Subcritical crack growth and lifetime prediction of chemically strengthened aluminosilicate glass

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Abstract

The effect of residual stress on subcritical crack growth in chemically strengthened aluminosilicate glass in air and water was firstly investigated using the double torsion (DT) technique. An experimental evaluation procedure was developed based on the DT method. The research demonstrates that high compressive stress (CS) and low central tension (CT) in chemically strengthened glass are beneficial in improving crack growth index and decreasing susceptibility to fatigue. Chemically strengthened glass with high CS and low CT exhibits a smaller proof-test ratio, which indicates better survival characteristics. The results are useful in designing the strength and optimizing the strengthening process by ion exchange to obtain a more robust glass with long service lifetime.

Key words

Chemically strengthened glass; Subcritical crack growth; Lifetime prediction

1. Introduction

The evolution of glass defects depends not only on stress but also on physical and chemical interactions with environment. In fact, glass is subject to subcritical crack growth from mechanical defects, with the environment acting as a stress corrosion agent that triggers the evolution of mechanical defects until unstable fracture occurs even under constant external stresses that are well below the theoretical strength limit predicted by Griffith. This phenomenon is also known as static fatigue and has attracted considerable interest [1,2] .

Like most other corrosion, glass stress-corrosion is governed by a set of physical and chemical phenomena occurring at micro- and nano-scopic scale with the important parameters being the chemical composition of glass, presence of water or water vapor in the atmosphere [3,4], environment temperature, and acidity [5]. The most credited theory states that the phenomena occur from the rupture of silicon-oxygen bonds in the glass structure because of the presence of environmental water molecules in chemical reactions. Kinetic scales of these reactions involve absolute temperature, and the activation energy is provided by external stress.

Currently, several techniques have been used to observe the dynamics of subcritical crack growth during stress corrosion. These techniques include double cantilever beam technique [4], double cleavage-drilled compression method [6–8], and the double torsion (DT) method [9,10]. Among these methods, DT is the most widely used and reliable method for measuring subcritical crack growth curves ($V-K_I$) and failure prediction of glass, because the DT method has considerable stability of the four-point bending loading configuration. In addition, DT does not require difficult monitoring of crack length during testing.

Many studies have been conducted on crack evolution behavior of silicate glass[11-18]. However, to our knowledge, no data on subcritical crack growth of chemically strengthened glass exist[19,20]. The exchange of small alkali ions in silicate glass by larger ions from a molten salt bath below T_g produces a compressive stress (CS) in the order of 100–800 MPa on the glass surface, which results in glass strengthening. Compared with thermal tempering process, chemical strengthening is advantageous because the developed surface compression is usually much higher. Thus, no measurable geometric distortion generally occurs, and the method can be readily applied to relatively thin and complex geometry products, such as tubes [21–24]. The stress distribution around Griffith crack tips varies after chemical strengthening because of high CS on the glass surface, which may result in different subcritical crack growth behaviors. However, the effect of CS on subcritical crack growth in chemically strengthened glass remains unclear to date.

In this paper, the topic addressed is how CS affects subcritical crack growth with a focus on chemically strengthened aluminosilicate glass. We also aim to gain new insights into the mechanism of stress corrosion and static fatigue of chemically strengthened glass. Results illustrate that higher CS and lower central tension (CT) in chemically strengthened glass causes lower susceptibility to fatigue. Glasses with high CS and low CT have smaller proof-test ratios, thereby indicating better survival characteristics.

2. Experimental procedure

The glass used in this work was 1.8 mm-thick aluminosilicate glass (Corning Gorilla 2318). According to the previous research [9], a variety of DT specimen geometries can be used. In this research, specimen and load geometry for precracking

and load relaxation are illustrated in Figure 1 (b). The specimen was a thin plate with dimensions of 90 mm $\times$ 30 mm $\times$ 1.8 mm (L $\times$ W $\times$ d). All specimens contained a laser-machined notch of 10 mm in length and 0.15 mm in width. A single side groove with a width (W_g) of 2.0 mm and web thickness (d_n) of 0.9 mm was machined using a diamond wheel. Single side groove specimens were placed on the test fixture with the side groove on the compressive surface of the specimen. The specimen is best loaded and supported by ball bearings. The distance between two loading ball bearings was 4 mm. The experimental configuration for glass precracking and load relaxation are illustrated in Figure 1(a).

