

# Microstructure and Properties of $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{P}_2\text{O}_5$ Glass-Ceramics

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**Abstract:** Serials  $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2$  matrix glasses and glass-ceramics with different content of  $\text{P}_2\text{O}_5$  were prepared by conventional melt cooling method and crystallization process. The effects of  $\text{P}_2\text{O}_5$  content on microstructure and properties such as viscosity, melting temperature and coefficient of thermal expansion (CTE) of  $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{P}_2\text{O}_5$  glasses and glass-ceramics were investigated by DSC, XRD, CTE and SEM. The results show the melting temperature and viscosity of LASP matrix glass decreases with the content of  $\text{P}_2\text{O}_5$ , while the glass will phase-separated when the content of  $\text{P}_2\text{O}_5$  is more than 8.0 wt%. The main crystal phase in researched glass-ceramics is beta-spodumene when crystallization temperature is 850°C. The content of  $\text{P}_2\text{O}_5$  has no obvious influence on the type of crystal phase in researched glass-ceramics.  $\text{P}_2\text{O}_5$  can promote the precipitation and growth of crystals in glass-ceramics, and slightly increase the CTE of matrix glass and glass-ceramics. Virgilite with negative CTE formed first in LASP glass-ceramics at low temperature, and then turned into beta-spodumene with increase of treating temperature.

**Keywords:** Glass-ceramics, microstructure, crystallization behavior,  $\text{P}_2\text{O}_5$ , coefficient of thermal expansion.

## 1. INTRODUCTION

$\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2$  (LAS) glass-ceramics have attracted considerable attention as a structural and functional material due to their excellent resistance to thermal shocks [1, 3]. The unique performance to enable widely used in cookware, heat exchangers of high temperature, and precision optical applications such as laser gyroscope, telescope mirror supports and projector lampshade [4,5]. Despite a great deal of effort has been invested to improve the performance of LAS glass-ceramics, and much researches on the relationships among chemical composition, preparation process and performance have been carried out, the microstructure and crystallization behavior of LAS glass-ceramics with different types of additives need study further. It is known to all that it is difficulty to prepare LAS matrix glass because of the high melting temperatures and high viscosity. In order to decrease its melting temperature and viscosity, different types of additives such as  $\text{Y}_2\text{O}_3$ ,  $\text{B}_2\text{O}_3$ , BaO, SrO, alkali oxides and alkali earth oxides have been doped in different research, but it would cause some negative impacts on the excellent properties of LAS glass-ceramics [6, 8].

$\text{P}_2\text{O}_5$  is usually used as nucleating agent in glass-ceramic systems [9, 10]. A lot of experiments have been done to research its influences on crystallization behavior and microstructure of glass-ceramics but few on viscosity or melting temperature of glass. In present study,  $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{P}_2\text{O}_5$  (LASP) matrix glass with different content of  $\text{P}_2\text{O}_5$  as fluxing agent were prepared by melt cooling method,

and the effects of  $\text{P}_2\text{O}_5$  content on the viscosity and thermal expansion coefficient, the crystallization temperature on microstructure, crystallization behavior and properties were discussed.

## 2. MATERIALS AND EXPERIMENTAL PROCEDURE

### 2.1. Preparation of the Matrix Glass of LASP

The chemical compositions (wt %) of the matrix glass were given in Table 1.  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{ZrO}_2$ , MgO, ZnO,  $\text{Li}_2\text{CO}_3$  and  $(\text{NH}_4)_2\text{HPO}_4$  were used as raw materials to prepare matrix glass, in which  $\text{Li}_2\text{CO}_3$  as the source of  $\text{Li}_2\text{O}$  and  $(\text{NH}_4)_2\text{HPO}_4$  as the source of  $\text{P}_2\text{O}_5$ , all raw materials are analytical grade. The weighted errors of all raw materials were controlled in the range of  $\pm 0.01\text{g}$ . Glass batch was thoroughly mixed in a planetary ball mill for 5 h and molten in alumina crucible at 1630 °C for 2 ~ 3 h. The bulk glass was prepared by pouring the homogeneous melt into a preheated steel mold, immediately transferred into a muffle furnace preheated to 650 °C and annealed at 650 °C for 2 h.

