Crystallization and thermo-mechanical properties of Li$_2$O-ZnO-CaO-SiO$_2$ glass-ceramics with In$_2$O$_3$ and Fe$_2$O$_3$ additives

Saad M. Salman, Samia N. Salama, Ebrahim A. Mahdy*
Glass Research Department, National Research Centre, Dokki, Cairo, Egypt

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Abstract

Li$_2$O-ZnO-CaO-SiO$_2$ based glasses were prepared by the conventional melting technique and subsequently converted to glass-ceramics by controlled crystallization. The nucleation and crystallization temperatures were determined by differential thermal analysis (DTA). The effects of adding In$_2$O$_3$ and Fe$_2$O$_3$ addition on the crystallization behaviour and thermo-mechanical properties of the prepared glass-ceramics were investigated. A study on the microstructure, close to the internal phases of the resulting glass-ceramics, was followed by using scanning electron microscope (SEM). The dilatometric thermal expansion and Vickers’ microhardness of the crystalline products were also evaluated. The crystalline phases that can be found in the resulting glass-ceramics, identified by X-ray diffraction (XRD) analysis, are α-quartz-[SiO$_2$], lithium zinc silicate-[Li$_2$ZnSiO$_4$], lithium disilicate-[Li$_2$Si$_2$O$_5$], wollastonite-[CaSiO$_3$], wollastonite containing iron, ferrobustamite-[(Ca$_{0.79}$Fe$_{0.21}$)SiO$_3$], and lithium indium silicate of pyroxene type-[LiInSi$_2$O$_6$]. Average thermal expansion coefficient (in the temperature range 25–700 °C) decreased from 191 × 10$^{-7}$ 1/°C to 115 × 10$^{-7}$ 1/°C and the Vickers’ microhardness increased from 3.56 to 5.44 GPa with the increase of In$_2$O$_3$ and Fe$_2$O$_3$ contents in the glass-ceramics. The changes in the obtained expansion coefficient and microhardness were due to the formation of different phases which in turn influenced the rigidity/bonding and microstructure in the resultant glass-ceramics.

Keywords: lithium zinc calcium silicate glass-ceramics, microstructure, hardness, thermal expansion

I. Introduction

Glass-ceramics are the micro- or nanocrystalline materials produced by controlling crystallization of glasses [1], in which the crystalline phases are nucleated and grown in glass via heat treatment [2]. It is believed that the first stage in glass-ceramic formation is generally an amorphous phase separation as a result of micro- or nano-scale immiscibility in glass, with subsequent growth occurring in these regions [1]. The properties of glass-ceramics depend on the type and amount of the crystalline phases present, as well as the composition of the glassy matrix [3]. The ability to control these two parameters depends on the original composition and the heat-treatment regime [4]. Glass-ceramics show favourable chemical, thermal, dielectric and biological properties superior to metals and various polymers. Furthermore, they are superior to normal glasses and conventional ceramics as well, especially in their tuneable thermo-physical properties and microstructure [4].

Glass-ceramics from the lithium zinc silicate, Lzs, system are a versatile class of material. There have been thoroughly studied not only because it can be prepared with a wide range of thermal expansion coefficients by controlling heat treatments [5], but also because some compositions within this system can yield useful glass-ceramics, in which orthosilicate can be crystallized. In this system, the study of the effect of substitutions with different cations can throw considerable light on the crystallization behaviour, formation of stable and metastable phases and compatibility relations between the different phases [6]. A number of studies have been carried out on the crystallization behaviour and thermo-physical properties of this system [7–10]. The influence of K$_2$O and P$_2$O$_5$ on crystallization sequence of Li$_2$O-ZnO-SiO$_2$ glass-ceramics containing low ZnO content was investigated by Chen and McMillan [9]. They confirmed that a small amount of

