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The mechanical performance of bi-treated glass

M. Zaccaria & M. Overend
Glass & Façade Technology Research Group, University of Cambridge, UK

ABSTRACT: Fully toughened glass (FTG) and chemically toughened glass (CTG) are the types of glass currently available if enhanced strength is required. However, whilst CTG shows a very high strength, it does not break safely; conversely FTG has a lower strength, but a safe failure. An ideal glass could be made combining the strength of CTG with the fragmentation of FTG. A new toughened soda lime silicate glass (SLSG), processed both thermally and chemically (T+C) or vice-versa (C+T), termed bi-treated glass (BTG) has been produced by Trend Marine Ltd. And is investigated by the authors. Before testing the specimens in a coaxial double ring (CDR) set-up, the stress profile was measured using photoelastic equipment. The current version of BTG failed to satisfy the strength and fragmentation requirements. This paper reports on the performance of the BTG and discusses the possible ways of improving it.

INTRODUCTION

In recent years a larger proportion of glass has been used for structurally challenging applications. Transparent roofs and staircases are just two examples of how designers are pushing the boundaries of the material. In order to have a more reliable structural behaviour the material can be subjected to toughening and laminating. Toughening can be performed thermally or chemically and it provides the glass with a pre-stress that prevents flaws from growing until the surface pre-compression is exceeded. These processes will be better examined in section 1. Laminating improves post-fracture behaviour by retaining the glass fragments after failure, but is not object of this paper.

Designing with glass involves several challenges and one of them is picking the suitable type of glass. Annealed glass (AG), FTG or CTG differ considerably in terms of strength and fragmentation. Table 1 shows a comparison of glass properties. It can be seen that CTG provides a considerably higher surface pre-compression (i.e. higher tensile strength), but it does not break safely whereas FTG has a lower surface pre-compression, although significantly higher than AG, but it breaks safely. Whichever glass is chosen, a commonly used design strategy is the `fail-safe approach` i.e. accept that glass can break during its service life and ensure that the consequences do not involve human injury or loss of business.

There is demand for an ideal glass that should be as strong as CTG, but that would also break safely like FTG. This would particularly be the case for applications where optical clarity is vital, therefore monolithic glass is preferred.

Table 1: Surface pre-compression and fragmentation of SLSG glasses

Type of glass	Surface pre-compression* (MPa)	Safe failure
AG	3-11	No
FTG	90-130	Yes
CTG	280-330	No

*Data from photoelastic analysis

In theory, in order to produce a glass with these properties a combination of thermal and chemical toughening would be needed. The authors named such a glass BTG. A first BTG process was patented by Hess et al. (1962), but is not clear if and how it actually performed better than either FTG or CTG.
More recently, a prototype batch of BTG was produced by Trend Marine Ltd., and investigated at Cambridge University. This paper presents a non-destructive analysis made with a scattered light polariscope (SCALP) to determine the stress profile on AG, FTG, CTG, BTG. It also describes the destructive tests, performed with a CDR set-up, to determine the strength of the different types of glass and to evaluate the performance of BTG compared to the other types of glass. Finally, the paper combines the results from the photoelastic analysis and the CDR test to establish if and how the surface residual stress is related to the strength.

1 RESIDUAL STRESS PROFILE

The most common method currently used to strengthen architectural glass is thermal toughening. The principle of the method was already known in the seventeenth century (“the Prince Rupert drop”) and it consists of cooling the glass rapidly from a temperature above its transition temperature T_g. In this way a residual stress of parabolic shape is generated across the glass thickness with compression at the surface, tension in the core and zero stress at about 21% of the thickness, figure 1.
Chemically toughened glass was developed in the late 1950’s and the most common process for SLSG consists of placing the glass in a salt bath of KNO_3 at a temperature of about 450°C for a time $\geq$ 8 hours. In this way the larger K^+ ions will replace Na^+ in the surface of the glass, creating a surface pre-compression of higher intensity than FTG, but not very deep. In general, the longer the process, the deeper the penetration of ions, which is usually $\leq$ 100 μm, figure 1. The level of stress obtained is also affected by the chemical composition of the glass. namely, it varies with the alkali-oxide concentration as Burggraaf & Cornelissen (1964) showed.

