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The Effect of Crystallization and Ion Exchange to the SLS Glass for High-Strength Transparent Bulletproof Materials

By

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Abstract:

A transparent bulletproof materials of soda-lime-silicate(SLS) glass has been ballistically tested after impacting by 5.45mm AK-74 steel core projectiles at distance of 50 meters. The thickness of satisfaction on bulletproof test was about 30mm. For application of transparent bulletproof materials, the SLS glass was heated by crystallization and ion exchange. The Vickers hardness, fracture toughness, and bending strength of crystallized SLS glass was about 704Hv, 0.9409MPa·$m^{1/2}$, and 452MPa which is about 24%, 31%, 201% higher than parent SLS glass, respectively. The vickers hardness, fracture toughness, and bending strength of ion exchanged SLS glass was about 657Hv, 0.7337MPa·$m^{1/2}$, and 791MPa which is about 16%, 2%, 430% higher than parent SLS glass, respectively. Transmittance of heat treated and SLS glass was decreased slightly at the visible range. The results prove that light-weight bullet proof can be fabricated by crystallization and ion exchange technique of SLS glass.

Keywords:

SLS glass, crystallization, ion exchange, bulletproof

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1. Introduction:

Transparent bulletproof glass is an important material in the defense industry. Bulletproof glass protected human life from the bullets. SLS glass was the most common form of produced glass. SLS glasses generally have excellent chemical durability and thermal properties, but poor mechanical properties [1,2]. SLS glass was consisted of 73% SiO_2, 14% Na_2O, 9% CaO, 0.15% Al_2O_3, 0.03% K_2O, 4% MgO, 0.02% TiO_2, 0.1% Fe_2O_3 [3,5,6] . The transition temperature (T_g) and crystallization temperature (T_c) were 564 ℃, 670 ℃, and thermal expansion coefficient (α) is 9.5 ppm /K at room temperature [4]. The hardness, fracture toughness, and bending strength of parent SLS glass were about 568 H_v, 0.7190 $MPa \cdot m^{1/2}$, and 150 MPa, respectively. For application of transparent bulletproof materials, SLS glass was heated by 2-step crystallization and ion exchange by employing screen printing technique in KNO_3 powder.

2. Experimental procedures:

SLS glass was prepared from finished product (KCC, 3.5 mm, Korea). For measurement of T_g, $T_{c(max)}$, measured by differential thermal analysis (TG/DTA-92, Setaram, France). DTA run was performed for SLS glass powder by 5 K/min heating rate. For measurement of the nucleation and crystal growth rates, glass samples were prepared 5 × 5 × 3.5 ㎜³ and polished with SiC paper (# 1,200 ~ 2,000). The nucleation and crystal growth rates were measured using two step heat treatment processes. SLS glass samples were heated at 5 ℃/min to the nucleation temperature between 525 ~ 625℃, (intervals of 25 ℃), then crystal growth was fixed at 700 ℃ (1 hr). The crystal growth temperatures ranged from 660 ~ 700 ℃ at intervals of 10 ℃. The nucleation condition was fixed at 575 ℃ for 1 hr. After cooling to the room temperature, crystallized glasses were determined to observe the number of nuclei (N_A) by optical microscopy (Olympus BX2M, Japan). The number of nuclei per unit volume (N_V) was calculated from [8]:

$$N_V = (\frac{2}{\pi}) \cdot N_A \cdot Y \quad (1)$$

where N_A is the number of nuclei per unit area and Y is the reciprocal of the mean diameter of nuclei. For all heat treated samples were excuted by X-ray differaction (D/max III, Rigaku, Japan) using Ni-filtered CuK_α radiation. Hardness and fracture toughness were measured using a Vickers Microhardness Tester (MXD-CX3E, Matsuzawa, Japan). More than 10 indentations were made for each specimen with a 10 second loading time at a maximum load of 500gf. The Vickers hardness was calculated from[1,7]:

$$H_V = 0.4636 \cdot (\frac{P}{a^2}) \quad (2)$$

where P is the load of indentation, a is the radius of indentation. The fracture toughness was calculated from[1,2]:

$$K_{IC} \cdot \Phi / H_V \cdot a^{\frac{1}{2}} = 0.15 \cdot K \cdot (c/a)^{-\frac{3}{2}} \quad (3)$$

where H_v is Vickers hardness, Φ is the restraint constant(≒3), and a is the radius of indentation, K is a constant(≒3.2), and c is crack length. For measurement of the mechanical strength, 3-point bending strength was employed to the bar type specimens using Universal Testing Machine (H10K-C, Hounsfield, U.K.).