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*Corresponding author: tel: +201068704325, fax: +201068202333, e-mail: emahdy10@yahoo.com.
K$_2$O changed the crystallization sequence and the phase composition of resulting glass-ceramics, while P$_2$O$_5$ produced a glass-ceramics with fine grained interlocking microstructures. West and Glasser [7] reported that, the introduction of calcium in Li$_2$O-ZnO-SiO$_2$ glass-ceramics at the expense of ZnO led to the formation of calcium monosilicate-wollastonite phase together with ceramics at the expense of ZnO. With crystalline phases, changing the ZnO content in the conversion of glass to glass-ceramic. With increasing microstructures. West and Glasser [7] reported that, the prepared samples were immediately melted several times at about 30 min intervals. The melts were mixed and melted in Pt–2% Rh crucible in an electric furnace with SiC heating elements at 1300–1350 °C for 3 h. Melting was continued until clear homogeneous melt was obtained; this was achieved by swirling the melt several times at about 30 min intervals. The melts were cast in warm stainless steel molds into rods and buttons shape. The prepared samples were immediately transferred to an annealing muffle furnace regulated at 450 °C. The furnace was switched off after 1 h and left to cool down to room temperature to minimize the strain of the prepared glasses.

2.2. Crystallization and glass-ceramic formation

Differential thermal analysis (DTA) was employed for recording the glass transition temperature ($T_g$) and crystallization temperature ($T_c$) for all glass samples. Based on the DTA results, the controlled heat-treatment was applied for each glass with the goal to obtain glass-ceramic materials of holocrystalline mass with minimum residual glassy phase and without deformation. Therefore, double stage heat-treatment regimes, were used for crystallization. The glasses were first heat treated in a muffle furnace at endothermic temperature of each glass composition - the glass sample was soaked for 5 h to provide sufficient nucleation sites. The temperature was then raised to the exothermic temperature, specific for each glass for 10 h. A heating rate of 10 °C/min was used during the double stage heat-treatment regimes.

2.3. Characterization

A differential thermal analyser (SDTQ600-IT Instruments, USA) was employed for recording the glass transition temperature ($T_g$) and crystallization temperature ($T_c$). Measurements were done in the temperature range of 25–1000 °C using a heating rate of 10 °C/min from ambient temperature to 1000 °C in a flowing high purity nitrogen environment. The cast glass was crushed and sieved in 90–125 μm to produce (30 g) glass powder suitable for DTA measurement.

Identification of various crystalline phases in glass-ceramic samples were carried out using powder X-ray diffractometer (PW 1830; PANalytical) with Ni filtered Cu-Kα radiation, 2θ range from 5° to 80° range and scanning speed of 2°/min. The crystalline phases were identified by matching the peak positions of the intense peaks with PDF card standard cards.

The fractured surfaces of the glass-ceramic samples were examined by scanning electron microscope (JEOL type JXA-840A Electron Probe Microanalyzer operated at 30 kV). The fractured surface of the samples was eroded with 1% (HF+HNO$_3$) solution for 60 s, and then sputtered with gold.

The thermal expansion coefficients ($\alpha$) of the crystalline samples were measured by using a thermo-

III. Results

3.1. Crystallization behaviour

The glass transition \( T_g \) and crystallization temperature \( T_c \) of each glass sample, deduced from the DTA results of each glass powder, are present in Figs. 1 and 2. The glass samples have a single endothermic dip before the onset of crystallization, which corresponds to the \( T_g \) with the inflection point situated between 482 and 510 °C. The exothermic peaks corresponding to the crystallization temperature \( T_c \) are observed in the temperature range 622–763 °C which indicates that the crystallization reaction takes place in the glass. From the DTA thermographs (Fig. 1), the additions of \( \text{In}_2\text{O}_3 \) in the base glass composition led to increase the temperatures which are needed to start the nucleation stage and crystal growth. While, the additions of iron oxide in \( \text{In}_2\text{O}_3 \)-containing glass (Fig. 2), led to shift the endothermic and exothermic dips to lower temperatures i.e. a lower energy is needed to obtain crystallization in the glasses.

The X-ray diffraction patterns of the thermally treated glasses are presented in Figs. 3 and 4. The XRD analysis of the base glass \( G_1 \) crystallized at 480° C/5 h—

mechanical analyser (NETZSCH DIL402PC) in the temperature range 25–700 °C with a heating rate of 5 °C/min using silica probe. The linear thermal expansion coefficient \( (\alpha) \) was automatically calculated using the general equation: \( \alpha = (\Delta L/L) \cdot (1/\Delta T) \); where: \( \Delta L \) is the increase in length, \( L \) is the original length of the specimen and \( \Delta T \) is the temperature interval over which the sample is heated.