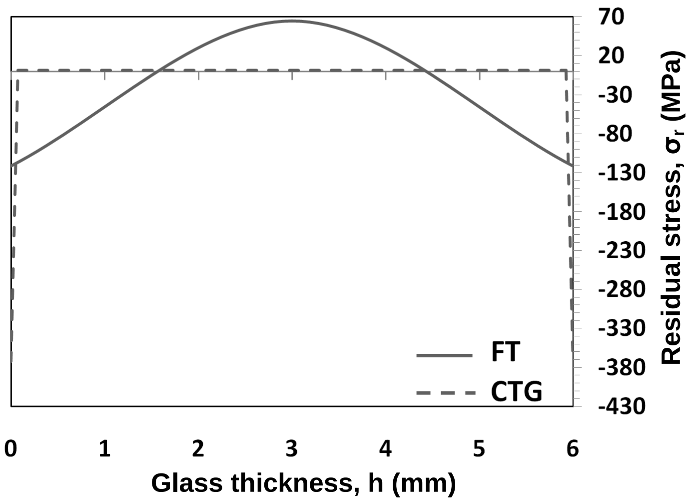

Figure 1: FTG and CTG stress profile

The stress profile provides information about the strength and fragmentation. Therefore glass properties can be measured with a simple non-destructive optical analysis. For example, equation 1 below should provide the strength of any type of toughened glass:

$$f_{app} = f_{AG} - \sigma_{rs} \tag{1}$$

where f_{app} = apparent strength, f_{AG} = inherent strength of AG, σ_{rs} = surface residual stress. However, as discussed in section 3, this equation has never been validated.

A relationship between the central tension and the weight of the fragments was proposed by Barsom (1968) and it is valid for FTG only:

$$\sigma_{rm}^{4} \left(\frac{M}{h}\right) = 5.489x10^{25} N^{5} / m^{9} \tag{2}$$

where σ_{rm} = residual stress at mid-thickness, M = mean weight of a glass fragment, h = thickness.

The depth of the compressive zone is also crucial, as a flaw deeper than that would considerably weaken the glass.

Therefore, in order to obtain a toughened glass with a desired behaviour it is necessary to engineer its stress profile.

A batches BTG processed thermally and then chemically (T+C), and vice versa (C+T) have been produced by Trend Marine Ltd and investigated by the authors. Although the resulting stress profiles differed from the FTG and CTG neither of the BTG glasses matched the requirements of high strength and safe failure (figure 4).

The ensuing parts of this paper will explain how the non-destructive and destructive test results have been combined with the purpose of understanding why the produced BTG do not perform as expected.

2 TEST PROCEDURE

SLSG panels with dimensions 300 x 300 x 6 mm, 10 specimens for each of the following glass types were investigated: AG, FTG, CTG, BTG (C+T), BTG (T+C). The glass was firstly scanned with SCALP-04 and SCALP-05 to read the stress profile.

The panels were then tested on a coaxial double ring (CDR) jig, figure 2. Since there is a difference in strength between the tin side and the air side, Howes (1978), a UV light device was used to detect it and for comparability of the results all samples were tested with the tin side as the tension face. Lusas finite element (FE) software (version 14.6) was used to calculate the stress at failure following the procedure explained in Zammit & Overend (2009). The fragmentation count was performed on an area where the load-induced stress was equal to 0.

Figure 2: test set-up.

2.1 Scalp scanning

The SCALP is a scattered light polariscope that uses a polarized diode laser, an optical modulator and a camera to measure the residual stress in glass. Details about its operation principles are provided by Aben (1993) and Anton & Aben (2003).

At the time that the investigation began SCALP-04 was available on the market. It allows to read through the whole thickness the stress profile of AG and FTG, but not for CTG. In the late 2012 the SCALP-05 was released. It features a flatter inclination that detects the stress distribution also for glass with shallow compressive layer, as CTG and BTG. It measures up to a depth of 2.2 mm though, so, not the whole thickness.

