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Preparation and Fabrication of Lithium Disilicate Glass Ceramic as Dental Crowns via Hot Pressing Method

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***Abstract*—**Two Lithium Disilicate (LD) glass ceramics based on SiO_2-Li_2O-K_2O-Al_2O_3 system were prepared through a glass melting method. The glass rods were then fabricated into dental crowns via a hot pressing at 900˚C and 850˚C in order to study the effect of the pressing temperatures on the phase formation and microstructure of the glasses. Different samples of as cast glass and heat treated samples (600˚C and 700˚C) were used to press for investigating the effect of an initial microstructure on the hot pressing technique. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were performed to determine the phase formation and microstructure of the samples, respectively. XRD results show that the main crystalline structure was $Li_2Si_2O_5$ by having Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, Li_2SiO_3, Ca_5 $(PO_4)_3F$ and SiO_2 as minor phases. Glass compositions with different heat treatment temperatures exhibited a difference phase formations but have less effect during pressing. SEM micrographs showed the microstructure of $Li_2Si_2O_5$ as lath-like shape in all glasses. With increasing the initial heat treatment temperature, the longer the lath-like crystals of lithium disilicate were increased especially when using glass heat treatment at 700˚C followed by pressing at 900˚C. This could be suggested that LD1 heat treatment at 700˚C which pressing at 900˚C presented the best formation by the hot pressing and compiled microstructure.

***Keywords*—**Lithium disilicate, Hot pressing, Dental crown, Microstructure.

I. Introduction

CURRENTLY, all-ceramic systems such as lithium disilicate glass ceramic (LD, $Li_2Si_2O_5$) are used to fabricate single and multiunit dental restorations usually for dental crowns, bridges and veneers because of its color close to natural teeth, esthetic results and its high mechanical properties [1], [2]. Particularly, lithium disilicate glass ceramics based on SiO_2-Li_2O-K_2O-Al_2O_3 system have been applied in dental crowns because they obtain a highly thermal shock resistant which is the cause of a low thermal expansion during processing. This kind of glass ceramic could be fabricated via either a lost wax casting or a CAD/CAM technique. The CAD/CAM technology is quite practical to produce customize dental for patients. In addition, CAD/CAM restorations are better-fitting, more durable and more natural looking (multi-colored and translucent, similar to natural teeth) than other machined restorations. However, using CAD/CAM restorations requires skill, experience of the dentists or laboratory technicians, and complexity of cases and treatment [3]. Another technology demonstrated to produce dental restorations effectively used now a day is a pressed technology due to a wide variety of pressable ceramics available. This technique applies the lost wax method under the pressure. A ceramic ingot is heated and then forced under pressure into a wax formed void.

Previously, we had proposed four different glass compositions based on SiO_2-Li_2O-K_2O-Al_2O_3 system which exhibited a high mechanical strength [4], [5]. It was successfully fabricated dental crowns by using the CAD/CAM technique on those glass compositions in our laboratory. In this work, we introduced a hot pressing technology, a fast technique and suitable for LD glass ceramics to produce dental crowns on our LD glass ceramic. Two different glass compositions were selected to investigate the effect of glass compositions on phase formation and microstructure of the dental crowns after hot pressing at different pressing temperatures. An initial microstructure according to the difference heat treatment temperatures was also studied theirs effect on the phase formation and microstructure of the final products.

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II. Procedure

A. Glass Preparation

Lithium disilicate glasses as shown in Table I were produced using a conventional glass melting process. Glass batch was prepared by melting in a platinum crucible at 1500˚C for 2 hrs. The melted mixtures were quenched in cold water to make frit. The glass frit was milled before being re-melted at the same condition and then cast into a warm graphite mold (14 mm in diameter x 100 mm long). Graphite mold was annealed in a furnace at 400˚C for 2 hrs to reduce the internal stress in the glass.