Figure 1(a) Experimental configuration for glass precracking and load relaxation. (b) Specimen and load geometry for precracking and load relaxation. (c) Typical image of indentation at the tip of the notch.

The ion exchange process was performed in an electric furnace. Prior to ion exchange, all glass specimens were annealed at 550 °C for 8 hours (8h) to remove residual stress in the glass. In the ion exchange process, annealed glass specimens were immersed into molten pure KNO₃ (purity > 99.9%) at 420 °C for different

durations (1, 3, and 12 h). K^+ ions in the salt diffused into the glass surface, and the exchanged Na^+ ions diffused into the molten salt. At the end of each ion-exchange cycle, glass specimens were carefully cleaned with deionized water. The magnitude of residual stress (CS and CT) and depth of stress layer (DOL) of the specimens after ion exchange were measured by the surface stress meter (FSM-6000LE) which is based on the theory of photoelasticity[25]. Firstly, the glass sample is put on the measuring area and make sure the glass surface (no groove side) fit with the triple prism to guarantee the birefringence fringes are detectable and clear , and then input parameters such as thickness, refractive index, photoelastic constant of glass and so on. At last, the CS, CT and DOL of chemically strengthened glass can be obtained.

The energy of crack initiation is considerably higher than that of propagation in chemically strengthened glass because the high CS (≥ 400 MPa) on the glass surface results in difficulty in precracking of chemically strengthened glass. Therefore, two methods were used to guarantee successful precracking. Firstly, the chemically strengthened glass specimens were annealed in the electric furnace at 500 °C for 3 h to decrease crack initiation energy. Then, an indentation was induced at the tip of the notch by Vicker indenter to ensure precracking along the groove (Figure 1c). The indentation force was 20 N. The CS, DOL, and CT of chemically strengthened aluminosilicate glass before and after annealing was shown in Table 1. It can be seen that glass with highest CS before annealing have lowest value after annealing. Glass with lowest CS before annealing have highest value after annealing. This phenomena may be related to the thermal history of chemically strengthened glass. With the ion exchange time increasing (long thermal history), the process of ion exchange may be more thoroughly, the glass structure was inclined to adapt the high temperature. In

addition, more fully stress relaxes due to structural relaxations associated with viscous flow during ion exchange process with longer time lead to the CS be less sensitive to the temperature[26].

Table 1 Compressive stress, depth of stress layer, and central tension of chemically strengthened aluminosilicate glass before and after annealing.

Ion Exchange Temperature (°C)	Ion Exchange Time (h)	Before Annealing			After Annealing		
		CS (MPa)	DOL (μm)	CT (MPa)	CS (MPa)	DOL (μm)	CT (MPa)
420	1	704	21	9	124	67	5
420	3	667	41	16	175	80	9
420	12	540	92	31	213	108	13

The interrelation between CS and CT is approximated by Equation (1) [27]:

$$CT = \frac{CS \times DOL}{T - 2 \times DOL} \quad (1)$$

Where CT is the central tensile stress, CS is the compressive stress, DOL is the depth of stress layer and T is the thickness of glass. It should be noted that a linear stress profile is assumed in the derivation of this formula. In actuality, the stress profile is non-linear resulting in some amount of error. At a given thickness, interrelation between these three parameters(CS, CT, DOL) is reasonable according to Table 1.

The precracking method involved the use of a constant displacement rate. The specimen was loaded at a slow crosshead speed (0.005 mm/min), until a crack formed and grew to the desired length (>20 mm), as noted by a rapid decrease in the load. Accordingly, maximum load P_{IC} can be obtained after precracking.

