology is, however, more pronounced with a 23% decrease in length of the long axis and a 22% decrease in the short axis of the grains resulting in a decrease in the aspect ratio of 27% (Figure 2 (a2) and (b2)). As stated in the previous section CeO_2 can act as a nucleating agent in these glass ceramics and this is exhibited by the observed change in crystal morphology here. The addition of 5% YSZ had a dramatic effect, changing the crystal morphology from tabular to equiaxed with a 77% decrease, from the GC specimens, with the aspect ratio approaching 1 (Figure 2 (a3) and

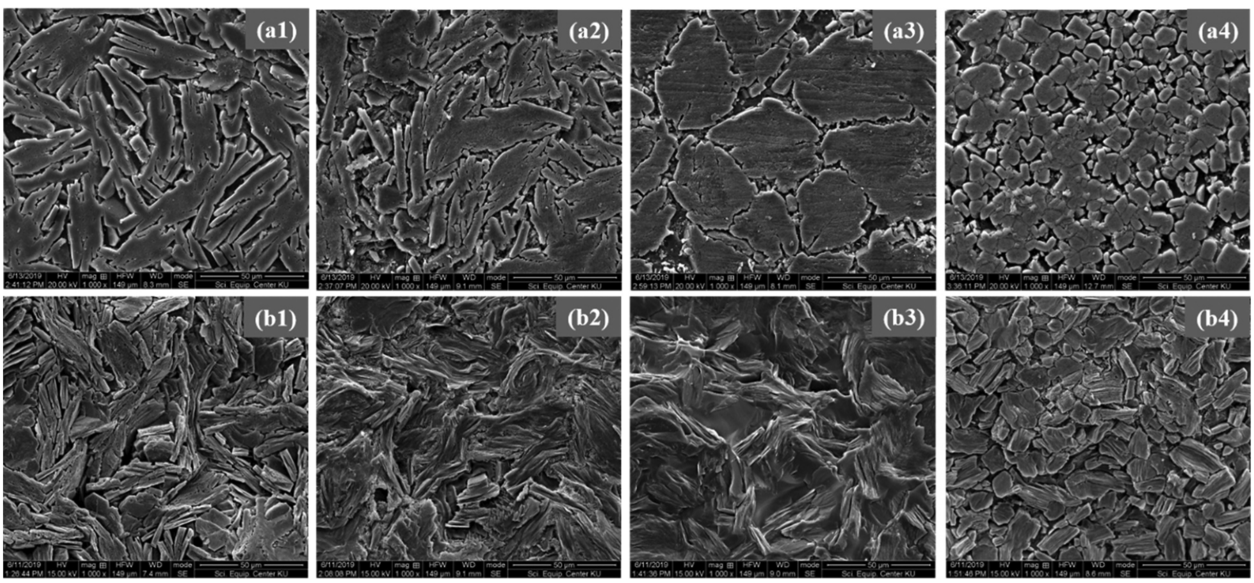

Figure 2. SEM micrographs of (a) the polished and etched surfaces (a1) GC, (a2) GCC, (a3) GCY and (a4) GCCY and (b) the fracture surface of the glass-ceramics (b1) GC, (b2) GCC, (b3) GCY and (b4) GCCY, 1000×.

Table 2. Percentage crystallinity, aspect ratio and crystal size.

Materials	% Crystallinity	Aspect ratio	Crystal size (μm)
GC	87.04	5.45 ± 0.79	-
GCC	90.36	3.98 ± 0.12	-
GCY	89.43	1.23 ± 0.13	~ 36.5
GCCY	86.67	1.26 ± 0.34	~ 7.8

(b3)). The addition of YSZ has been shown to slow the crystal growth kinetics in these glass ceramics slowing the cooling and allowing the growth of more equiaxed crystals. The addition of both 1% CeO to the specimens with 5% YSZ had, not surprisingly, the effect of a large decrease in the mica crystal size, from □36.5 μm in GCY to □7.8 μm in GCCY, (≈80%) further, with no change in morphology (Figure 2 (a4) and (b4)).