TABLE I
Glass Compositions

Glass	Chemical compositions (mol %)						
	SiO_2	K_2O	LiO_2	Al_2O_3	P_2O_5	CaF_2	MgO
LD1	62.0	1.0	28.0	2.0	2.0	3.0	2.0
LD2	59.5	1.0	31.5	2.0	2.0	3.0	1.0

B. Heat Treatment

Samples were cut into the glass ingot size 12 mm in diameter x 20 mm in height and then heat treated in a furnace (Linn High Therm, Germany). The glasses were heated to a nucleating temperature of 600˚C and 700˚C with a heating rate of 5˚C/min, held for 2 hrs and then cooled to room

temperature at 5˚C/min. Table II refers to as-cast glass and glass ceramics.

TABLE II
HEAT TREATMENT PROFILES OF THE LITHIUM DISILICATE GLASSES

	Samples	Heat treatment profiles
Set 1	LD1_Non	as cast glass
	LD1_600	600˚C, 5˚C / min.
	LD1_700	700˚C, 5˚C / min.
Set 2	LD2_Non	as cast glass
	LD2_600	600˚C, 5˚C / min.
	LD2_700	700˚C, 5˚C / min.

C. Machining System Using Hot Pressing Technology

All samples were fabricated into the dental crowns using the hot pressing oven Programat EP 3000G2 (Invacare Vivadent Technical). Two pressing profiles were performed on the samples at 900˚C (according to the instruction for use of the machine for High translucent (HT) glass ceramic) and 850˚C according to the thermal properties of the glasses [1] as presented in Table III.

TABLE III
PRESSING PROFILES OF LD SAMPLES

Processing Temp.	Value range				
	B	t	T	H	E
900 ˚C	700	60	900	25	250
850 ˚C	700	60	850	25	250

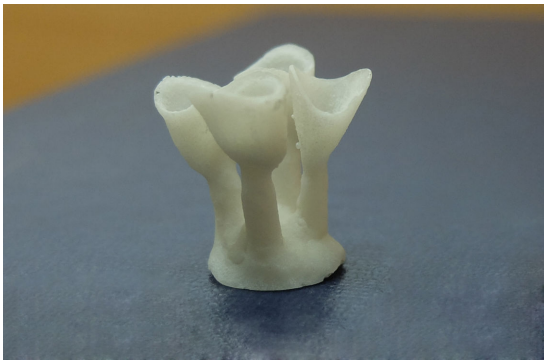
B: stand-by temp., t: temp. Increase ˚c /min, t: holding temp., H: holding time, e: abort speed

D. Sample Characterization

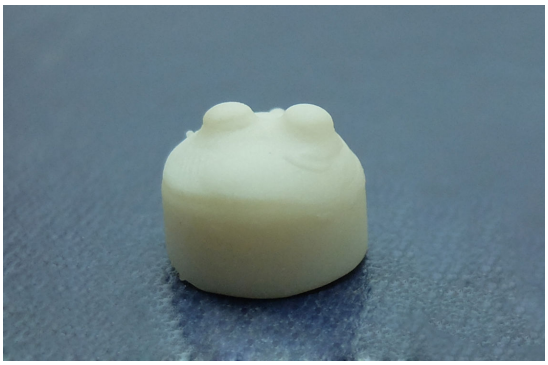
Phase formations of all samples was analyses by X-ray diffraction (Rigaku TTRAX III) operating from 20 to 60˚, 2θ at a scan speed of 2° 2θ/min and a step size of 0.02° 2θ with CuK_α radiation (K_α = 1.5406 nm) at 300 mA and 50 kV. The diffraction patterns with the ICDD (JCPDS) standard were used to identify crystal structure of samples. Microstructure of LD samples was observed by scanning electron microscope (SEM: Hitachi S-3400N) operating with 20kV at 5000X magnification.

III. RESULTS AND DISCUSSION

The appearances of samples after pressing at 900˚C and 850˚C are shown in Fig. 1. At 900˚C, the glasses can be formed the dental crowns (Fig. 1 (a)). In contrast, the glass sample cannot be fabricated into the dental crown specimens as required by pressing at 850˚C (Fig. 1 (b)). This indicates that the pressing profile at 850˚C does not reach the glass softening points. Therefore, the glasses could not flow into cavity of the mold which resulting in incomplete forming.