### 2.2. Measurement of Viscosity for the Matrix Glass

Parts of the matrix glass samples were cut into pieces with 30–40 g for each one by a diamond cutting machine (Model: SYJH-150). The samples were placed into the high temperature rotational viscosimeter (Model: Rheotronic II) to measure viscosity of glass above the softening point. The viscosity of glasses can be fitted to the Flusher equation [11]:

$$\text{Log}_{10}\eta = A + [B/(T - T_0)]$$

where  $\eta$  is the viscosity (Pa·s), T is the temperature (°C) and A, B and  $T_0$  are the adjustable constants.

### 2.3. Measurement of DSC Curve

In order to investigate the influence of  $\text{P}_2\text{O}_5$  content on glass transition temperature ( $T_g$ ), starting crystallization

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**Table 1. Chemical Compositions of the Matrix Glass (wt %)**

Specimens No.	SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub> +ZrO <sub>2</sub>	Li <sub>2</sub> O	MgO+ZnO	P <sub>2</sub> O <sub>5</sub>	Σ
P0	88.74	4.74	4.35	2.17	0.00	100.00
P2	86.96	4.64	4.26	2.13	2.00	100.00
P4	85.19	4.54	4.17	2.09	4.00	100.00
P6	83.41	4.45	4.09	2.04	6.00	100.00
P8	81.64	4.36	4.00	2.00	8.00	100.00
P10	79.87	4.27	3.91	1.96	10.00	100.00

temperature ( $T_c$ ) and crystallization peak temperature ( $T_p$ ), a differential scanning calorimeter (DSC, Netzsch 404PC) was used to examine the thermal effect with  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> as a reference material and draw the DSC curve in the temperature range from 25 °C to 900 °C at a heating rate of 10 °C/min.

#### 2.4. Preparation and Characterization of Glass-Ceramics

According to the DSC curve, nucleation and crystallization temperature were determined and the glass was converted to glass-ceramics. In present work, the specimen P8 is selected as the research specimen; the nucleation temperature is set as 750 °C for 1h and crystallization temperature set as 800 °C, 850 °C, 900 °C, 950 °C, and 1000 °C, respectively. The heat treatment conditions for the glass-ceramics are list in Table 2. The crystal phases in the glass-ceramics were determined by X-ray powder diffraction analysis system (Model: D/Max-2500).

#### 2.5. Thermal Expansion Coefficient of the Matrix Glass and Glass-Ceramic

The matrix glass and glass-ceramic samples were prepared into the dimensions 5 mm×5 mm×20 mm with accuracy ±0.02 mm by cutting, grinding and polishing to measure their length change  $\Delta L$  with temperature, draw thermal expansion curves, and calculate mean CTE from 25 °C to 700 °C at a heating rate of 10 °C/min.

#### 2.6. Observation of the Microstructure in the Glass-Ceramics

The prepared LASP glass-ceramic specimens were polished, etched with 5% HF solution for 90 s, cleaned by ultrasonic, coated with gold and examined by a Hitachi X-650 scanning electron microscopy (SEM).

### 3. RESULT AND DISCUSSION

#### 3.1. The Appearance of Prepared LASP Matrix Glasses

There are few bubbles in specimen P0 but no obvious bubbles in matrix glass P2 ~ P10, this indicates appropriate content of P<sub>2</sub>O<sub>5</sub> can decrease viscosity of LAS glass. A remarkable difference for all the specimens is that glass P0 ~ P8 are transparent and colorless while glass P10 is inhomogeneous. Parts of glass P10 are white and others are transparent, which suggests the glass P10 is phase separated badly and the suitable P<sub>2</sub>O<sub>5</sub> content in LAS system is not more than 8.0 wt%. So, glass P10 is not researched further in this study.