$$\sigma = 3 \cdot P \cdot L / 2 \cdot w \cdot t^2 \quad (4)$$

where P is maxium load, L is outside the interval , w is width, t is thickness. To measure 3-point bending strength glass bars with diamensions of 3 × 4 × 36 ㎜³ were cut out from SLS glasses and polished. The KNO_3 powder (Ducsan, Extra pure, Korea) was prepared for ion exchanged. For measurement of the depth profile for Na^+ and K^+ was prepared Electron Probe Micro Analyzer (EPMA, JXA-8900R, JEOL, Japan). Transmittance was measured using UV/VIS/NIR Spectrometer (Jasco, V-570, Japan). The scan speed and range of wavelength were 400nm/min, in the range of 200 ~ 800 nm.

3. Results and discussion:

Fig. 1 shows that the glass transition temperature (T_g) and crystallization temperature ($T_{c,max}$) were 575 ℃, 680 ℃. The glass specimen was nucleated at 5 ℃/min to the temperature range of 525 ~ 625℃ with intervals of 25 ℃ for 1 ~ 144 hr, and then crystallized at 700 ℃ for 1 hr. The crystal growth temperatures ranged from 660 ~

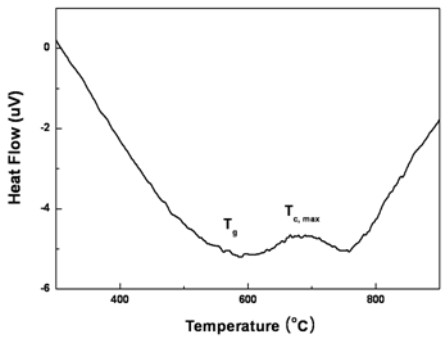

Figure (1): DTA curve of SLS glass at 5K/min heating rate

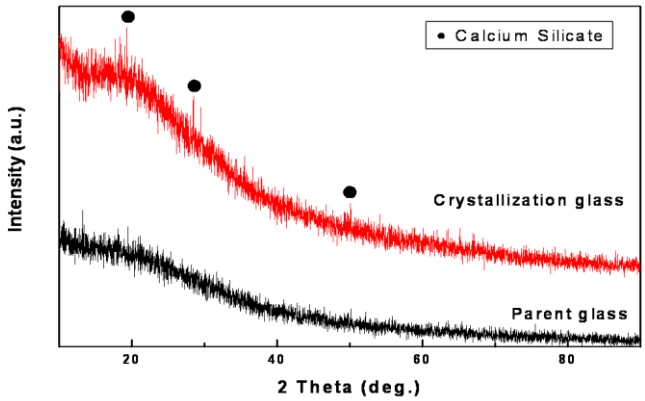

Figure (2): XRD patterns of parent and crystallized SLS glass(crystallization at 575 ℃ /144 hr, 650 ℃/0.5 hr)

700 ℃ at intervals of 10 ℃. The nucleation condition was fixed at 575 ℃ for 1 hr. It is found from the XRD data in Fig. 2 that precipitation of calcium silicate is observed. Fig. 3, 4 exhibits the nucleation and crystal growth rate as a function of temperature. The maximum nucleation and crystal growth temperature are 575 and 680 ℃, respectively, and they have values of 3.8078×10^5 /㎣·hr and about 21 ㎚/min, respectively.