The fracture surfaces revealed interlocking plate-like crystals with the crack path appearing to be mainly in the thin glassy phase. Some evidence of crystal fracture is observed. The difference in aspect ratios again observed in these fractographs with the GC and GCC materials showed large plate-like crystals with high aspect ratios while the GCY and GCCY low aspect ratios. The added YSZ particles can act as a most efficient nucleating agent to promote nucleation of mica crystals.

3.3. Optical properties

The color of a dental restorative material should have a yellow to the reddish-yellow gradient to be acceptable. Photographs, showing the difference in color and translucency, of the glass ceramics produced are shown in Figure 3. The translucency parameter indicates the reflected color difference, over white and black backgrounds, between materials with uniform thickness. It is considered to give the best measurement of human perception of translucency [30]. It is specified as one of the primary factors in controlling esthetics and used for the selection of materials for dental applications [31]. A high translucency parameter (TP) indicates that the material is less opaque. A material with a TP of 100 is considered transparent; a TP of 0 indicates that the material is opaque. In addition, the contrast ratio, another measure of translucency, was measured so as to compare results with the most recent literature [18]. The mean of contrast ratio and translucency parameter are reported in Table 2. After the heat treatment process, the color of the resulting glass-ceramic changed with the CeO_2 additions producing a yellowish hue [32] and the addition of YSZ producing a whiter material [17,33] (Figure 3).

The addition of YSZ also caused a higher opacity [34,35], especially without CeO_2 additions. The general transparency and opacity are influenced by the number and/or grain size of crystals [36]. For the GCC and GCY specimens the addition of YSZ and CeO_2 had the effect of slightly decreasing the contrast ratio for the former and slightly increasing it for the latter. Comparison of Tables 2 and 3, as well as reference to Figure 3, can help explain the observed effects on the optical properties measured. The addition of CeO_2 (GC-GCC) to the glass ceramics caused a decrease in average crystal size with a concomitant increase in translucency (a decrease in the contrast ratio and an increase in the translucency parameter). Many studies have described how a smaller mean grain size has led to an enhanced translucency [37–39]. The addition of CeO_2 has been found to improve translucency giving a higher translucency parameter [40].

With the addition of YSZ to the base GC, a small decrease in average grain size might expect no significant difference in translucency, which is the case here, see Figure 3 where there is little difference in opacity, though the GCY specimens appears whiter. If ZrO_2 is used as a nucleating agent it forms a whiter glass-ceramic after the heat-treatment process [17,33].

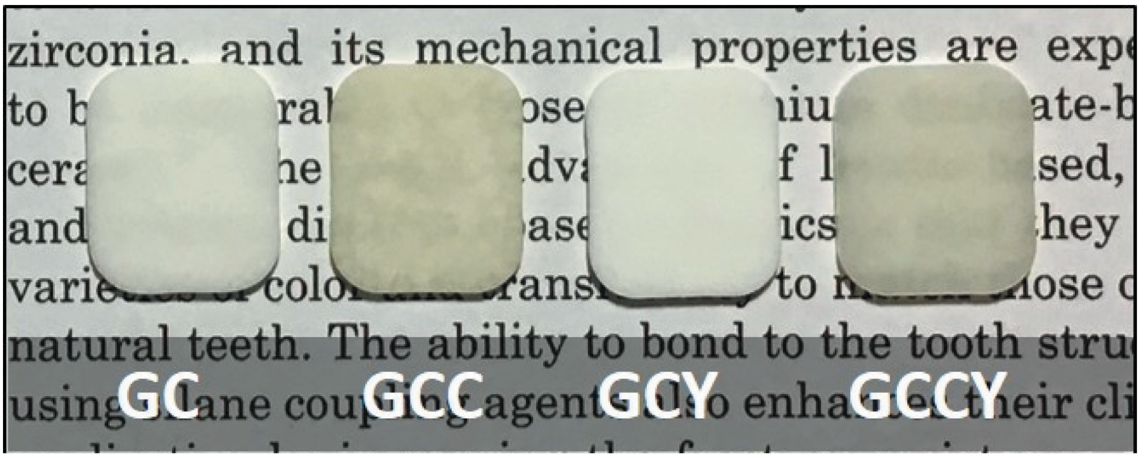

Figure 3. Optical appearance for 1 mm thick glass-ceramics.