#### 3.2. The Viscosity of Prepared LASP Matrix Glasses

The relationship between viscosity and temperature of the glass specimens is shown in Fig. (1). The curves in Fig. (1) show the viscosity decreased with the increase of P<sub>2</sub>O<sub>5</sub> content and temperature in region of 1200 °C – 1650 °C. It suggests that appropriate content of P<sub>2</sub>O<sub>5</sub> can reduce the melting temperature and decrease high-temperature viscosity of the LAS glass. Murat B. also found that the viscosity of SrO–TiO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub>–B<sub>2</sub>O<sub>3</sub> glass decreased consistently as the P<sub>2</sub>O<sub>5</sub> content increased [12]. The point temperature at the viscosity of 10 Pa·s is referred to the melting temperature of glass [6], so the melting temperature of P0, P2, P4, P6 and P8 are 1635 °C, 1628 °C, 1613 °C, 1592 °C and 1568 °C, respectively. It can be seen that, when the content of P<sub>2</sub>O<sub>5</sub> increased to 8.0 wt%, the melting temperature of LASP glass decreased 67 °C (Fig. 2). The main reason is P<sub>2</sub>O<sub>5</sub> possess much lower melting point than those of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>, and introducing P<sub>2</sub>O<sub>5</sub> lowered the eutectic point of the LAS system.

**Table 2. Heat Treatment Condition for Glass-Ceramic Specimens**

Specimen No.	Nucleation		Crystallization	
	Temperature/°C	Hold Time/h	Temperature/°C	Hold Time/h
T0	750	1.0		
T1	750	1.0	800	1.0
T2	750	1.0	850	1.0
T3	750	1.0	900	1.0
T4	750	1.0	950	1.0
T5	750	1.0	1000	1.0

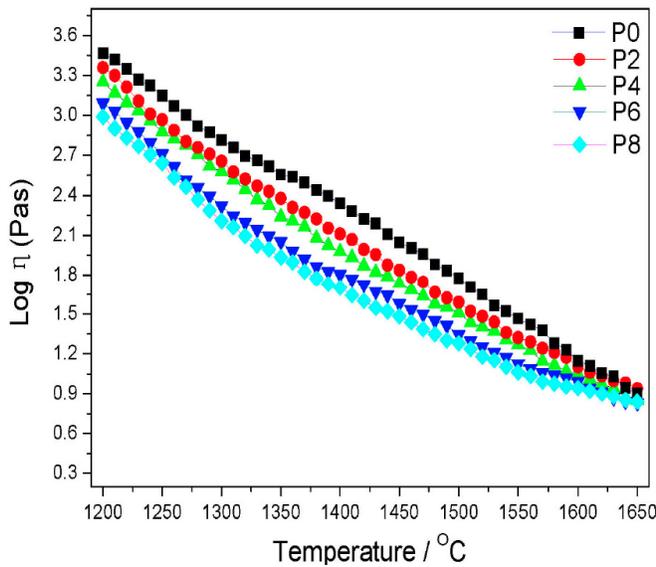


Fig. (1). Relationship between temperature and viscosity of glass with different content of  $\text{P}_2\text{O}_5$ .

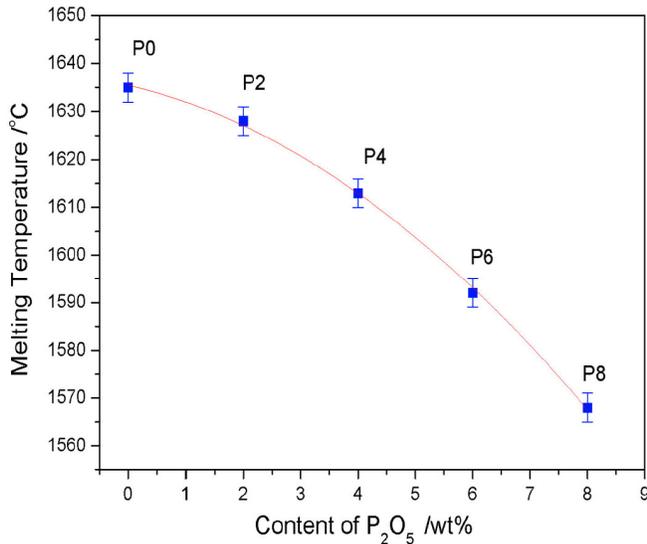


Fig. (2). Melting temperature of LASP glass with different content of  $\text{P}_2\text{O}_5$ .