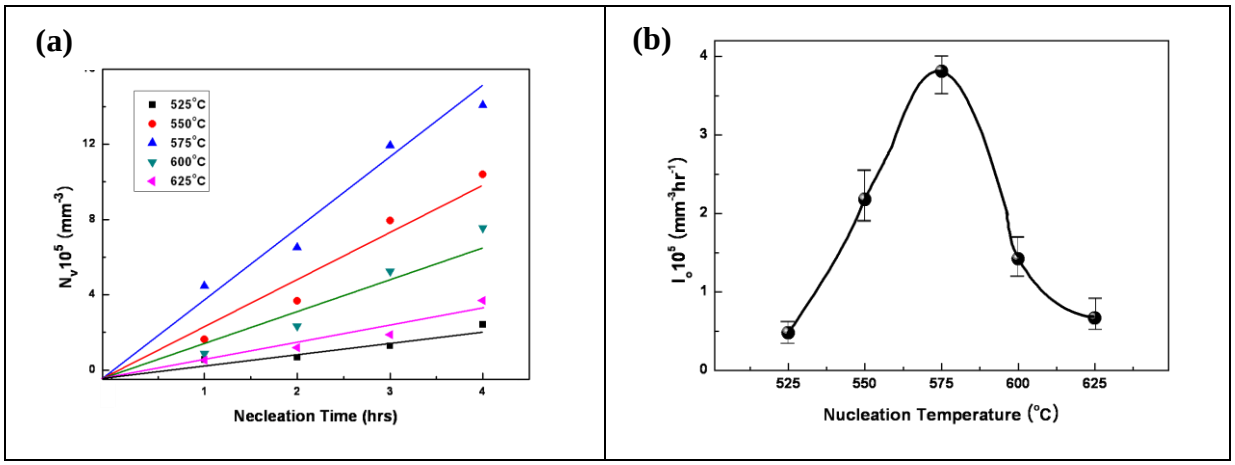

Figure (3): (a) Number of nuclei as a function of nucleation time(crystal growth at 700℃, 1hr), (b) Nucleation rates as a function of nucleation temperature for SLS glass

Fig. 5 shows that the change of nucleated number according to the nucleation time. The number of nuclei (N_A) was increasing with increasing the nucleation time from 162/㎟ (1 hr) to 567 /㎟ (4 hr). Fig. 4 shows the crystal diameter as a function of crystal growth time. It is also found out that crystal diameters are increasing with increasing the crystal growth time from 477 ㎚ (680 ℃ for 15 min) to 821 ㎚ (680 ℃ for 60 min).

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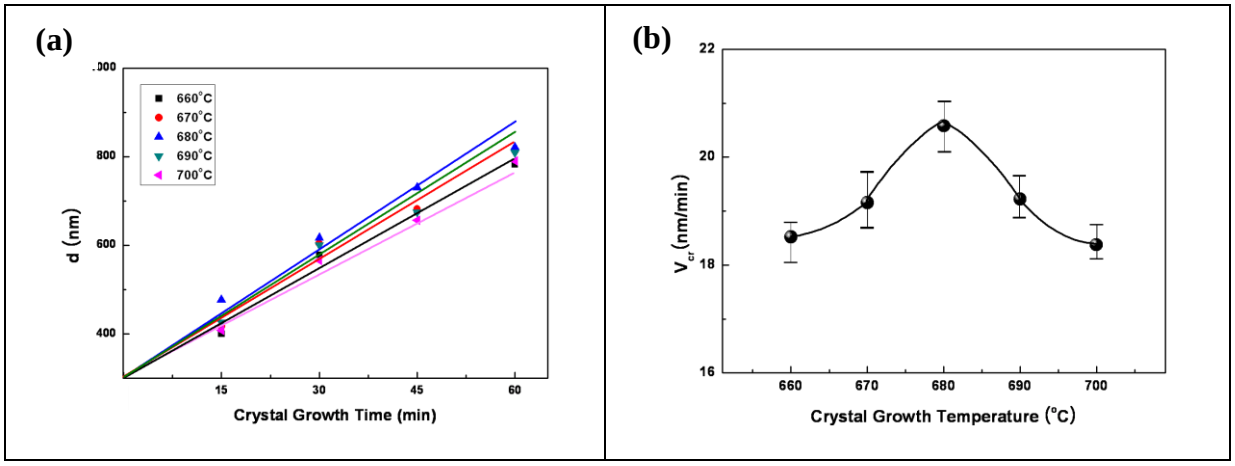