Table 3. L*a*b*, contrast ratio and translucency of the glass-ceramics.

Materials	ΔL*	Δa*	Δb*	Contrast ratio	Translucency
GC	−10.643	−3.53	−0.13	0.89 ± 0.00	0. 40 ± 0.12
GCC	−5.67	−4.08	−6.83	0.75 ± 0.00	11.07 ± 0.09
GCY	−9.67	−3.87	−0.28	0.94 ± 0.00	2.40 ± 0.27
GCCY	−4.32	−5.89	−7.64	0.45 ± 0.01	12.87 ± 0.27

For the GCCY material there was a large decrease in grain size and shape. This, together with the addition of the CeO_2, resulted in a large increase in translucency over the GC and GCY materials. Altering the grains to a more uniform size and morphology and reducing the number and size of the porosity can lead to improvement in translucency [24]. A more moderate increase over the GCC materials is observed suggesting that the addition of CeO_2 has more of an impact on translucency than the grain size.

Regarding the contrast ratio, GCCY had the lowest value with a higher translucency than the other materials, as just discussed. Contrast ratios and translucency parameters are considered important variables for dental ceramics selection and are well-established parameters [27,41]. However, in the end, the color and translucency matching must be decided by the dentist and the patient in consultation. The contrast ratios, for 1 mm thick specimens, have values close to commercially available prosthetics [42]. Comparison of these materials to commercially available materials is important. Table 4 provides such a comparison. The GC material (0.89) is like nCoris AL (0.89), GCC (0.75) close to IPS e.max ZirCADv (0.78) and IPS e.max MO (0.71), GCY (0.94) close to In-Ceram Zirconia (1.00) and GCCY (0.45) close to IPS e.max HT (0.48) (Table 4). For natural teeth, the contrast ratio of human enamel and dentine are 0.45 and 0.65, respectively [43] (Table 4). The translucency parameters also showed comparable values to that of commercial materials [44]; c.f. GCC (11.07) and GCCY (12.87) with Ivoclar IPS e.max Zir-CAD (11.48) and Sirona inCoris ZI (12.64). The mean translucency values of 1 mm thick human enamel and dentin are 18.7, and 16.4, respectively [45]. The combination of CeO_2 and YSZ (GCCY) shows a contrast ratio and a translucency parameter similar to those of human enamel and other commercially available materials. The GCCY material had the smallest crystal size as in Figure 2 (b4), therefore, it exhibited more translucency and CeO_2 influenced the color of yellowish and yellow-brownish shades close to natural teeth.

Table 5. Chemical solubility of glass-ceramics.

Materials	Chemical solubility (μg/cm^2)
GC	732.37 ± 30.18
GCC	674.30 ± 37.25
GCY	499.34 ± 38.74
GCCY	142.25 ± 20.88

3.4. Mechanical properties

The YSZ and CeO_2 additives caused an increase in strength and microhardness. For GC and GCC, the similar microstructures resulted in similar biaxial flexural strengths and hardness (Figure 4). The more pronounced change in crystal morphology discussed in section 3.2 resulted in a more pronounced effect on hardness with a smaller effect of strength. The propagation of a crack front depends on microstructural factors such as crystal size, crystal shape, the aspect ratio of the crystal and crystallinity [46]. As the per cent crystallinity is hardly affected, there would be little effect from this on the observed strengths. The fracture front in these materials tend to travel through the more brittle glassy phase between the crystals. Inspection of Figure 2 (a) shows that the thickness of the glassy phase decreases slightly. The crystal size does decrease but not significantly. These two factors may suggest why no large increase in strength was observed even for the GCCY material where a large decrease in crystal size was observed. Higher aspect ratios for these glass ceramics has been found to cause lower strengths [47]. This is supported by the results given here.