### 3.3. DSC Analysis of LASP Matrix Glasses

Fig. (3) shows the DSC curve of the  $\text{P}_2\text{O}_5$  doped LAS glass. The  $T_g$  ranges from  $640^\circ\text{C}$  to  $680^\circ\text{C}$ , and two  $T_g$  appear when the content of  $\text{P}_2\text{O}_5$  is more than 4 wt%, indicating both  $\text{SiO}_2$  and  $\text{P}_2\text{O}_5$  act as glass network former in the  $\text{P}_2\text{O}_5$  doped LAS system. The homogeneity of the glass microstructure is influenced by the difference of the glass formation ability (GFA) of the glass network former. Once more than one glass network former exist in the glass, the glass became unstable and phase separated may happen. That is why glass P10 is inhomogeneous when the content of  $\text{P}_2\text{O}_5$  is more than 8%.

The  $T_c$  of the prepared LASP matrix glass ranges from  $770^\circ\text{C}$  to  $820^\circ\text{C}$ , and lowers by increase of  $\text{P}_2\text{O}_5$  content. It suggests the value of activation energy for crystallization decreased by introduction of  $\text{P}_2\text{O}_5$  in LAS glass. The  $T_p$  of the prepared glass is not changed obviously, which suggests the main crystal phase is not varied with increase of  $\text{P}_2\text{O}_5$ .

The endothermic peak near  $T_g$  corresponds to the thermal effect coming from molecular rearrangement in glass structure, which means the crystal nuclei can be formed if the glass is treated at  $640^\circ\text{C}$  to  $680^\circ\text{C}$ . In present work,  $680^\circ\text{C}$  is chosen as the nucleation temperature and  $850^\circ\text{C}$  is chosen as the crystallization temperature for all the prepared LAS glass.

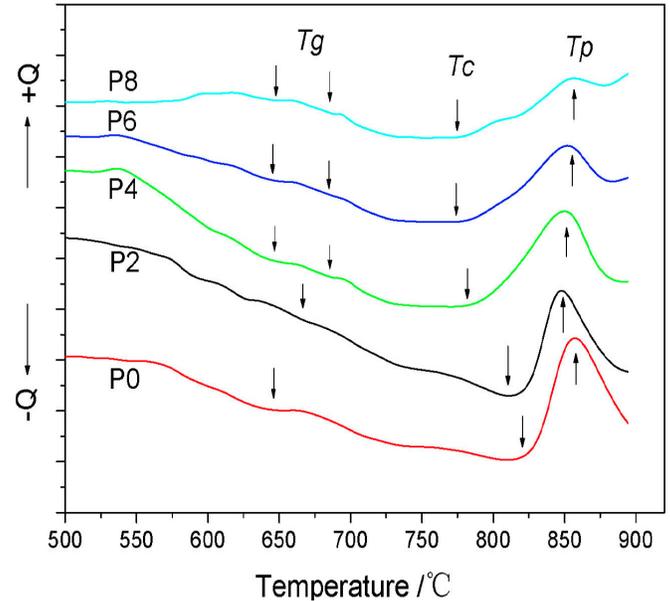


Fig. (3). DSC curves of LASP glass specimens containing different content of  $\text{P}_2\text{O}_5$ .

### 3.4. CTE of Matrix Glasses and Glass-Ceramics

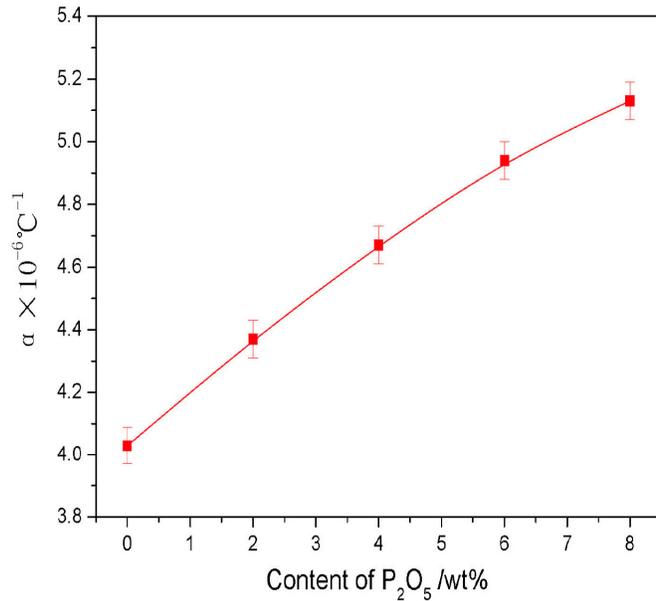
Figs. (4, 5) show the thermal expansion behaviors of the matrix glasses and the glass-ceramics crystallized at  $850^\circ\text{C}$ , respectively. From Fig. (4), the matrix glasses have large CTE from room temperature to  $700^\circ\text{C}$ , and the CTE increases with temperature and the content of  $\text{P}_2\text{O}_5$  in LAS glass. CTE of phosphate glasses is much larger than that of silicate glass, the latter is about  $5 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  while the former even larger than  $13.5 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  [13]. As a glass network former [14, 15], once  $\text{P}_2\text{O}_5$  is introduced into the silicate glass, the CTE of silicate glass will be increased inevitably.