(a)



(b)

Fig. 1 Glass samples after pressing at (a) 900˚C and (b) 850˚C

The XRD results of LD1 and LD2 after pressing at 900°C by using as-cast glass and glass ceramic samples are shown in Fig. 2 and phase formations for all samples are summarized in Table IV. LD1_Non showed the crystal structures of $Li_2Si_2O_5$, Li_3O_4, and SiO_2. In LD1_600 and LD1_700, the crystals of $Li_2Si_2O_5$, Li_3O_4, $Li_{0.6}Al_{0.6}Si_2O_6$, SiO_2, and F_2 were indexed. With increasing heat treatment temperatures, the peaks of Li_3O_4, $Li_{0.6}Al_{0.6}Si_2O_6$, and F_2 clearer observed.

For LD2 pressing at 900˚C, LD2_Non consisted of $Li_2Si_2O_5$, $Li_{0.6}Al_{0.6}Si_2O_6$, and $Ca_5(PO_4)_3F$ crystals. In LD2_600 and LD2_700, $Li_2Si_2O_5$, Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, $Ca_5(PO_4)_3F$, Li_2SiO_3 can be indexed. The intensity of SiO_2 crystals seemed disappeared and replaced by the formation of Li_2SiO_3 phase instead after increasing heat treatment temperature.

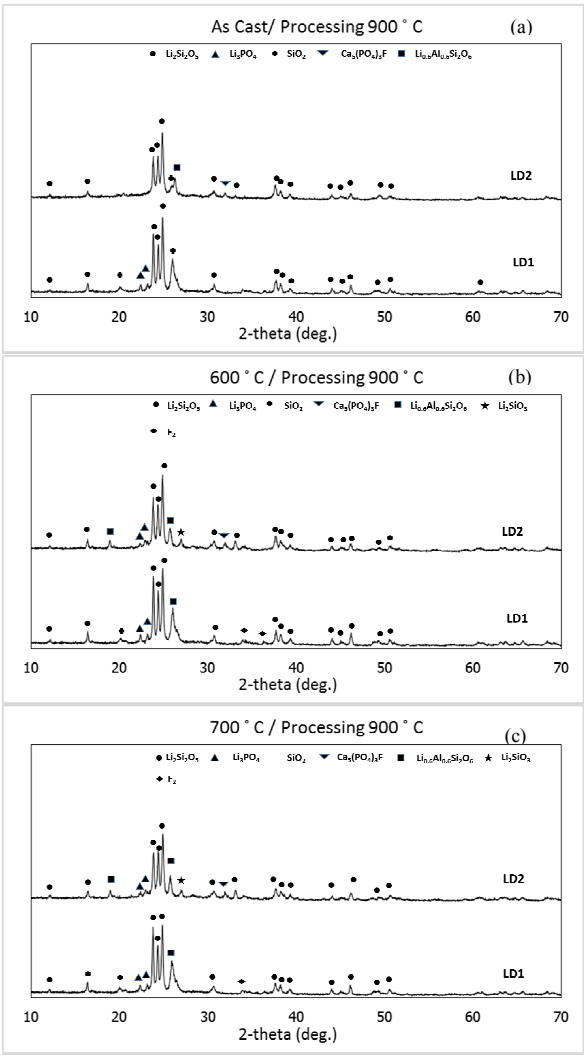


Fig. 2 XRD patterns of (a) as cast, (b) 600˚C, and (c) 700˚C for pressing temperature at 900˚C of LD1 and LD2