The precracked specimen was loaded at a relatively rapid crosshead speed (0.25 mm/min) to a load P ($0.9P_{IC}$), which is necessary to initiate the rapid fracture of the specimen. With sufficient load the crack starts moving rapidly, that is, a large relaxation is observed, the crosshead is arrested, and the load is monitored as a function of time. The duration of the test was 30 min. After the test was completed, the specimen was removed from the loading fixture, and the final crack length was determined through dye penetration. By measuring P and the corresponding dP/dt , K_I and V can be calculated according to following equations [28]:

$$K_I = PW_m \left[\frac{3}{Wd^3d_n(1-\nu)\xi} \right]^{1/2} \quad (2)$$

$$V = da / dt \Big|_y = -(Pa)_{i,f} \cdot dP / dt \Big|_y / P^2 \quad (3)$$

where ν is the Poisson's ratio, ξ is corrected factor, and a_i and a_f are the initial and final crack length, respectively. The dynamics of crack propagation during stress corrosion can be described uniquely by the following power law between stress intensity factor (K_I) and crack velocity (V) for many materials [29]:

$$V = AK_I^n \quad (4)$$

where A is a constant and n is crack growth index. Smaller n indicates greater susceptibility to fatigue.

To investigate the effect of water on subcritical crack growth in chemical strengthened glass, several specimens were dipped in deionized water for 24, 48, and 72 h at 25 °C. Precracking and load relaxation of these specimens are also completed in deionized water at 25 °C.

The fracture toughness (K_{IC}) of chemically strengthened aluminosilicate glass was also obtained by DT method. Specimen geometry and precracking method are the

same. Once precracked, the specimens were unloaded for crack length measurement and then failed at a stroke rate of 2 mm/min. Five samples were tested in each group. Fracture toughness was calculated from the following [29]:

$$K_{IC} = P_m W_m \left[\frac{3}{W d^3 d_n (1-\nu) \xi} \right]^{1/2} \quad (5)$$

where P_m is the fracture load.

3. Results and discussion

Figure 2 shows the typical load relaxation curve of chemically strengthened aluminosilicate glass (CS = 124 MPa). The inset shows a typical image of the crack growth process. As shown in the image, the load relaxation rate decreases with increasing time, thereby indicating that the crack velocity is decreasing.

Figure 2 Typical load relaxation curve of chemically strengthened aluminosilicate glass. The inset is the typical image of the crack growth process at different time.

Fracture toughness as a function of CS and CT for chemically strengthened aluminosilicate glass is shown in Figure 3. From Figure 3(a), mean fracture toughness is decreasing with the increase in compressive stress. The mean fracture toughness of annealed glass (CS=0MPa) is $0.73 \pm 0.02 \text{ MPa}\cdot\text{m}^{1/2}$, which is consistent with a previous work [30]. However, for chemically strengthened glass, mean fracture

toughness values are only 0.72 ± 0.02 , 0.65 ± 0.02 , and 0.62 ± 0.02 MPa·m^{1/2} for CS values of 124, 175, and 213 MPa, respectively. Fracture toughness as a function of CT also have the same change tendency with CS as shown in the inset of Figure 3(a). In order to fully investigate the influence of CS and CT on fracture toughness, the contour plot of CS–CT– K_{IC} relationship of chemically strengthened aluminosilicate glass was shown in Figure 3(b). Although mean fracture toughness values decrease with CS and CT in single plot of CS- K_{IC} and CT- K_{IC} as shown in Figure 3(a), the CS-CT- K_{IC} relationship demonstrate that the mean K_{IC} values increased with increasing CS and decreasing CT, thereby indicating that fracture toughness depends on the relative values of CS and CT (CS/CT). High CS and low CT in chemically strengthened glass is beneficial for improving K_{IC} .