The CTE of glass-ceramic specimen treated at  $850^\circ\text{C}$  ranges from  $-0.2 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  to  $1.2 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  at  $700^\circ\text{C}$ , which is far smaller than that of corresponding matrix glass. The CTE of glass-ceramic mainly depends on the crystal phase kinds and volume content in the matrix glass. The LASP glass-ceramic with low even negative CTE means the CTE of the separated crystallites must be low. A remarkable phenomenon is the CTE of LASP glass-ceramic increases slightly but that of the matrix glass increases obviously with the content of  $\text{P}_2\text{O}_5$ . This suggests that introduction of  $\text{P}_2\text{O}_5$  doesn't change the main crystal phase in the glass-ceramics, and the tiny increase of CTE results from the residual glass phase in glass-ceramics.

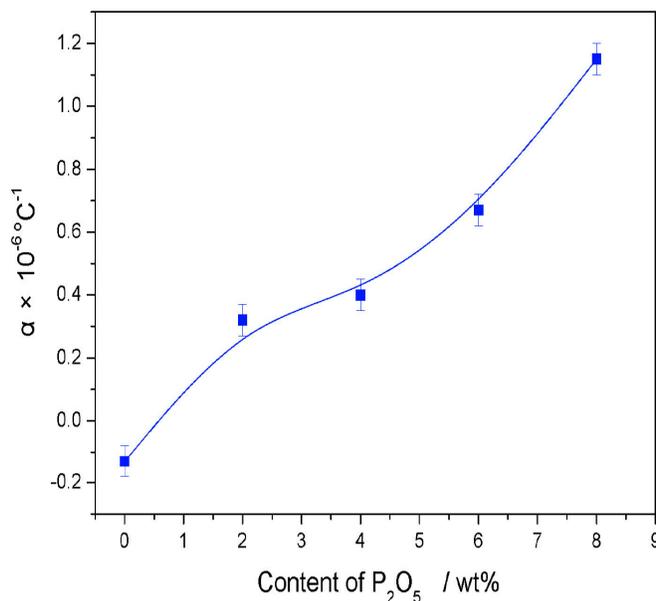
### 3.5. Effect of $\text{P}_2\text{O}_5$ Content on Crystallization Phase of Glass-Ceramics

Fig. (6) shows the XRD powder patterns of all the LASP glass-ceramics treated at  $850^\circ\text{C}$  for 1h. Only one main crystal phase, namely  $\beta$ -spodumene ( $\text{LiAlSi}_2\text{O}_6$ , JCPDF card