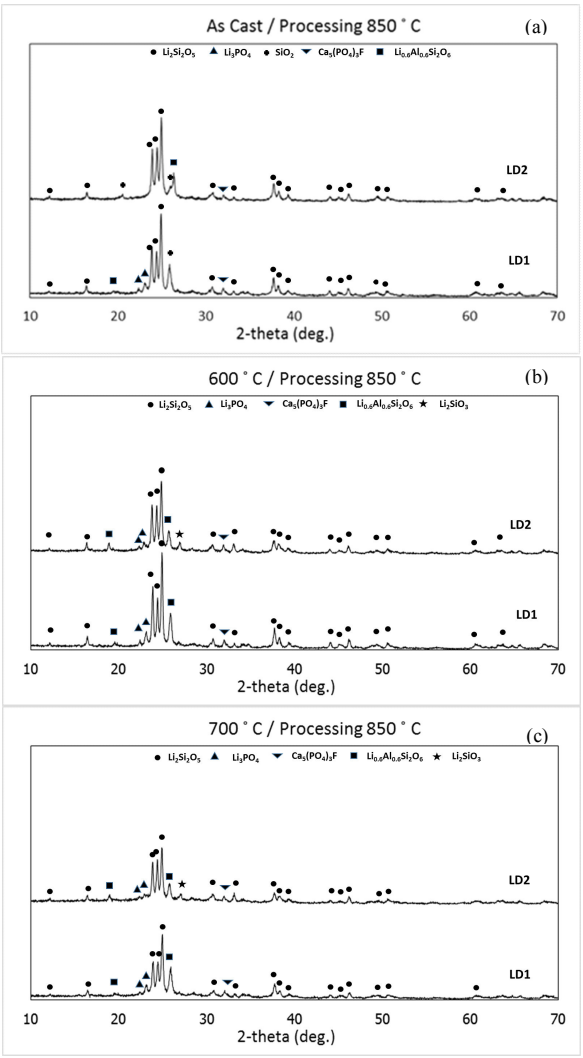


Fig. 3 XRD patterns of (a) as cast, (b) 600˚C, and (c) 700˚C for pressing temperature at 850˚C for LD1 and LD2

Fig. 3 shows XRD patterns of LD1 and LD2 pressed at 850˚C. The crystal structures found in LD2_Non were $Li_2Si_2O_5$, $Li_{0.6}Al_{0.6}Si_2O_6$, SiO_2, and $Ca_5(PO_4)_3F$. For LD2_600 and LD2_700, the phase formations of Li_3PO_4 and Li_2SiO_3 were observed, with no crystals of SiO_2. At this pressing temperature, the phase formation of LD1 was similar to LD2, with no crystal formation of Li_2SiO_3.

In comparison between different glass compositions, we can see the formation of Li_2SiO_3 in LD2_600 and LD2_700 samples, when pressing at both 850˚C and 900˚C. And the formation of SiO_2 only observed in LD1 pressing at 900°C. This could be explained by the difference of Si:Li ratio in glass compositions which could leaded to the change of the microstructure of $Li_2Si_2O_5$ crystals [1]. In addition, LD1 contained a high percentage of MgO resulting in less viscosity compared to LD2 [1]. This could describe the phenomenon of the easy pressing LD1 over LD2.

TABLE IV
SUMMARY OF CRYSTALLINE PHASE

Glass	HT Temp.	Pressing Temp.(˚C)	Crystalline phases
LD1	Non	900	$Li_2Si_2O_5$, Li_3PO_4, SiO_2
	600		$Li_2Si_2O_5$, Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, SiO_2, F_2
	700		$Li_2Si_2O_5$, Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, SiO_2, F_2
	Non	850	$Li_2Si_2O_5$, Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, $Ca_5(PO_4)_3F$
	600		$Li_2Si_2O_5$, Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, $Ca_5(PO_4)_3F$
	700		$Li_2Si_2O_5$, Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, $Ca_5(PO_4)_3F$
LD2	Non	900	$Li_2Si_2O_5$, $Li_{0.6}Al_{0.6}Si_2O_6$, $Ca_5(PO_4)_3F$
	600		$Li_2Si_2O_5$, Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, $Ca_5(PO_4)_3F$, Li_2SiO_3
	700		$Li_2Si_2O_5$, Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, $Ca_5(PO_4)_3F$, Li_2SiO_3
	Non	850	$Li_2Si_2O_5$, $Li_{0.6}Al_{0.6}Si_2O_6$, SiO_2, $Ca_5(PO_4)_3F$
	600		$Li_2Si_2O_5$, Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, $Ca_5(PO_4)_3F$, Li_2SiO_3
	700		$Li_2Si_2O_5$, Li_3PO_4, $Li_{0.6}Al_{0.6}Si_2O_6$, $Ca_5(PO_4)_3F$, Li_2SiO_3

