Thermal and Chemical Strengthening Effects on Crack Initiation and Strength of Float Glass

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Crack-initiation behavior of chemically and thermally strengthened float glass was studied using a recording microindenter. Post-test microscopy was performed to characterize differences in indentation patterns and crack lengths. Dramatic differences in crack initiation were observed between the samples. Strengths of specimens impacted with quartz sand were measured in biaxial flexure. The ion-exchanged specimens had the highest strengths in the as-received condition. However, the thermally strengthened specimens had the highest strengths after impact. The higher strengths after impact shown by the thermally strengthened specimens correlate well with the fact that these specimens also had the shortest radial cracks. The combination of recording microindentation testing and strength testing proved to be an effective way to compare the behavior of these specimens when subjected to contact damage. The thermally strengthened glasses tested in this study showed the best resistance to cracking in the microindentation tests, and they had the highest strengths after impact with sharp quartz-sand particles.

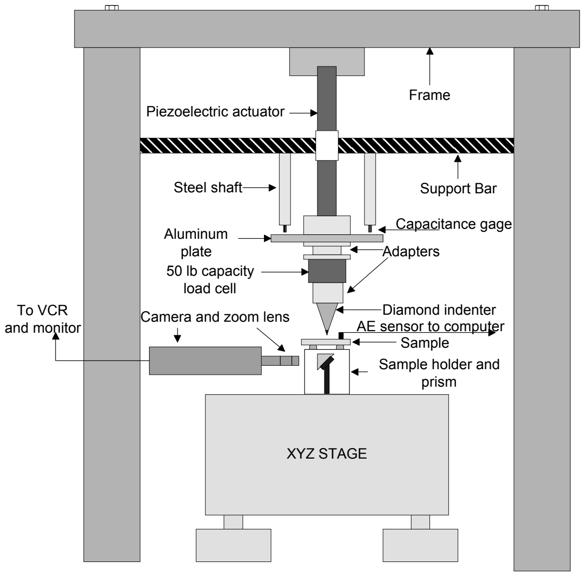
Introduction

Contact between a glass surface and hard objects (particles or larger objects) often produces cracks that lead to strength reductions. Surface compressive stresses provide some protection against the production of contact damage. The two standard methods for producing surface compressive stresses (with balancing internal tension) are thermal and chemical strengthening, but the stress profiles are very different for these two methods. Thermal strengthening produces a compressive layer that extends to a depth below the surface that is about one-fifth of the glass thickness. Chemical strengthening produces a compressive layer that is on the order of 100 μm deep. Since the degree of protection against contact damage and the subsequent influence on strength should be related to the level and profile of surface compressive stress, the present investigation was designed to study this correlation using established laboratory techniques of producing contact damage using microindentation.

Experimental Procedure

Float glass specimens subjected to five different strengthening treatments were used in the study. Three ion-exchange and two thermal-tempering treatments were used, and annealed float glass was also included. The experimental set-up used to make the indentations and to record the acoustic emission and cracking processes is shown schematically in Figure 1. A piezoelectric actuator was used to push a Vickers diamond into the sample, while two capacitance displacement gages and a strain-gage load cell recorded the displacement into the glass and load on the indenter, respectively. An acoustic emission sensor was attached to the sample via high-vacuum grease, and a video camera system recorded the entire process on tape. The resolution of the displacement gages was ~ 14 nanometers, while the load cell resolution was ~ 0.74 grams (0.0073 Newtons).

Figure 1. Schematic representation of the experimental set-up used to study the crack initiation behavior.



The indenter, acoustic emission, and video camera systems were started simultaneously. The displacement rate of the indenter into the glass was set at 0.2 μm/s, and the maximum load was set to 2 Kg. Upon reaching the maximum load, the indenter was withdrawn at the same rate until the load reached zero grams. The formation of cracks was monitored throughout the entire loading/unloading cycle as a function of load. The tin sides of the samples were indented. Fifteen indentations were made on each sample, and indentations were performed far enough apart such that interactions between indentations were of no concern. Post-test microscopy was performed on the indentation sites.