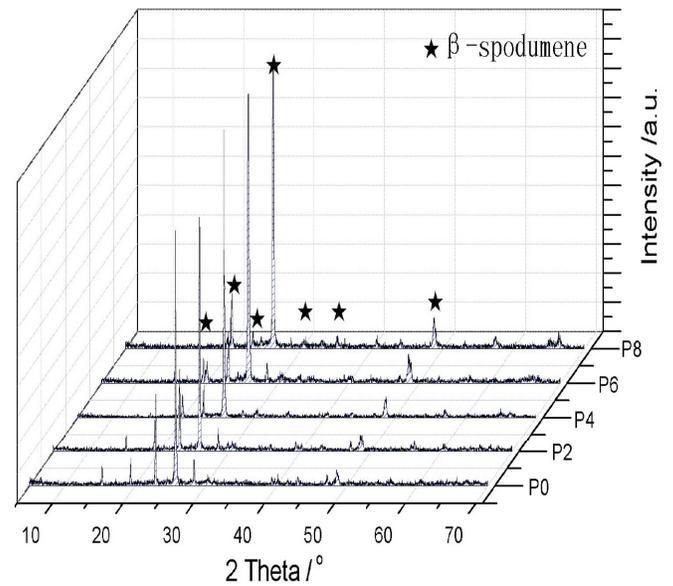
No. 35-0797), can be observed in all the glass-ceramic specimens. The remaining peaks in curves can't be recognized because it is too weak to identify. This accord with the DSC curves of the matrix glasses, in which only one obvious exothermic peak appears. The formation of  $\beta$ -spodumene crystalline results in the low even negative CTE of the LASP glass-ceramics. The same crystal phase existing in all the glass-ceramic suggests that introduce or increase the content of  $P_2O_5$  has no obvious impacts on the type of crystallization phase of LAS glass-ceramic. Further research can be seen that the intensity of the main peak at  $25^\circ$  increases with the content of  $P_2O_5$ . This means that  $P_2O_5$  can promote the precipitation and growth of crystals in glass-ceramics. The possible reason is  $P_2O_5$  act as the role of nucleating agent in glass-ceramics.



**Fig. (4).** CTE curves of matrix glass specimens containing different content of  $P_2O_5$ .



**Fig. (5).** Relationship between content of  $P_2O_5$  and CTE of glass-ceramic crystallized at  $850^\circ\text{C}$  for 1 h.

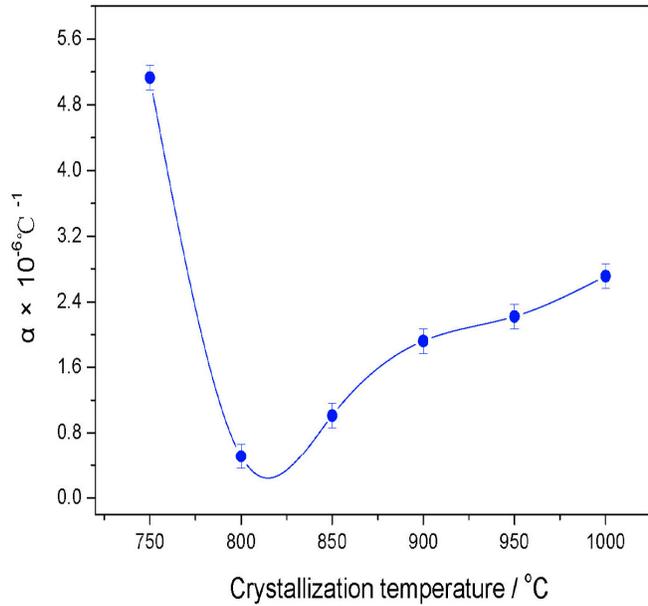


**Fig. (6).** XRD patterns of glass-ceramic specimens crystallized at  $850^\circ\text{C}$  for 1 h.

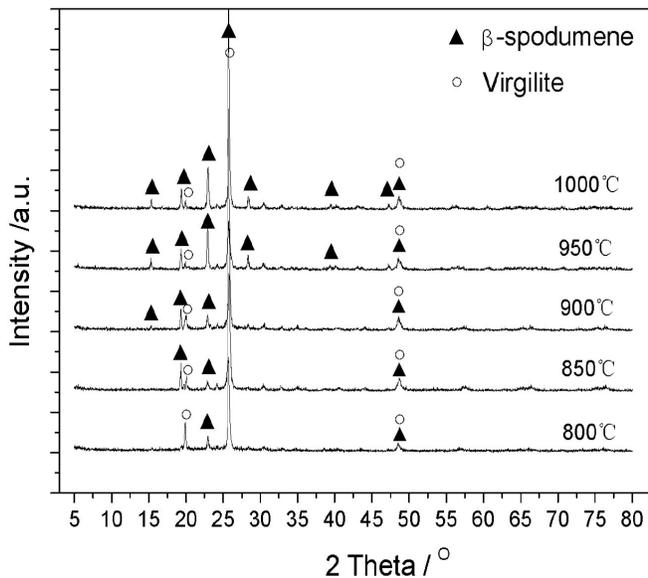
### 3.6. Effect of Crystallization Temperature on CTE and Crystallization Behavior of Glass-Ceramics