Figure (4): (a) Crystal diameter as a function crystal growth time for SLS glass (nucleation at 575℃, 1hr), (b) Crystal growth rates as a function of temperature for SLS glass

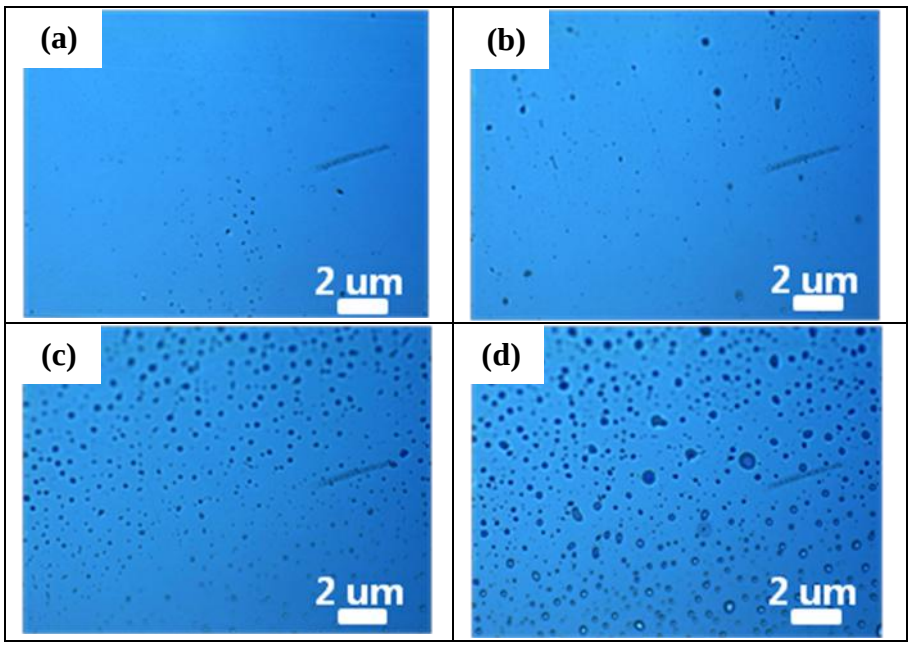
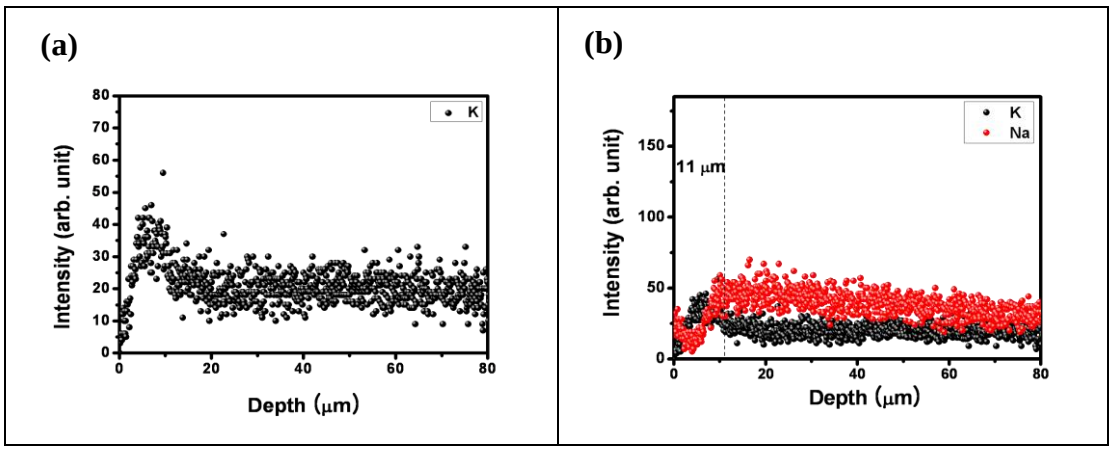


Figure (5): Optical microscope image of nucleated SLS glass at 575 ℃ (a) 1, (b) 2, (c) 3, and (d) 4 hr (crystal growth at 700 ℃ for 1hr).