Figure 3 (a) CS- K_{IC} and CT- K_{IC} (inset) diagrams for chemically strengthened aluminosilicate glass. The red line indicates fitting results. (b) The count color plot of the CS-CT- K_{IC} relationship.

Fracture of glasses and ceramics is always initiated by tensile stress and can often be traced to the propagation of surface flaws through the bulk material[31,32]. A practical means of increasing the tensile strength of ceramics is to create a

compressive skin on the surface. The replacement of smaller mobile sodium ions in the glass phase with larger potassium ions is the most common exchange process and is the type used in this system. This type of process has been demonstrated by multiple researchers to be an effective means of improving the breaking strength of glass [33]. For chemically strengthened glass, high CS and low CT improve breaking strength[34]. In consequence, mean K_{IC} values increase with increasing CS and decreasing CT.

Figure 4 shows the fracture behavior of chemically strengthened aluminosilicate glass at 25 °C at different soaking times in water. The red line indicates fitting results using Equation (4). We can see that all crack velocity and stress intensity factor relationships can be described well by Equation (4). The effect of CS and CT on the fracture behavior of chemically strengthened glass at 25 °C in air is shown in Figure 4a. Figure 4a shows that the crack grew rapidly with increasing stress intensity factor. For annealed glass, the crack velocity reached 7.2×10^{-5} m/s when the stress intensity factor is approximately $0.49 \text{ MPa}\cdot\text{m}^{1/2}$. Compared with that in annealed glass, crack velocity in chemically strengthened glass is only 1.0×10^{-5} m/s (CS = 124 MPa, CT=5MPa) and 2.8×10^{-5} m/s (CS=175MPa, CT=9MPa). The effect of increasing CS and CT (CS=213MPa, CT=13MPa) is shown as a shift of curves toward a lower stress intensity factor. For a given stress intensity factor, the crack velocity in chemically strengthened glass increases with CS, which may due to the larger CT in chemically strengthened glass with higher CS (Table 1). The crack growth in chemically strengthened glass is affected by both CS and CT (CS/CT relative value). With considerably higher CT (lower CS/CT relative value), the chemically strengthened glass will be inclined to spontaneously fracture due to the disequilibrium of CS and

CT. That is to say there may exist a threshold for CS/CT relative value on the crack velocity. If CS/CT relative value is larger than the threshold, CS dominates the crack growth, otherwise CT is dominant. Figure 4b shows the effect of CS and CT on the fracture behavior of chemically strengthened aluminosilicate glass at 25 °C after 24 h soaking time in water. Compared with $V-K_I$ diagram in air, the curves of glass that is soaked for 24 h shift toward a lower stress intensity factor. According to a previous research, water molecules can enhance stress-corrosion crack growth in glass and ceramic [35,36]. The proposed mechanism indicates a chemical reaction between water and strained Si-O-Si bonds at the crack tip. In addition, tensile stress is produced at the crack tip because of the exchange of hydrogen ions in the water with sodium ions in glass, which can also enhance crack growth [37]. The effect of CS and CT on the fracture behavior of chemically strengthened aluminosilicate glass at 25 °C and after 48 h and 72 h soaking time in water is shown in Figure 4c and 4d, respectively. The effect of decreasing CS/CT relative value is seen as a shift of curves toward a lower stress intensity factor which is consistent with the results in air. However, as shown in Figure 4b, the crack velocity in glass after 24 h soaking time is higher than that after 48 h and the shift of curves is different from other three figures. This may result from crack healing effects (e.g. crack tip blunting) due to soaking in water in the absence of tensile stress[5]. When a crack forms in glass, water from the surrounding environment rushes into the open crack and from there diffuses into the glass via the crack tip. Diffusion of the water occurs because high tensile stresses at the tip enhance the rate of diffusion enormously[38]. Once in the glass structure, water causes the glass to swell, and because the glass is constrained from expanding freely, a compressive stress builds up at the fresh fracture surface which arrests the

crack propagation[38]. This phenomenon needs further exploration.