Biaxial flexure strengths of the supplied specimens were measured before and after impact with quartz sand. The concentric-ring method was used, with the diameters of the supporting and loading rings being 44 mm and 16 mm respectively. Ten specimens were broken for each test condition.

Results

For all treatments, no radial cracks initiated on loading. This was expected based on previous work in the area[1]. However, acoustic emission was detected on loading to differing degrees for the different samples. In particular, the thermally tempered samples had the most acoustic emission activity, followed by the annealed sample, and then the three ion-exchanged samples for the loading portions of the tests. Note that all of the tests on the thermally strengthened samples had activity on loading, while this was not true for the ion-exchanged and for the annealed samples. Table I summarizes all the acoustic emission activity for the different samples. The numbers in brackets for the hits on loading refer to the number of tests out of fifteen that exhibited acoustic emission activity on loading. IE 1-3 are the ion-exchanged specimens, with depths of compression of 66 μm (IE 1), 125 μm

(IE 2), and 150 μm (IE 3). The thermally strengthened specimens are TS 1 (DSR = 25) and TS 2 (DSR = 30).

Table I. Acoustic emission data summary.

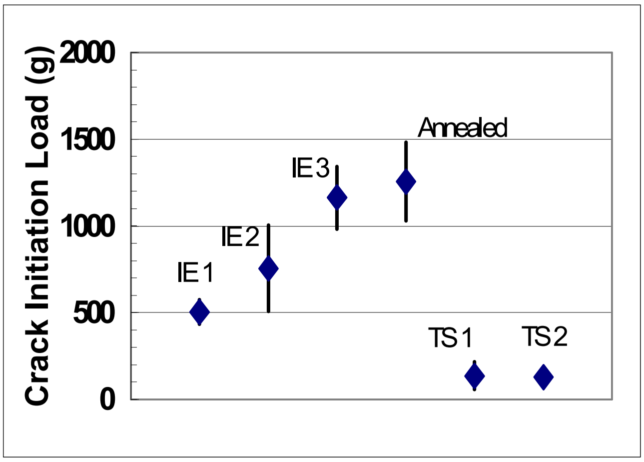
Specimen	Hits on Loading	Hits on Unloading	Total AE Energy
IE 1	0.5 ± 0.6 (5/15)	6.7 ± 2.5	34.2 ± 19.1
IE 2	0.5 ± 0.9 (4/15)	6.9 ± 2.5	16.8 ± 15.3
IE 3	0.1 ± 0.4 (2/15)	8.2 ± 3.3	25.7 ± 19.6
Annealed	1.6 ± 1.6 (10/15)	3.3 ± 1.5	74.9 ± 9.1
TS 1	5.6 ± 2.3 (15/15)	5.7 ± 1.3	29.5 ± 12.2
TS 2	11.9 ± 2.6 (15/15)	6.1 ± 1.8	38.5 ± 18.7

On unloading, all indentation tests for all six treatments exhibited acoustic emission activity, which was nearly the same, except that for the annealed sample, which showed lower activity on unloading.

On unloading, primary radial cracks initiated for all six treatments and for all indentations. The cracks 'popped-in' suddenly and were always detected by the AE unit. Four primary radial cracks always formed, but not all four initiated at the same time. Primary radial crack formation was sometimes accompanied by the formation of secondary radial cracks shortly afterwards. Deep lateral cracks formed near the end of complete unloading, and additional secondary radial cracks sometimes accompanied the lateral cracks. Sometimes secondary radials only initiated with the lateral cracks. Shallow lateral cracks, which broke through to the surface occasionally, occurred as well.