The glass-ceramics treated at different temperature often have various CTE. As a composite material, glass-ceramics compounded by the glass phase and crystal phase, and the CTE of glass-ceramics depends on the type and volume content of crystals in matrix glass. Fig. (7) is the CTE of the glass-ceramic specimen P8 treated at different temperature. The CTE is about  $5.1 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$  when specimen P8 treated at  $750^\circ\text{C}$  for 1 h, it is almost the same to the CTE of the glass specimen P8. This means the specimen P8 treated at  $750^\circ\text{C}$  have the similar microstructure to the matrix glass, containing almost no crystal. Increase treating temperature to  $800^\circ\text{C}$ , the CTE dropped sharply to  $0.7 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ . With the further increase of crystallization temperature from  $800^\circ\text{C}$  to  $1000^\circ\text{C}$ , the CTE of specimen P8 increase slowly from  $0.7 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$  to  $2.8 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ . It can be seen from the figure that a minimum of CTE can be obtained when appropriate crystallization temperature is selected.

Fig. (8) is the XRD patterns of specimens P8 treated at different temperature. Two types of crystal phases, virgilite and beta-spodumene, can be observed in all the specimens T1 to T5. In the section of 3.5, only one crystal phase (beta-spodumene) has been found in specimen T2. By comparing to specimen T1, another crystal phase (virgilite) can be observed. It can be seen from the figure the main crystal phase is virgilite when crystallization temperature is lower than  $850^\circ\text{C}$ , and turn into beta-spodumene when increase treating temperature. Virgilite has a disordered  $\beta$ -quartz structure, and has similar thermal expansion character to  $\beta$ -quartz. Literature suggests that virgilite posses negative coefficient of thermal expansion, and a small quantity of virgilite can decrease the CTE of glass-ceramics dramatically [16]. This is accord with the present work. When specimen T1 is treated at  $800^\circ\text{C}$ , the CTE is the smallest one because the main crystal phase is virgilite. Increase temperature to  $850^\circ\text{C}$  and higher, much beta-spodumene crystal phase appears and virgilite disappears gradually, so the CTE of specimen increases with temperature.



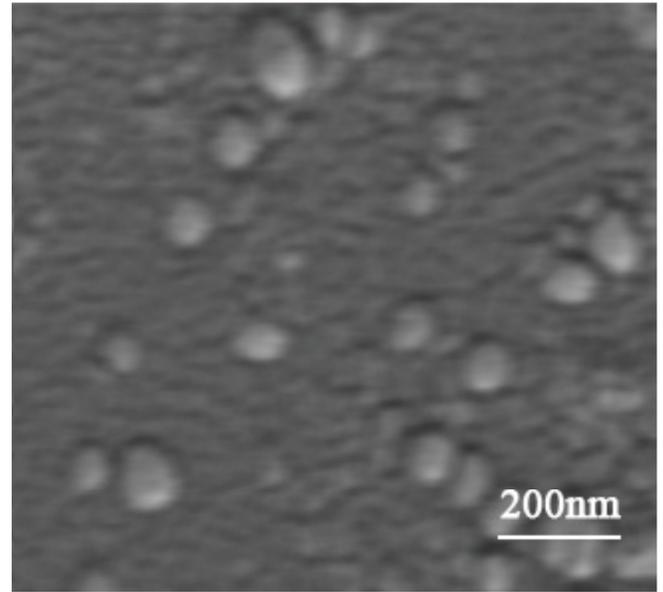
**Fig. (7).** Relationship between crystallization temperature and CTE of glass-ceramic specimen P8.