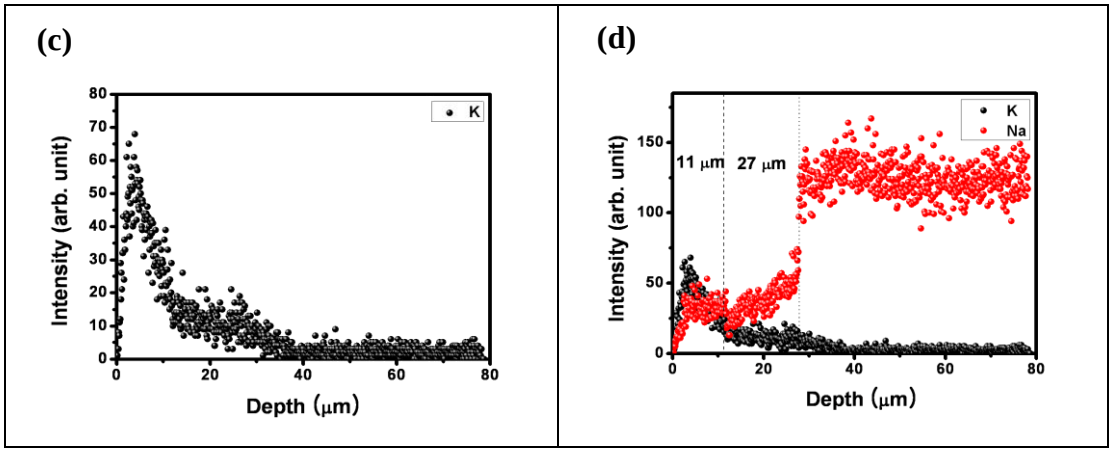


Figure (6): EPMA line profile of ion exchanged SLS glass by KNO_3 powder 480 ℃ at for (a), (b) 10min, (c), (d) 30min

Fig. 6 shows that EPMA line profile for the ion exchanged SLS glass was treated in KNO_3 powder containing different time. In this process, K^+, Na^+ ion exchange takes place at the glass surface and creates a compressed stress which raise to the mechanical strength of the glass [3]. With the increasing heat-treatment time from 10 min to 30 min, the depth profile was increasing from 11 ㎛ to 38 ㎛, but mechanical properties were reduced (Fig. 8). It was also found out that excessive heat treatment brings about stress relaxation [2]. Therefore, we have to find the best conditions of ion exchange according to the temperature and time. Fig. 7, 8 shows that mechanical properties of crystallized and ion exchanged SLS glass. The mechanical properties of crystallized SLS glass were increased by increasing the nucleation time. Because of the crystallized SLS glass was increased by increasing density by crystal phase.

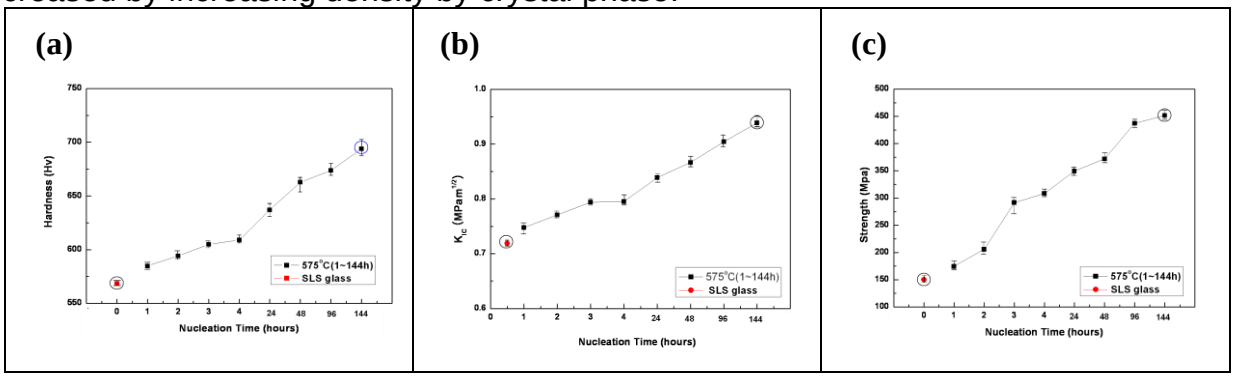


Figure (7): Hardness, Fracture toughness, and Strength of crystallized SLS glass at various nucleation times(crystal growth at 650 ℃ for 0.5 hr)