Table 4. The contrast ratio was compared between materials in research with commercial materials and natural teeth.

Materials in research	Contrast ratio	Commercial Materials [42]	Contrast ratio	Part of the natural tooth [43]	Contrast ratio
GC	0.89	In-Ceram Zirconia	1.00	Dentine	0.65
GCC	0.75	nCoris AL	0.89	Enamel	0.45
GCY	0.94	IPS e.max ZirCADv	0.78		
GCCY	0.45	IPS e.max MO	0.71		
		IPS e.max HT	0.48		

Hardness was affected by the aspect ratio of the crystals, the crystallinity and spatial area of the grains [47]. Mica is a relatively soft crystal when compared to the glass. The indent size is comparable to the grain size for the GC and GCC materials, with a resultant little difference in hardness. With the change in morphology on the addition of the YSZ, hardness increased and for the GCCY materials a very large increase in hardness was observed. This is due to the closer spacing of the harder, glassy phase between the softer crystals causing more resistance to indent penetration. The much smaller crystals in the GCY and GCCY specimens may also be harder simply due to their reduced size. The range of biaxial strengths and hardness were 105–120 MPa and 2.4–6.5 GPa. This makes them acceptable as restorative dental materials according to ISO 6872 Type 2 Class 1 and 2.

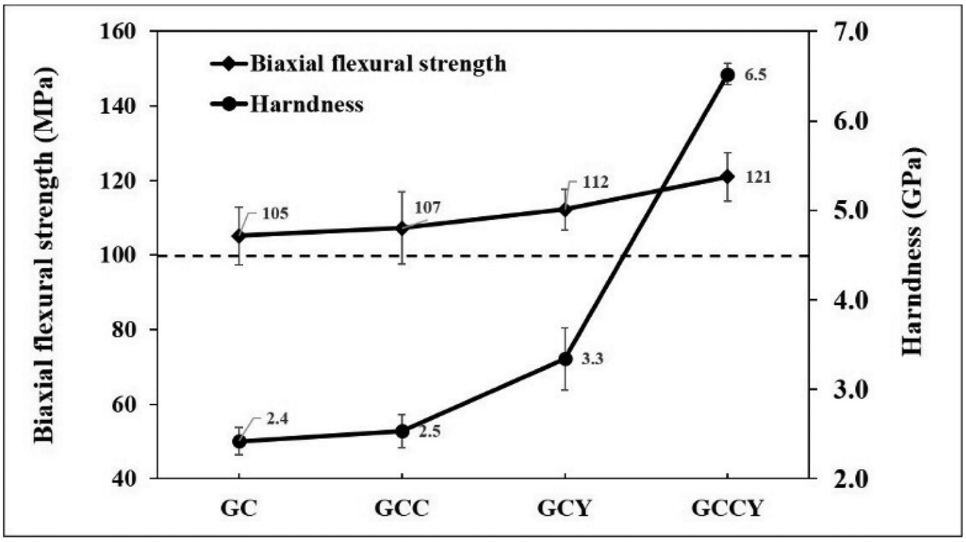

Figure 4. Biaxial flexural strength and hardness results of the glass-ceramics.

Figure 5 show micro indentation cracking of the mica glass-ceramics Inspection of Figure 5 shows that determination of the crack lengths for fracture toughness calculation is not possible. For the GCCY specimens, the cracks were able to be measured resulting in the determination of a fracture toughness of 2.7 MPa□m$^{1/2}$. This makes this material acceptable as restorative dental material according to ISO 6872 Type 2 Class 1 and 2.