SEM images, Fig. 4 reveals the non-uniform size and randomly lath-like crystals of $Li_2Si_2O_5$ oriented in a residual glassy matrix for all samples. This was in consistent with XRD patterns. The higher the heat-treated temperatures, the longer the lath-like crystals appeared in the samples. The finer grain of lath-like crystals was also found in LD2 rather than in LD1.

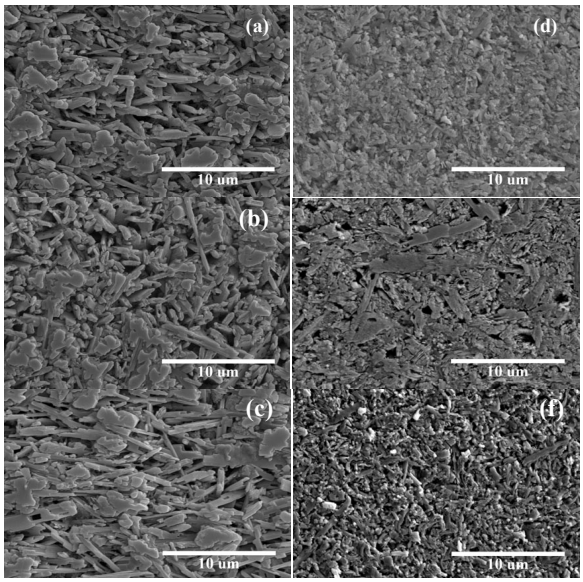


Fig. 4 SEM images of (a) LD1 as cast, (b) LD1 600˚C, (c) LD1 700˚C, (d) LD2 as cast, (e) LD2 600 ˚C, and (f) LD2 700 ˚C for 900˚C pressing temperature

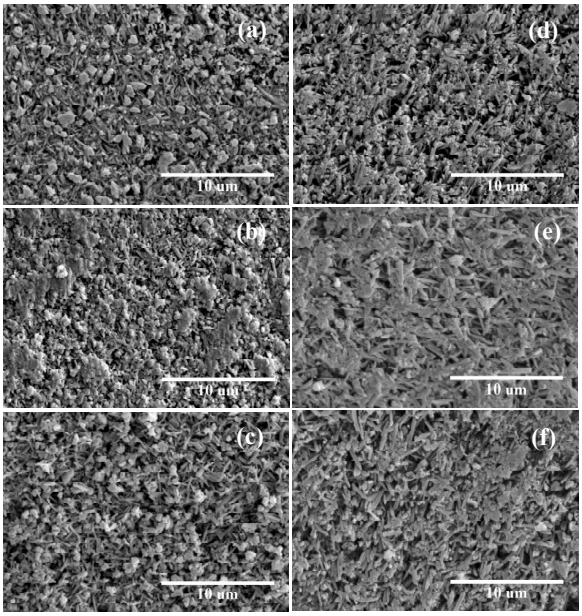


Fig. 5 SEM images of (a) LD1 as cast, (b) LD1 600˚C, (c) LD1 700˚C, (d) LD2 as cast, (e) LD2 600 ˚C, and (f) LD2 700 ˚C for 850˚C pressing temperature

IV. Conclusion

Two lithium disilicate (LD) glass compositions in this study were possibly fabricated by the hot pressing technique. No significance in glass composition could be observed during pressing. An initial heat treatment temperature induced different phase formation but rather has less effect on pressing. Pressing temperature rather play an important role in phase formation, microstructure (crystal size) and pressing ability of lithium disilicate glass ceramics. Therefore, it is necessary to carefully optimize the pressing temperature for suitable of each glass composition. In this study, we suggested LD1 heat-treated at 700˚C followed by hot pressing at 900˚C has a potentially used as a dental crown material.

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