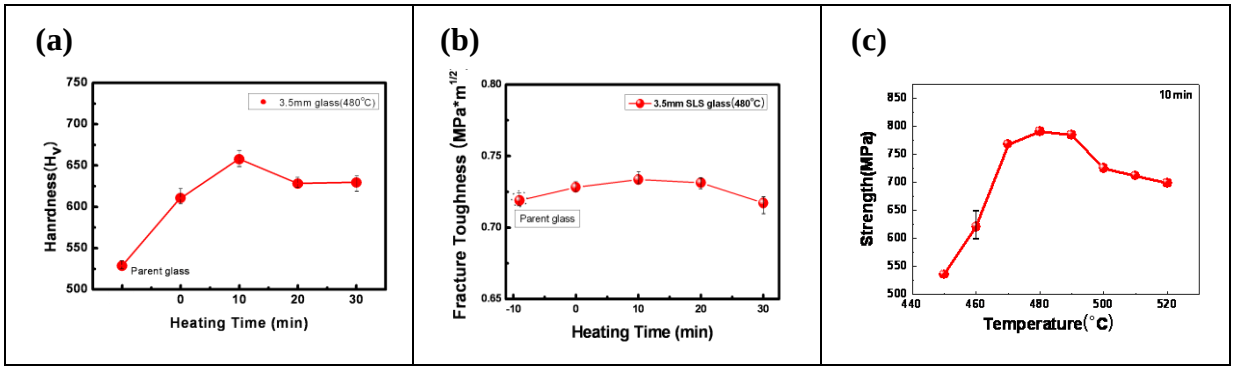


Figure (8): Hardness, Fracture toughness, and Strength of ion exchanged SLS glass at various time, temperature

The Vickers hardness, fracture toughness, and bending strength of crystallized SLS glass (at 575 ℃ for 144 hr, 650 ℃ for 0.5 hr) were about 704 H_v, 0.9409 MPa·$m^{1/2}$, and 452

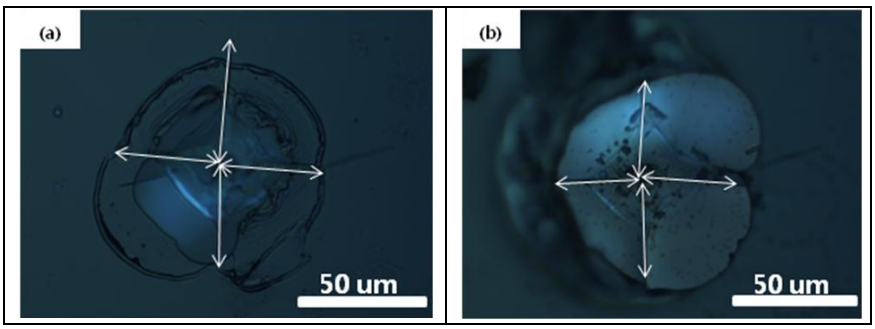


Figure (9): Crack length of SLS glass. (a) parent SLS glass, (b) crystallized SLS glass at 575 ℃ for 144 hr, 650 ℃ for 0.5 hr

MPa which is about 24%, 31%, 201% higher than parent SLS glass, respectively. The crack length of parent and crystallized SLS glass were 52.7 um, 50.3 um, respectively. The result revealed that the crack length was increasing of hardness, fracture toughness of glass (Table 1).

Table (1): Mechanical properties of crystallized SLS glass at various nucleation times(crystal growth at 700 ℃ for 1 hr)

	Nucleation Time (hr)	Hardness (H_v)	Fracture Toughness (MPa·$m^{1/2}$)	Strength (MPa)
Parent glass	0	568.7	0.7190	150
Crystallized SLS glass	1	584.8	0.7480	174
	2	594.2	0.7715	205
	3	604.9	0.7896	291
	4	609.0	0.7917	308

The Vickers hardness, fracture toughness, and bending strength of ion exchanged SLS glass (at 480 ℃ for 10 min) were about 657 H_v, 0.7337 MPa·$m^{1/2}$, and 791 MPa which is about 16%, 2%, 430% higher than parent SLS glass, respectively.