Figure 4 Fracture behavior of chemically strengthened aluminosilicate glass at 25 °C for different soaking times in water: (a) 0 h; (b) 24 h; (c) 48 h; and (d) 72 h. The red line indicates fitting results using Eq. (4)

In addition, the crack growth index, n , can also be obtained by fitting the $V-K_I$ data. The CS– n relationship of chemically strengthened aluminosilicate glass at different soaking times is shown in Figure 5a. The red line indicates the fitting results by the following exponential function:

$$y = \exp(Ax^2 + Bx + C) \quad (6)$$

where y is the crack growth index, x is the CS or CT of chemically strengthened glass, and A , B , and C are constants.

Figure 5 (a) Experimental CS– n relationship of chemically strengthened aluminosilicate glass at different soaking times. The red line indicates fitting results. (b) CS– n relationship of chemically strengthened aluminosilicate glass at different soaking times obtained by Eq. (6).

The fitting results in Table 2 show that the CS– n relationship can be excellently described by the exponential function. Crack growth index, n , decreased with increasing CS. The crack growth index in air is higher than that in water, which indicates that the susceptibility to fatigue of glass in water is larger than that in air. Figure 5b shows the prediction results for the CS– n relationship of chemically strengthened aluminosilicate glass at different soaking times by Equation (6). According to a previous work, the variation of n broadly ranges from 12 to 50, depending on numerous parameters, such as water, temperature, and pH [29]. In this paper, the minimum value of $n=12$ is employed. Therefore, the corresponding CS can be obtained, namely, approximately 220 (72 h), 250 (48 h), 270 (air), and 300 MPa (24 h). These CS values may be the upper limit for chemically strengthened glass that can be precracked. Otherwise, if the compressive stress in chemically strengthened glass is higher than upper limit, the energy of crack initiation during precracking will be increased. When the energy of crack initiation is higher than crack propagation

during precracking, the fracture will occur. The lower limit of CS for chemical strengthened glass is 0 MPa. Thus, to observe subcritical crack growth in chemically strengthened glass, the required maximal CS for chemically strengthened glass are about 220 (72 h), 250 (48 h), 270 (air), and 300 MPa (24 h).

Table 2 Fitting results of CS- n relationship by Eq (6)

Environment (Soaking time)	A	B	C	R ²
Air	-1.21E-5	6.65E-4	3.20	0.970
24h	-6.49E-6	2.86E-4	2.97	0.998
48h	-8.47E-6	6.72E-4	2.83	0.994
72h	-1.43E-5	1.42E-3	2.86	1.000

CT- n relationship of chemically strengthened aluminosilicate glass at different soaking times is shown in Figure 6a. The red line indicates fitting results by Equation (6). The values of fitting parameters are shown in Table 3. We can see that CT- n relationship can also be well described by the exponential function. The crack growth index, n , in air is higher than in water, thereby indicating glass is greater susceptibility to fatigue in water than in air. Figure 6b shows the prediction results for the CT- n relationship of chemically strengthened aluminosilicate glass at different soaking times by Equation (6). Similarly to the CS- n relationship, the required maximal CT to observe subcritical crack growth in chemically strengthened glass can also be obtained, with valued of about 13 (72 h), 16 (48 h), 18 (air), and 21 MPa (24 h).

Figure 6 (a) Experimental CT– n relationship of chemically strengthened aluminosilicate glass at different soaking times. The red line indicates fitting results. (b) CT– n relationship of chemically strengthened aluminosilicate glass at different soaking times obtained by Eq. (6).

Table 3 Fitting results of CT– n relationship by Eq (6)

Environment (Soaking time)	A	B	C	R^2
Air	-1.23E-3	-1.56E-2	3.20	0.920
24h	-6.80E-4	-9.00E-3	2.97	1.000
48h	-7.97E-4	-8.68E-3	2.84	0.961
72h	-2.14E-3	7.74E-4	2.86	0.984

To evaluate the relationship between CS, CT, and n , a contour color plot of CS–CT– n at different soaking times is shown in Figure 7. Crack growth index is evidently dependent on CS and CT. High CS and low CT in chemically strengthened glass is beneficial for improving crack growth index and consequently decreasing susceptibility to fatigue.