Readings were made on three points per face of glass panel. Two orthogonal readings were taken at each point: one in the x direction and one in the y direction. The locations of the points were the center of the panel, the loading and support circumferences (figure 3). The results of the investigation are shown in table 2.

The expertise of Glasstress Ltd. was required for BTG as the entry point of the laser beam needed to be adjusted for any type of glass. AG and FTG were investigated with a SCALP-04, CTG and BTG with SCALP-05.

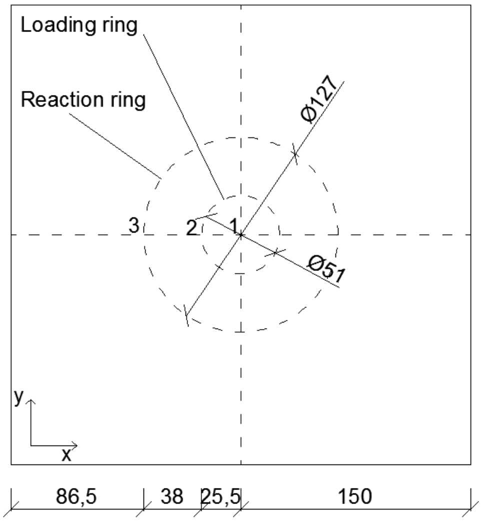

Figure 3: plan view of glass sample measurement points

Figure 4 shows the stress profile for both types of BTG. It can be seen that for both processes the resulting surface pre-compression is higher than FTG, but lower than CTG (data of FTG and CTG pre-compression are shown in table 1 and 2), but only BTG (C+T) broke safely. The readings of figure 4 were taken at the Glasstress Ltd. laboratory and they clearly show the potential of modifying the stress profile with high pre-compression and high central tension. However, measurements and analysis conducted in Cambridge gave different outcomes, with a pre-compression for the BTG (C+T) even lower than FTG. These results for the BTG (C+T) are currently being investigated further to establish which of the two used instruments performed the correct measurements. This uncertainty may be due to the point at which the light enters the glass, parameter that may vary from glass to glass.

2.2 CDR results

Coaxial double ring tests (loading ring has diameter 51 mm, the reaction ring 127 mm) were performed to determine the load at failure which was then used as input in lusas FE software to calculate the stress at failure. Mean values are reported in table 2. The speed of the crosshead was 0.025 mm/s which leads to a test duration of approximately 2 minutes.

In order to preserve the fragments a plastic film was attached on both sides of the glass. After failure, fragments in a squared area of 50 x 50 mm were counted as indicated by the EN12150-1:2000, figure 5. The geometry and the loading conditions differed from the above-mentioned standard, but the fragment count was made over an unstressed area. Results of the count are shown in table 2. The table also include values of the coefficient of variation, defined as the standard deviation over the mean.

