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DOI
10.1007/s40940-017-0047-4

Publication date
2017

Document Version
Final published version

Published in
Glass Structures and Engineering

Citation (APA)
Veer, F., Bristogianni, T., & Baardolf, G. (2017). A case study of apparently spontaneous fracture. *Glass Structures and Engineering*, *3 (2018)*, 109-117. https://doi.org/10.1007/s40940-017-0047-4

Glass Struct. Eng.
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RESEARCH PAPER

A case study of apparently spontaneous fracture

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Received: 24 October 2016 / Accepted: 15 August 2017

Abstract Spontaneous failures in thermally toughened glass are frequently reported, although the actual percentage is quite small, suppliers in the Netherlands reporting one or two cases a year while thousands of tempered panels are supplied. These cases are rarely investigated in the Netherlands because the broken thermally toughened glass is usually not in one piece and can thus not be sent for analysis. In 2015 several sliding thermally toughened glass single curved vehicle roof windows failed within several months. A butterfly pattern was seen and NiS failure was suspected by the window manufacturer. As a coloured adhesive foil had been applied during manufacturing the glass shards were kept together. The broken glass was shipped back to the supplier and was together with several non-broken roof windows sent to Delft University of Technology for analysis. The compressive surface pre-stress in the intact roof panels was measured with a SCALP 5 device. It was found that the compressive surface pre-stress varied, as bands of low compressive surface pre-stress alternating with bands of high compressive surface pre-stress appeared in the specimens. Destructive testing of the intact windows showed that the windows failed in the bands of low compressive surface pre-stress. The actual bending strength was far below the strength thermally toughened glass should have. Microscopic analysis of the butterfly fragment of the "spontaneously" failed roof windows showed that the failure was not caused by any inclusion in the glass, but started at the surface of the glass and was probably due to overloading. It is concluded that the failures were not spontaneous but the result of overloading due to uneven tempering of the glass.

Keywords Spontaneous failure · Uneven tempering · Quality control

1 Introduction

Every glass specialist has experience of or knows stories of spontaneous failure of thermally toughened glass. In actual fact, the percentage of spontaneous failures in modern glass is quite small. The association of spontaneous failure of thermally toughened glass with NiS inclusions comes from the early days of float glass where several times batches were contaminated and several production runs turned out to have significant problems. Improvements in the float process eliminating sulphur from fuel and Ni from the raw material that is molten have reduced the occurrence of NiS failures significantly. A good analysis of the problem is given by Jacob (2001). The common practice of heat soaking should eliminate most of the NiS failure (Gelder 2001), however NiS failure cannot be entirely eliminated through heat soaking. Even though it is rare, spontaneous failure of thermally toughened glass can have significant consequences (Harris et al. 2003a, b). Using the right methodology NiS failures can be prop-

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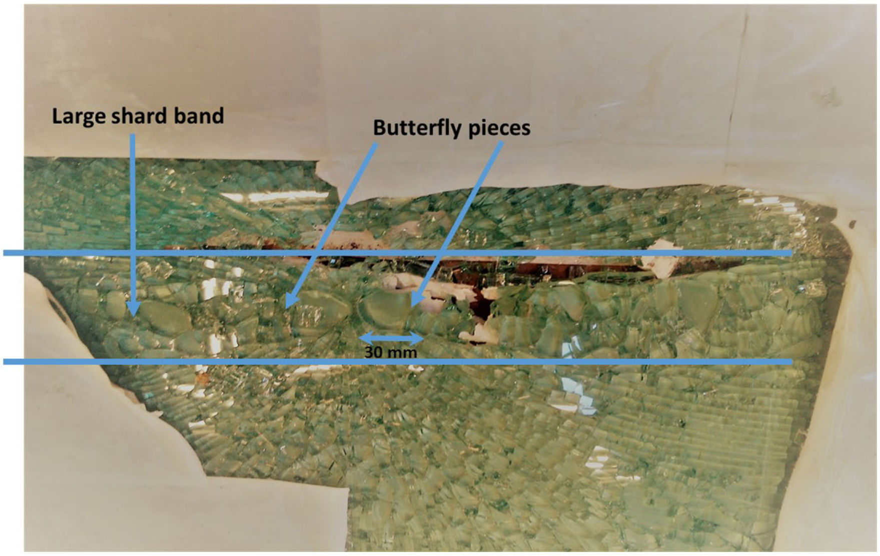


Fig. 1 Band of large shards and butterfly in spontaneously failed vehicle roof window supplied to authors after in service failure. Length of butterfly fragment around 30 mm, width of small shards about 4 mm

erly identified (Harris et al. 2003a, b). The physics of the NiS problem is explained by (Yousfi et al. 2010). The probability aspects of NiS failure are explained by (Schneider et al. 2012).