3.5. Chemical solubility

In general, the amorphous phases are more soluble than the crystalline phases [48]. The actual crystalline phase will also affect solubility. Comparison of Tables 2 and 5 shows no clear correlation between the level of crystallinity and chemical solubility in the materials studied here. However, as stated previously, the thickness of the glassy phase reduced which may affect the access of the solvent. However, it should be considered that the crystalline phases present also contribute to controlling solubility in these glass-ceramics. A significant decrease in solubility was observed with the addition of the YSZ suggesting the lower chemical solubility may be attributed to the presence of the tetragonal zirconia phase which presents higher resistance to chemical solubility than the fluorapatite and other phases [7,18,34]. The GCCY material with the

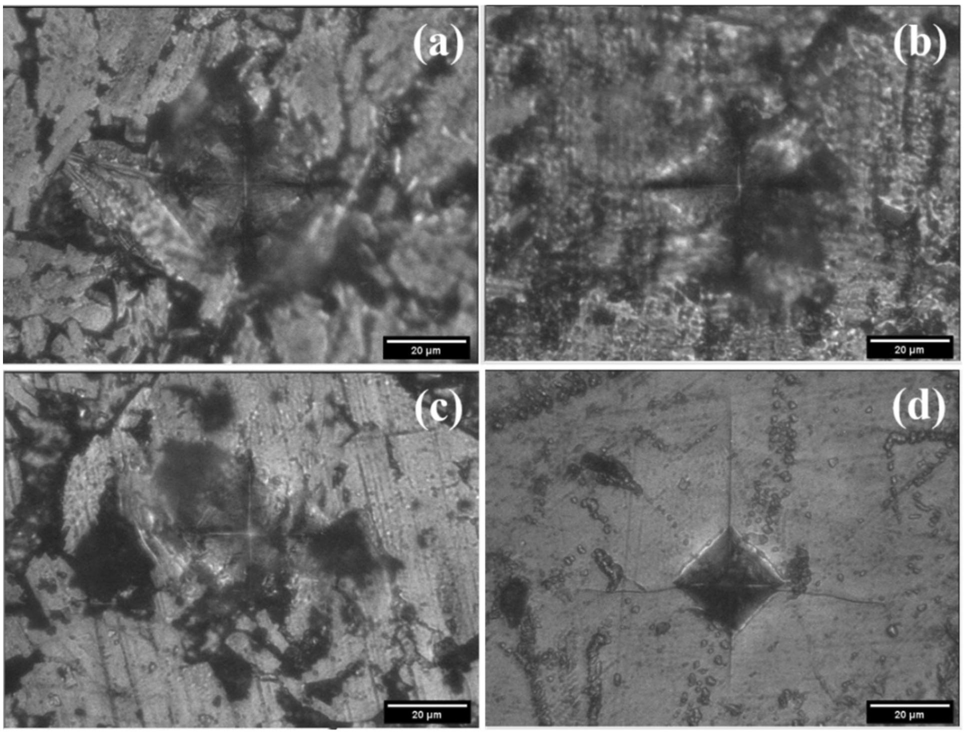

Figure 5. Microhardness indentations in glass-ceramics (a) GC, (b) GCC, (c) GCY and (d) GCCY, 50x.

lowest chemical solubility of 142–732 µg/cm^2, is acceptable as a dental material according to ISO 6872 Type 2 Class 2b [21].

4. Conclusions

The addition of YSZ and CeO_2 have affected the biaxial strength, hardness, optical and chemical properties of the glass ceramic used in this study. Their addition did not significantly affect the phases formed. In general, the addition of these materials increased mechanical properties and reduced chemical solubility. The largest increased in mechanical properties and the largest decreased in solubility was from the combined addition. These changes can be attributed to the change in grain morphology introduced by the additives. Translucency was increased by the addition of YSZ and decreased by the addition of CeO_2 in line with other studies.

All the glass-ceramics developed here can be used as restorative dental materials according to ISO 6872 Type 2 Class 2b (partially or fully covered substructure ceramic for single-unit anterior or posterior prostheses adhesively cemented) [21]. This good combination of properties and esthetics made these mica ceramics great candidates for a dental ceramic.

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Disclosure of potential conflicts of interest

No potential conflict of interest was reported by the author(s).

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