**Fig. (8).** XRD patterns of glass-ceramic specimens P8 treated at different temperature.

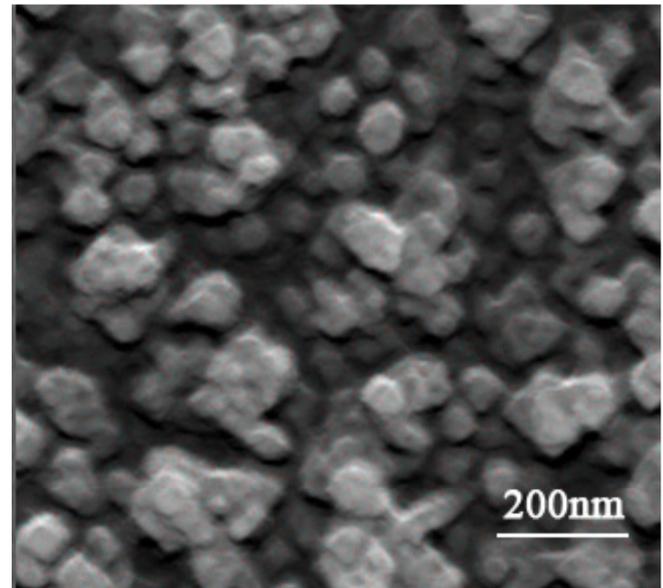
### 3.7. Effect of Crystallization Temperature on Microstructure of Glass-Ceramics

Fig. (9) shows the SEM photograph of glass-ceramics specimen T1, T2 and T4. A small amount of spherical crystals with size of 60nm can be observed in Fig. (9a). According to the analysis above, the spherical crystals are virgilite. Increase crystallization temperature to 850°C, the spherical virgilite crystals is hardly to find, and much beta-spodumene crystals with irregular shape appears. When crystallization temperature increases to 950°C, the spherical virgilite crystals can't be found in glass-ceramics anymore, and most of the area occupied by beta-spodumene crystals. It can be seen from the photographs that the size of crystals is about 60nm for all the specimens, while the type and volume content of crystals change with crystallization temperatures.



(a) Treated at 800 for 1h

**Fig. (9a).** SEM of glass-ceramic specimens containing  $\text{P}_2\text{O}_5$  8.0wt% treated at different temperatures.



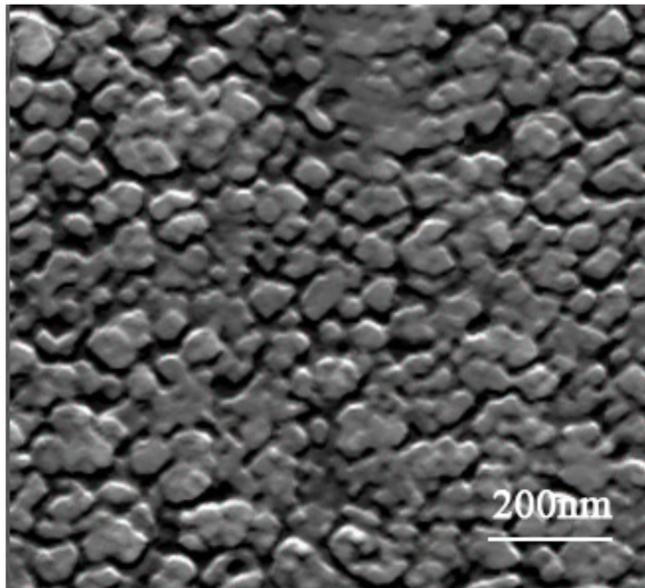
(b) Treated at 850 for 1h

**Fig. (9b).** SEM of glass-ceramic specimens containing  $\text{P}_2\text{O}_5$  8.0wt% treated at different temperatures.

## 4. CONCLUSION

1. The melting temperature and viscosity of LASP matrix glass decreases with the content of  $\text{P}_2\text{O}_5$ , while the glass will phase-separated when the content of  $\text{P}_2\text{O}_5$  is more than 8.0 wt%.
2. The main crystal phase in LASP glass-ceramics is beta-spodumene when crystallization temperature is 850°C. The content of  $\text{P}_2\text{O}_5$  in LASP glass system has no obvious influence on the type of crystal phase in glass-ceramics.

3.  $P_2O_5$  can promote the precipitation and growth of crystals in glass-ceramics, and slightly increase the CTE of matrix glass and glass-ceramics of LASP.
4. Virgilite with negative CTE formed first in LASP glass-ceramics at low temperature, and then turned into beta-spodumene with increase of treating temperature.



(c) Treated at 950 °C for 1h

**Fig. (9c).** SEM of glass-ceramic specimens containing  $P_2O_5$  8.0wt% treated at different temperatures.

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