The problem in practice is that monolithic thermally toughened glass usually falls down in a shower of small shards. The puzzle of putting it together and finding the critical fragments at the source of the failure is usually omitted. With laminated thermally toughened glass the failure pattern can be easily identified but usually the presence of a butterfly is taken as an indication of NiS failure. As (Harris et al. 2003a, b) shows this is a dangerous assumption as butterfly like patterns are also visible when failure is caused by other causes.

The harsh reality is that most spontaneous failures of thermally toughened glass are not investigated. The actual percentage of NiS failures among the total of spontaneous failures in modern glass is unknown. There are other inclusions in glass and spontaneous failure can occur for other reasons than NiS inclusions (Cave 2016). The automatic conclusion of NiS failure as the cause of spontaneous failure should thus be considered premature.

In 2015 an opportunity for academic research into a case study occurred when a manufacturer of sliding vehicle roof windows experienced several spontaneous failures over a period of several months while in the years before no problems were experienced. The failures took place under different climatic conditions, in both warm and cold weather. Although thermal stress from the window being warmed by solar radiation might be a contributory factor in certain failures, the failures in cold weather imply that this could not be the main reason for the failures.

As the thermally toughened 8 mm thick non-laminated curved sliding roof windows were covered with a coloured self-adhesive polymer foil the fragments were held together and a clear butterfly pattern was visible as the source of the fracture. It was also clear that there were bands of large glass shards alternating with band of small glass shards.

The broken windows and several surplus non-broken windows were supplied to the Delft University of Technology Glass research group for analysis.

2 The spontaneously broken windows

The spontaneously broken windows showed two clear characteristic features. Firstly, there were alternating bands of large and small shards, illustrated in Fig. 1, indicating uneven tempering. Secondly there were pairs of shards that looked like a butterfly pattern at what looked like the initial point of failure from the radiating fragmentation pattern centred on the butterfly, as is shown in Fig. 2. It should be noted that as the glass was monolithic and only held together by the foil, the butterfly shards were slightly displaced, showing a gap between them. The butterfly shards were carefully removed and fractographically examined.

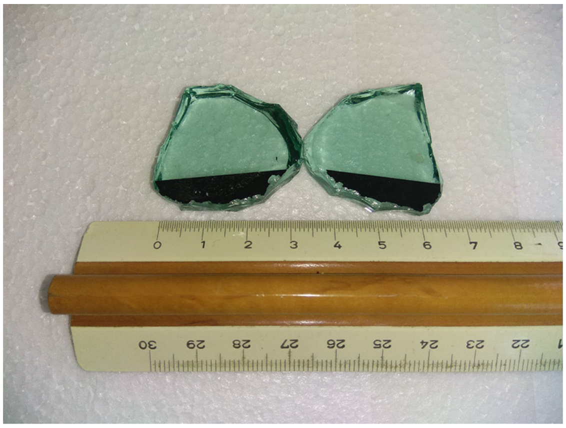


Fig. 2 Butterfly fragments removed from "spontaneously" failed window

3 Chemical analysis of the spontaneously broken windows

X-ray fluorescence (X-RF) of fragments of the spontaneously failed windows was conducted. Fragments close to the initial fracture origin were removed. Approximately 2000 mm^2 was used for the analysis. This showed that the local chemical composition was within the tolerances for the composition set by the codes. No Ni could be detected. This either denotes an absence of Nickel in the fragments investigated or an amount less than the detection threshold. The absence of Ni does however not preclude NiS failure as the NiS inclusions are localised. An analysis of a limited volume does not therefore guarantee that NiS present in the plate will be found.

The small variances in composition between the different windows suggests that the original glass was made on different days. This in itself is an indication against NiS effects as it is unlikely the production would have been contaminated with Ni repeatedly or over a long period without more problems being reported. As far as is known there have been no other complaints about the glass produced by this factory. The chemical compositions are given in Table 1. The SnO_2 content in the spontaneously broken window is the result of the Tin side being used for the X-RF analysis. Any values given as 0 are below the detection threshold of 0.01–0.05% depending on the atomic weight of the element.