A microhardness tester (Shimadzu, Type-HMV, Japan) with applying loads of 100 g for 15 s was used to carry out the Vickers’ microhardness measurements of the glass-ceramic samples. The samples were cut using a low speed diamond saw after controlled heat treatment, the specimens were carefully polished using 500, 800, and 1000 grit SiC paper and 3 and 1 mm diamond paste to obtain smooth and flat parallel surface glass-ceramic samples before indentation. At least 10 indentation readings for each sample were tested to get average value.

Figure 1. DTA data of the investigated glasses (G₁–G₄)

Figure 2. DTA data of the investigated glasses (G₄–G₆)
745 °C/10 h (Fig. 3, pattern I, Table 2), showed that α-quartz (SiO₂, Card No. 33-1161) crystallized together with lithium disilicate (Li₂Si₂O₅, Card No. 17-0447), wollastonite (CaSiO₃, Card No. 29-0372), and lithium zinc silicate (Li₂ZnSiO₄, Card No. 15-56) phases. The addition of small amount of In₂O₃ (0.20 mol%) in the base glass G₂ heat treated at 490 °C/5 h–745 °C/10 h (Fig. 3, pattern II, Table 2), does not lead to the formation of different phases which were obtained in the crystallized base glass G₁ (In₂O₃-free sample). However, the increase of In₂O₃ addition (0.61–1.01 mol%) in the glasses (the samples G₃ and G₄, heat treated at 495 °C/5 h–755 °C/10 h and 510 °C/5 h–765 °C/10 h respectively) led to the crystallization of pyroxene like phase, such as lithium indium silicate (LiInSi₂O₆, Card No. 33-0799) together with α-quartz, lithium disilicate, wollastonite and lithium zinc silicate (Fig. 3, patterns III–IV, Table 2).

The microstructure of the glass-ceramics was investigated by scanning electron microscopy. SEM of the fractured surface shows the effects of adding In₂O₃ and Fe₂O₃ on the microstructure of the prepared glass-ceramics (Fig. 5). SEM of the crystallized base glass G₁ (without In₂O₃ and Fe₂O₃), showed that volume crystallization of dendritic-like growths were formed (Fig. 5a).

Table 2. The crystalline phases developed in the studied glass-ceramics

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heat-treatment [°C/h]</th>
<th>Crystalline phases formed</th>
</tr>
</thead>
<tbody>
<tr>
<td>G₁</td>
<td>480/5–745/10</td>
<td>α-Quartz (SiO₂) + LS₂ (Li₂Si₂O₅) + Wo (CaSiO₃) + LZS (Li₂ZnSiO₄)</td>
</tr>
<tr>
<td>G₂</td>
<td>490/5–745/10</td>
<td>α-Quartz + LS₂ + Wo + LNZs</td>
</tr>
<tr>
<td>G₃</td>
<td>495/5–755/10</td>
<td>α-Quartz + LS₂ + Wo + LNZs + LiInS (LiInSi₂O₆)</td>
</tr>
<tr>
<td>G₄</td>
<td>510/5–765/10</td>
<td>α-Quartz + LS₂ + LNZs + Wo + LiInS</td>
</tr>
<tr>
<td>G₅</td>
<td>505/5–760/10</td>
<td>LNZs + α-Quartz + LS₂ + Wo (containing iron) + LiInS S</td>
</tr>
<tr>
<td>G₆</td>
<td>485/5–740/10</td>
<td>LNZs + α-Quartz + Fbus. ((Ca₀.79Fe₀.21)SiO₃) + LS₂ + LiInS</td>
</tr>
</tbody>
</table>
Figure 5. SEM micrograph of fracture surface of glass-ceramics: a) G_1 crystallized at 480 °C/5 h–745 °C/10 h, b) G_4 crystallized at 510 °C/5 h–765 °C/10 h and c) G_6 crystallized at 485 °C/5 h–740 °C/10 h

However, the addition of In_2O_3 in the glass composition led to form a volume crystallization of fine grained-like growths (Fig. 5b), and ultra-fine microstructure was obtained by adding iron oxide in the In_2O_3 containing glass-ceramic sample (Fig. 5c).