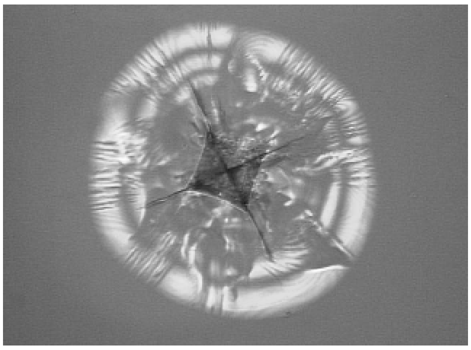
The most obvious difference in crack initiation was the load at which the first primary radial cracks initiated on unloading for the different samples. Figure 2 shows the averages and standard deviations for this in graphical form. The thermally tempered glasses had primary radial cracks form at the lowest loads on unloading. For the same maximum test load, differences in the loads at which cracks 'pop-in' is indicative of the different stress levels in the glasses as a result of the different strengthening methods. In order for the stress to build-up to the critical level required for crack initiation, the internal compressive stresses must first be overcome. For the tempered glasses, there must have been higher compressive stresses present at the initiation depth in the glass, compared to the other samples, thus requiring the reaching of a lower load during unloading to build the stress level up to the critical value. The depth of strengthening is just as important a factor as the stress level at that depth in the crack initiation behavior.

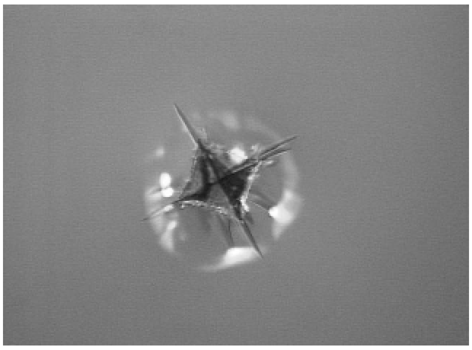
Figure 2. Plot of the data for crack initiation loads during unloading showing the large differences in these loads for the different specimens.



Indentation site morphology also showed differences, particularly between the thermally tempered glasses and the ion-exchanged glasses. Much larger subsurface lateral cracks were observed in the ion-exchanged samples, compared to the tempered samples, as seen in Figure 3. Primary radial crack lengths for the tempered and exchanged glasses were similar, but much longer cracks were observed in the annealed sample.

Figure 3. An indentation site in IE 2 ion-exchanged glass (left) and thermally tempered glass, TS 1 (right). Both images are 400 μm wide by 300 μm tall.





The strength results are summarized in Table II. The ion-exchanged specimens have the highest strengths in the as-received condition, as expected. Very high surface compressive stresses can be obtained through chemical strengthening. However, the thermally strengthened specimens have the highest strengths after impact with the sharp quartz-sand particles. The greater depth of surface compression helps in strength retention after impact under these conditions. Comparing the strength results with the crack-initiation results, the higher strengths after impact shown by the thermally strengthened specimens correlate well with the fact that these specimens also had the shortest radial cracks.

Table II. Summary of strength data.

Specimen	Strength before impact (MPa)	Strength after impact (MPa)
IE 1	448 ± 17	53 ± 4
IE 2	366 ± 76	66 ± 12
IE 3	287 ± 14	94 ± 37
Annealed	165 ± 27	36 ± 3
TS 1	214 ± 41	116 ± 12
TS 2	227 ± 42	119 ± 11

Summary

The combination of recording microindentation testing and strength testing proved to be an effective way to compare the behavior of these specimens when subjected to contact damage. Recording microindentation provides a way to study cracking behavior under uniform, well-controlled conditions. Impact with particles is less uniform and controlled, especially with respect to particle-to-particle variations in size and shape. However, impact testing more closely duplicates what happens to the glass under service conditions.

The thermally strengthened glasses tested in this study showed the best resistance to cracking in the microindentation tests, and they had the highest strengths after impact with sharp quartz-sand particles.

Acknowledgments

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[1] R.F. Cook and G.M. Pharr, J. Am. Ceram. Soc. **73**, p. 787 (1990).