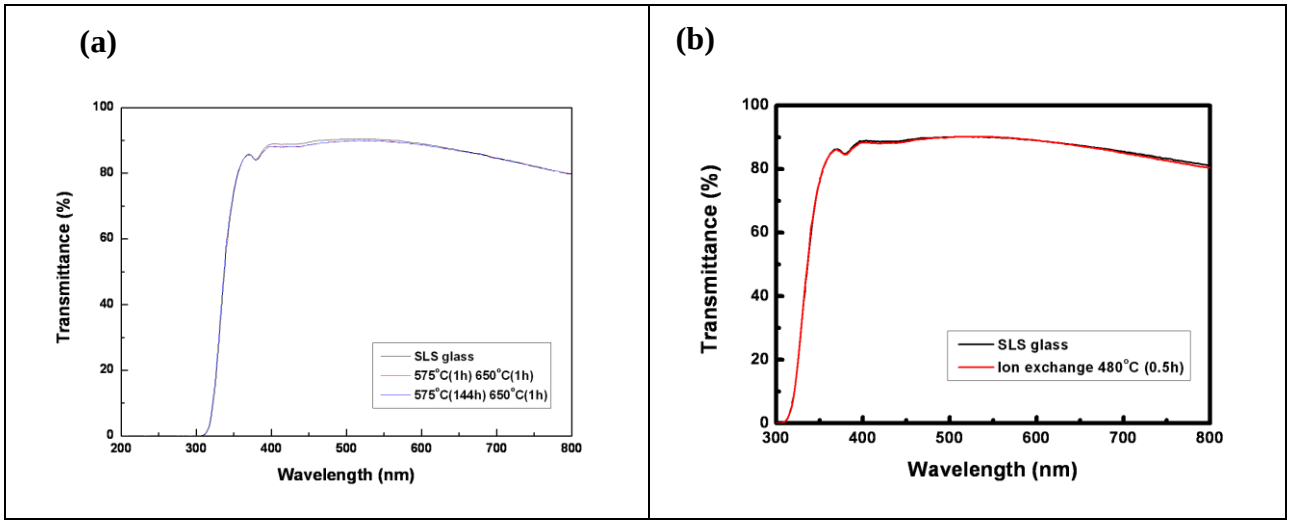


Figure (10): Transmittance of crystallized and ion exchanged SLS glass

Transmittance of ion exchanged and crystallized SLS glass was decreased slightly (within 0.5%). The visible transmittance was decreased with increasing crystal size. It is evident that the gradual decrease of visible transmittance is due to light scattering from the crystal phase particles. Scattering was increased with increasing crystal size.

4. Conclusions:

For application in light-weight bulletproof glass, SLS glass was strengthened by crystallization and ion exchange. Properties of crystallized and ion exchanged glass heated at different conditions were examined. As a result, The vickers hardness, fracture toughness, and bending strength of crystallized SLS glass (at 575 ℃ for 144 hr, 650 ℃ for 0.5 hr) were about 704 H_v, 0.9409 MPa·$m^{1/2}$, and 452 MPa which is about 24%, 31%, 201% higher than parent SLS glass, respectively. And Vickers hardness, fracture toughness, and bending strength of ion exchanged SLS glass (at 480 ℃ for 10 min) were about 657 H_v, 0.7337 MPa·$m^{1/2}$, and 791 MPa which is about 16%, 2%, 430% higher than parent SLS glass, respectively. Transmittance of ion exchanged and crystallized glass were decreased slightly at the visible range. A transparent bulletproof materials of soda-lime-silicate (SLS) glass has been ballistically tested after impacting by 5.45 mm AK-74 steel core projectiles at distance of 50 meters. The thickness of bulletproof materials were about 30 mm(Korea Military Academy). It can be expected of transparent bulletproof materials in more light-weight and thinner (over 10 ~ 15%) by ion exchange and crystallization.

Acknowledgements:

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