3.2. Thermo-mechanical properties

The measurements of the coefficients of thermal expansion (α) of the studied glass-ceramic materials were carried out in the temperature range 25–700 °C. The obtained data are graphically represented in Fig. 6. The obtained data clearly indicated that the thermal expansion coefficients decreased from 191×10^-7 1/°C to 150×10^-7 1/°C (in the temperature range 25–700°C) when In_2O_3 was added in the glass compositions (the samples G_2–G_4). Also, the additions of iron oxide in In_2O_3-containing glass (the glass-ceramic samples G_5 and G_6) led to decrease the α-values of the resultant crystalline materials from 141×10^-7 1/°C to 115×10^-7 1/°C in the temperature range 25–700°C (Fig. 6).

The Vickers’ microhardness was determined for all prepared glass-ceramics and the obtained results are presented in Fig. 7. The microhardness values of the investigated samples are ranging from 3.56 to 5.44 GPa. The lowest microhardness value has the base crystalline sample G_1 (Fig. 7) and the additions of In_2O_3 or Fe_2O_3 in the glass compositions led to increase the microhardness values of the investigated crystalline products (Fig. 7).

IV. Discussion

4.1. Crystallization – DTA analysis

The increase in both T_g and T_c temperatures with increasing the In_2O_3 content in Zn-bearing glass (Fig. 1) may be related to the increase in coherence of the glass which consequently leads to less mobility of the structural elements in the glass structure and strengthening the glass network structure [18]. The increase of the temperature needed for the crystallization process was expected. The results also showed that the additions of Fe_2O_3 in the In_2O_3-containing glass have significant effects on lowering the T_g and T_c temperatures (Fig. 2). The number of non-bridging oxygen (NBO) increased with increasing the Fe_2O_3 content in the glasses, such increase led to decrease the temperature of endothermic peaks, i.e. a low energy is needed to induce crystallization. Li et al. [19] reported that iron oxide acted as a flux and phase-separation accelerator during melting. Fayon et al. [20–23] stated that Fe^{3+} ions are expected to occupy both tetrahedral and octahedral local sites positions in the glass network. Both of these Fe^{3+} sites formed the linkages of Si–O–Fe and In–O–Fe type. The divalent iron ions are expected to occupy only interstitial positions and act as modifiers, similar to lithium ions, depolymerizing the glass network by creating more bonding defects and non-bridging oxygens (NBO) [24]. The FeO_4 tetrahedral had a negative charge and hence required cations like Li^+ ions for charge compensation.
4.2. Crystallization – XRD data

The XRD patterns of the glass-ceramics showed the formation of various crystalline phases (Figs. 3 and 4). The type and proportions of the various crystalline phases in the prepared thermally treated glasses were markedly dependent on the composition of the parent glass and heat treatment applied. The X-ray diffraction analysis (Fig. 3, Pattern I) indicated that α-quartz, lithium disilicate, wollastonite and lithium zinc silicate were developed as being major phases from the base glass G1. West and Glasser [7,8] examined the stable and metastable phase relations in the Li2O-ZnO-SiO2 system depending on the composition. They found that, lithium disilicate, lithium metasilicate, lithium zinc silicate and silica can be present as being major phases in this type of glass-ceramics. These phases are characterized by their high or moderately high thermal expansion coefficients making the glass-ceramic suitable for seals. Salman et al. [25] reported that, in the Li2O-ZnO-SiO2 system, Li2ZnSiO4 phase was developed due to the reaction of ZnO with Li2SiO3. The formation of lithium disilicate (LD) is preceded by the crystallization of lithium metasilicate (LS). First, lithium metasilicate crystallizes in the glass at temperatures in the range of 650–700 °C and then reacts with SiO2 to form LD at higher temperatures.

The XRD analysis (Fig. 3, Pattern II) revealed that no In2O3-containing phases could be identified in the sample G2 with low In2O3 addition (0.2 mol%), however, the pyroxene member of LiInSi2O6 phase crystallized as a result of increasing In2O3 content up to 0.61 mol% (Fig. 3, Pattern III). Further increase of In2O3 addition up to 1.01 mol% (the sample G4) led to increase the crystallization of lithium indium pyroxene phase together with α-quartz, lithium disilicate, wollastonite and lithium zinc silicate (Fig. 3, Pattern IV). The present results suggest that the increase of In2O3 content enhances the development of LiInSi2O6 phase of pyroxene-type.