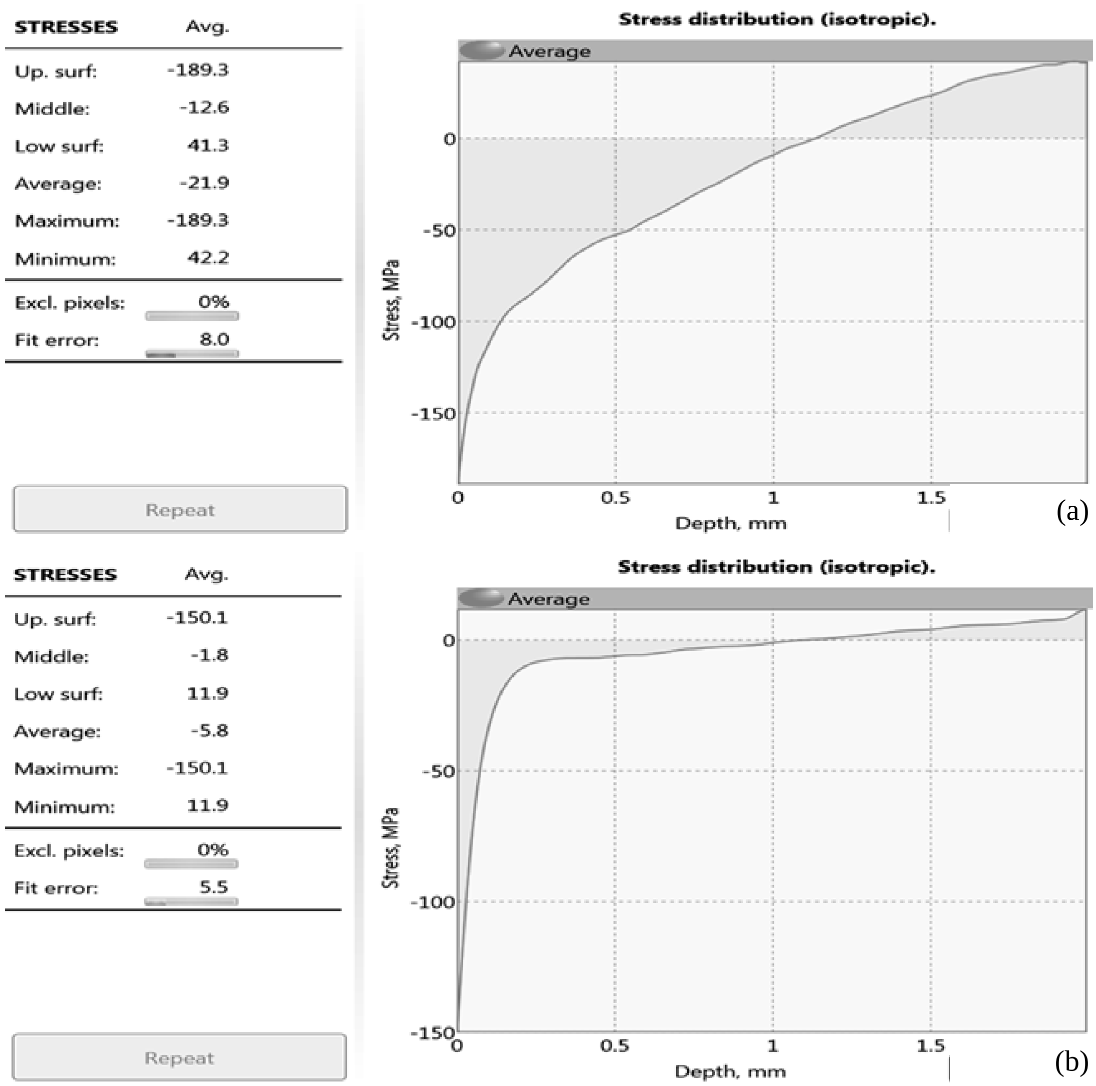

Figure 4: SCALP reading of a) BTG (C+T) b) BTG (T+C), SCALP software screenshot.

Table 2. summary of non-destructive and destructive tests. Coefficient of variation is shown in brackets

Glass type	Surface residual stress, σ_{rs}		Mid-thick res. Stress, σ_{rm}		Depth at $\sigma_{rs} = 0$		Mean failure stress		Mean effective stress*		Fragment count
	(MPa)	(%)	(MPa)	(%)	(mm)	(%)	(MPa)	(%)	(MPa)	(%)	
AG	-4	21	0.7	41	2	15	101	21	97	23	4
FTG	-117	7	59	15	1.16	5	288	30	171	50	178
CTG	-327	10	0.9	14	0.1	15	568	24	241	65	31
BTG (C+T)	-108	4	57	18	1.13	3	214	20	105	40	177
BTG (T+C)	-146	8	7.4	8	1.09	3	488	22	342	31	39

*Effective stress = failure stress – surface residual stress.