Figure7 Experimental CS–CT– n relationship of chemically strengthened aluminosilicate glass at different soaking times. (a) 0 h; (b) 24 h; (c) 48 h; and (d) 72 h

The most effective approach toward failure prediction for glass is proof testing [39]. The proof test is designed to impose a load that is larger than the maximum expected service load on a component. Thus, a limit is set for the maximum size of flaws that can be present in the component after the completion of the proof test. The minimum time-to-failure, $t_{\min}$, also known as the service lifetime for glass, can be obtained from crack propagation data using Equation (7) [40–43].

$$t_{\min} = 2\sigma_a^{-2} (K_{IC}\sigma_a / \sigma_p)^{2-n} / AY^2(n-2) \quad (7)$$

where σ_a is a constant applied stress, σ_p is the proof stress, σ_p/σ_a is the proof test ratio, A is a constant, and n is the crack growth index, which can be obtained by fitting the V – K_I data. Y is the shape factor.

Figure 8 Proof-test diagrams for chemically strengthened aluminosilicate glass with different residual stress (air). (a) CS = 0 MPa, CT=0MPa; (b) CS = 124 MPa, CT=5MPa; (c) CS = 175 MPa, CT=9MPa; and (d) CS = 213 MPa, CT=13MPa.

Figure 8 shows the proof-test diagrams for chemically strengthened aluminosilicate glass with different residual stress (in air), (a) CS = 0 MPa, CT=0MPa; (b) CS = 124 MPa, CT=5MPa; (c) CS = 175 MPa, CT=9MPa; and (d) CS = 213 MPa, CT=13MPa. In these logarithmic plots of the expected minimum failure time versus service stress, a series of straight lines were obtained (with a slope of -2), with each line corresponding to a different proof-test ratio (σ_p/σ_a). We take one glass product as an example. To ensure the survival of this product, a survival time of ~5 years should be guaranteed by the proof test. At a service stress of 20 MPa, a proof-test ratio of 3.5 is required for chemically strengthened glass (CS = 124 MPa, CT=5MPa) to obtain 5

year survival, compared with a ratio of 4.5 for annealed glass. Thus, for the same survival time, a thicker plate of annealed glass must be used. However, for chemically strengthened glass with residual stress values of (CS = 175 MPa, CT=9MPa) and (CS = 213 MPa, CT=13MPa), proof-test ratios of 4.1 and 4.7, respectively, are required. Thus, proof-test ratio is dependent on the residual stress.

Figure 9 CS–CT–proof-test ratio diagram of chemically strengthened aluminosilicate glass.

To evaluate the relationship among CS, CT, and proof-test ratio, a contour color plot of CS–CT–(σ_p/σ_a) is shown in Figure 9. It can clearly be seen that high CS and low CT in chemically strengthened glass is beneficial for decreasing the proof-test ratio, which indicates that chemically strengthened glass with higher CS and lower CT presents better survival characteristics. These results provide useful information for optimizing the processing of chemically strengthened glass.

4. Conclusions

In summary, the effect of residual stress on the subcritical crack growth of chemically strengthened aluminosilicate glass in air and in water has been firstly

investigated by fracture mechanics techniques. An experimental evaluation procedure has been developed based on the double torsion method. High CS and low CT in chemically strengthened glass are beneficial in improving crack growth index and decreasing susceptibility to fatigue. High CS and low CT for chemically strengthened glass shows a smaller proof-test ratio, thereby indicating better survival characteristics. These results would help optimize the strengthening process by ion exchange to produce robust glass with long service lifetimes and have application for many industries including front windshield for commercial aircraft.

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