The state of iron, its coordination and concentration in the glasses mainly determined the nature of the phases formed [26,27]. The iron content has a great influence on the oxidation state of iron oxide in glass-ceramic. The ferrous cations occupy only octahedral sites which are increased in the low iron-content compositions [28]. At low Fe2O3 addition (1.04 mol%) in In2O3-containing glass G5 lithium zinc silicate, α-quartz, lithium disilicate, wollastonite and lithium indium silicate phases predominantly crystallized and no Fe2O3-containing phases could be detected. However, the displacement of the major characteristics d-spacing lines of wollastonite phase towards higher 2θ values was observed. This may support the suggestion that the Fe2+ was incorporated in wollastonite open structure [29] and that wollastonite containing-iron could be formed. On increasing Fe2O3 content (1.73 mol%) in the glass composition (the sample G6) wollastonite molecules captured more Fe2+ in its structure and converted to ferrobustamite (Ca0.70Fe0.38)2SiO5 phase (Fig. 4, Pattern III, Table 2). This ferrobustamite phase has a pyroxenoid triclinic structure, very similar to that of β-wollastonite-(CaSiO3) [29]. A miscibility gap between wollastonite and ferrobustamite was discovered by Shimazaki and Yamanaka [30]. They suggested that ferrobustamite with iron content larger than that of (Ca5/6Fe1/8)2SiO5 will be stable at lower temperatures, probably 300–600 °C.

4.3. Crystallization – SEM study

The microstructure of glass-ceramics is very important and determines suitable mechanical properties [31]. Scanning electron microscope revealed that the grain microstructures were changed from volume crystallization of dendritic-like growths, in the sample G1, without In2O3 or Fe2O3, (Fig. 5a) to fine microstructure in G4, with In2O3, (Fig. 5b) or, ultra-fine microstructure in G6, with In2O3 and Fe2O3, (Fig. 5c). This is attributed to the fact that the co-crystallization of multi phases in the glass-ceramic led to form fine microstructure [4]. The variations of temperature during the heat treatment of the glass-ceramic formation also considered. The presence of iron oxide in the glasses increases the crystallization centers and stimulates the crystallization of the glass during the reheating process giving rise to volume crystallization of medium to fine grained microstructure [27].
4.4. Properties – Thermal expansion coefficient

The properties of the Zn-containing glasses are depending greatly on their composition which governs in turn the glass structure [32]. Properties of their crystalline phases are also affected by the structure, type and composition of the phases developed during crystallization process and by the originating of residual glassy matrix. The amount of the different crystalline phases is greatly important for optimizing the properties of glasses, which influences the thermo-mechanical properties of the glass-ceramics [11].

Glass-ceramics are remarkable due to very wide range of thermal expansion coefficients which can be obtained. At one extreme, there are materials having zero or even negative coefficients of thermal expansion, while, at the other extreme, materials having expansion coefficient close to those of useful metals are also available [33]. It is generally agreed that properties, such as the coefficient of thermal expansion, depend on the nature and volume of the precipitated phases, as well as the heat-treatment history [4]. Changes in the composition of the basic glass, say, substitution between different metal ions, always play a very important role in the amendment of the thermal expansion coefficient.

The thermal expansion coefficients of the investigated glass-ceramics after heat treatment are ranged from 191×10⁻⁷ 1/°C to 115×10⁻⁷ 1/°C in the temperature range 25–700 °C (Fig. 6). Lithium zinc silicate glasses generally crystallized to produce crystal phases such as lithium zinc orthosilicate (LZS) which have α-values (110–180)×10⁻⁷ 1/°C (25–1030 °C) [11]. While, the α-value of wollastonite is 94×10⁻⁷ 1/°C (100–200 °C) [34], lithium disilicate is 110×10⁻⁷ 1/°C (20–600 °C) [6] and α-quartz is 237×10⁻⁷ 1/°C (20–600 °C) [6]. According to the previous consideration, it follows therefore that, the decrease in the α-values of the studied glass-ceramics containing In₃O₅ in comparison to the crystalline sample G₁ (without In₃O₅) could be attributed to the formation of LiInSi₂O₆ phase having relatively low expanding coefficients [6,35]. The obtained data are in agreement with the previous results published by Salman and Salama [36], confirming that the crystallization of lithium indium silicate (LiInSi₂O₆) phase gives low thermal expansion coefficient of the glass-ceramic.