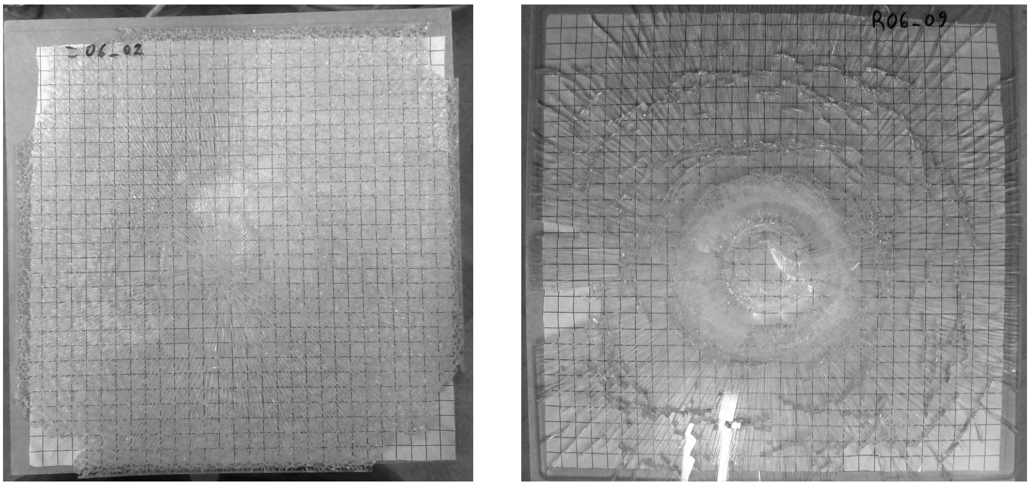

Figure 5: BTG fragment count: left BTG (C+T) breaking safely, right BTG (T+C) breaking unsafely.

3 DISCUSSION

Table 2 compares the results of the non-destructive and destructive analysis. CTG is the strongest glass tested, followed by BTG (T+C), FTG, BTG (C+T) and AG. The surface residual stress follows the same ranking indicating that the two are related. However, in order to validate equation (1), the effective stress should be a constant and equal to f_{AG}, namely the strength of the material. This was not the case, this agrees with the hypotesis of crack healing whereby Nielsen et. al (2010) put foward an equativo to account for this phenomenon:

$$f_{app} = f_{AG} - \sigma_{rs} + f_{other} \tag{3}$$

where f_{other} represents the crack healing due to the temperature of the process, its magnitude is function of the temperature and of the type of process, but it has not been determined yet.

A prediction of the number of fragments using equation (2) has not been conducted yet, but is part of the future work. However, from the fragment count made according to the EN12150:1-2000 it can be noted that FTG and BTG (C+T) are the only ones to comply with the standard and can be classified as safety glass. This agrees with the measured mid-stress value. Depth at 0 stress was small for CTG only, whereas all the other process allowed a deeper compression zone.

At this stage it can be concluded that if BTG is stronger than FTG, but it does not break safely. Whereas if it breaks safely is not as strong as FTG. Therefore, FTG and CTG are both preferable than BTG. The cause of these under-performances are probably due to the high temperature of the process, which triggers a stress relaxation. The observation that can be made is that the last process in the BTG eliminates the previous one.

Our future work in this field is to improve the performance of BTG by investigating and subsequently combining the two constituent processes. The combination will happen at a later stage when the interaction of the two processes, especially regarding the stress relaxation has been established. In particular, the methods and the techniques to use will be:

- Nielsen's model (2010) to simulate the residual stress due to tempering;
- Generate experimental stress profile plots of SLSG undergoing chemical toughening for 3 glasses differing in alkali-oxide concentration for 3 different times of treatment and 3 different temperatures, as Spoor & Burggraaf (1966) did for the aluminosilicate glass.
- The combination of the two models will indicate whether is preferable to process the glass thermally and chemically (or vice versa) as well as giving the optimal process parameters.

4 CONCLUSIONS

Different types of toughened glass along with AG have been analysed non-destructively and destructively. It has been shown that as expected a higher surface residual stress corresponds to a higher strength, but the final relationship still needs to account for crack healing that occurs during the treatment processes. The main aim of the research was to evaluate BTG. It has been shown that the BTG glass tested can provide a strength higher than FTG or a safe failure, but it was not possible to achieve both characteristics simultaneously. This study, together with further modeling will be used to investigate ways of optimising the BTG. The expectations are that BTG will be stronger than FTG with a safer failure than CTG. BTG would be suitable for applications that require strength, optical clarity and safe fragmentation in a monolithic glass format.

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