The addition of Fe₂O₃ in the glass composition (the samples G₅ and G₆) led to decrease in the thermal expansion coefficient values compared to the sample G₁ without Fe₂O₃. This may be due to the incorporation of iron oxide in the wollastonite structure and formation of solid solution in the form of ferrobustamite ((Ca₀.⁷₉Fe₀.₂₁)₂(SiO₄)) instead of wollastonite phase. However, there is no available data about the α-value of ferrobustamite phase. Glass compositions crystallizing in form of solid solutions should be of importance from the point of view of improvement the physical properties of the material [37]. Matsueda [38] reconfirmed that ferrobustamite is distinct from wollastonite in optical and chemical properties as well as cell dimensions, and considered paragenetic relations between ferrobustamite and wollastonite. The structure variation due to Ca-Fe substitution in the ferrobustamite solid solution is reflected in its cell parameter [39]. Since thermal expansion of cation polyhedra is in general geometrically similar to the expansion due to cation substitution, thermal expansion of the cation octahedra in ferrobustamite would be expected to impose a similar rotation effect upon silicate tetrahedral.

From the obtained data, it is clear that the difference in content and type of crystal phases formed in the evaluated crystalline solid samples may lead to differences in their thermal expansion coefficients (Fig. 7). Salman and Mostafa [40] pointed out that the presence of iron oxide increases the number of crystallization centres and stimulates the crystallization of the glass during the reheating process, giving to rise volume crystallization of medium to fine grained textures. Thus, the increase of crystal content is advantageous in reducing the linear thermal expansion coefficient and improving the thermal stability [41]. The degree of crystallization might also have a significant influence on the thermal expansion coefficient [42].

4.5. Properties – Microhardness

Hardness, defined as the material resistance to localized deformation, is an important mechanical property of materials. Hard materials have many industrial applications wherever resistance to abrasion and wear are important [43]. The hardness of glass-ceramic is related to both crystalline and residual glassy phases. The changes of the crystalline phases with temperature certainly affect the physical properties of the glass-ceramics [44].

Hardness of glass-ceramics is proportional to its wear resistance. The microhardness values of the glass-ceramic samples are presented in Fig. 7. The reported data (3.56–5.44 GPa) showed that, the modification of the glass composition by adding In₃O₅ and Fe₂O₃ enables formation of glass-ceramic materials with good mechanical properties. The reason might be the formation of finer grains microstructure than those formed in the base crystalline sample G₁ (without In₃O₅ and Fe₂O₃) as indicated from the SEM micrographs (Fig. 5).

The microhardness of glass-ceramics generally increased with the increase of the crystallization tendency, smaller crystalline grains as well as formation of fine microstructure [45]. The presence of iron ions in glass-ceramics (the samples G₅ and G₆), that served as nucleation accelerator, enhanced the crystallization process and this promoted internal melt precipitation of the crystal [46]. Under certain conditions, the formation of more crystal nuclei decreases the growth rate of single crystals, leading to finer crystal grains (Figs. 5b,c). Song et al. [47] reported that the amount of crystal particles increases when the size decreases which is followed with a reduction in the content of the glass phase. These all make the structure more compact, and can meet the requirements of wear resistance in the glass-ceramic.
V. Conclusions

The contributions of In$_2$O$_3$ and Fe$_2$O$_3$ to the crystallization of the glasses based on Li$_2$O-ZnO-CaO-SiO$_2$ system have been studied. The developed crystalline phases after controlling heat treatments were α-quartz (SiO$_2$), lithium zinc silicate (Li$_2$ZnSiO$_4$), wollastonite (CaSiO$_3$), lithium disilicate-(Li$_2$Si$_2$O$_5$), wollastonite containing-iron, ferrobustamite ((Ca$_{10.70}$Fe$_{0.21}$)SiO$_3$) and lithium indium silicate (LiInSi$_2$O$_6$) phases. The thermal expansion coefficient and microhardness of the corresponding glass-ceramics were also measured. The obtained results showed that the α-values of the crystalline products, in the temperature range 25–700 °C, decreased from 191×10$^{-7}$ 1/°C to 115×10$^{-7}$ 1/°C by addition of In$_2$O$_3$ and Fe$_2$O$_3$. While Vickers’ microhardness values of the glass-ceramics increased from 3.56 to 5.44 GPa with addition of In$_2$O$_3$ and Fe$_2$O$_3$. Thus, the incorporation of indium and iron oxides in varying concentrations improved the mechanical properties and the thermal stability in the resultant glass-ceramics.

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