4 Compressive surface pre-stress measurements with a SCALP 5 device

Using a factory calibrated SCALP 5 device with the GlassStress 5.8 software the compressive surface pre-stress was measured on the supplied intact windows, according to the procedure given by (Veer and Rodicheff 2016). Across the width of the specimen a band was marked with paper tape, halfway along the length of the specimen. On the paper tape the measurement intervals were marked with a marker. Each interval was 40 mm long, this is the contact length of the SCALP 5 sensor on the substrate. This was considered enough to detect

Table 1 Chemical composition of float glass and the three fractured roof panels

Constituent	Float glass composition by the norm (wt%)	Non-spontaneously broken window tested in laboratory (wt%)	Spontaneously broken window 1 (wt%)	Spontaneously broken window 2 (wt%)
SiO_2	71.9	74	72.7	74
Al_2O_3	0.08	0.7	0.5	0.4
MgO	5.64	4.1	3.9	4.1
CaO	9.23	7.7	7.7	7.9
Na_2O	13.1	12.8	12.3	12.6
K_2O	0.02	0.06	0.12	0.11
Fe_2O_3	0.04	0.52	0.50	0.62
MnO_2	0	0.02	0.01	0.01
TiO_2	0.01	0.03	0.02	0.02
SO_3	0	0.10	0.06	0.14
SnO_2	0	0	2.28	0

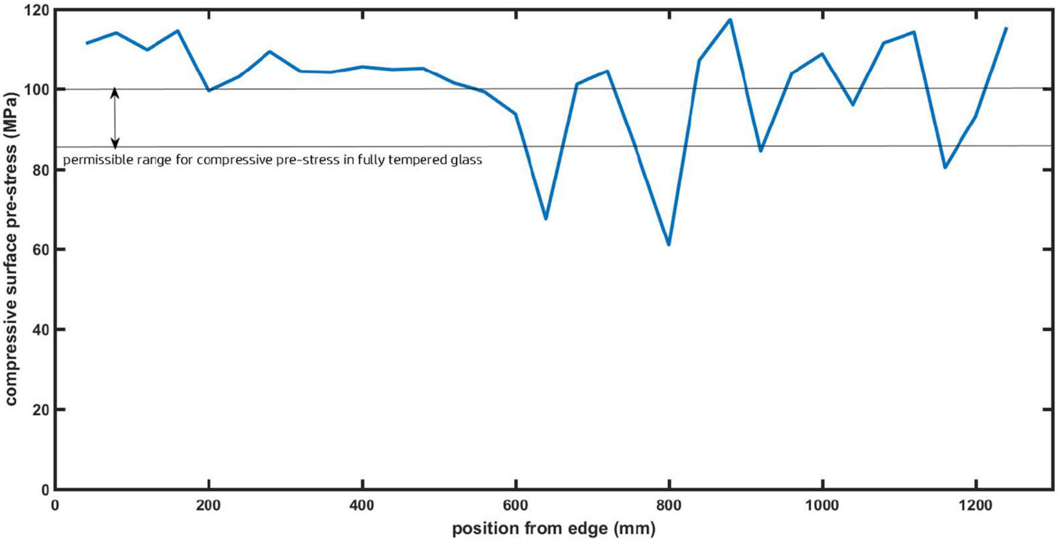


Fig. 3 Measured compressive surface pre-stress at the centre of the length of the window, measured across the width of the window from left edge to right edge for specimen 1

a pattern of inhomogeneous pre-stress. Interval 1 being at one edge of the specimen and the last interval being at the other edge of the specimen. Within this band, measurements were conducted from one side to the other in a single session. The compressive surface pre-stress was measured repeatedly at the first location and the last location and was found to be consistent within 5%, which is considered the standard measuring error for a SCALP 5. After that single measurements were made at the marked locations, measuring a series of local stresses in a band along the width of the specimen. The surface was cleaned with propanol before each measurement, fresh coupling liquid was applied, and the measurement was conducted. This is essential to make reliable measurements. Moving the SCALP 5 device even slightly along a glass surface redistributes the contact liquid. A too thin contact liquid layer can result in anomalous readings with very high or very low results. Very high or low readings can thus result from improper measuring technique.

It should be noted that Glass Stress has modified the software in 2015 and introduced a calibration program for the SCALP 5 sensors. The combination of calibration and the new software makes the measurements much more consistent and reproducible than was possible before 2015. However, it should be noted that the SCALP 5 really needs a skilled operator and consistent measuring technique. High local peaks in pre-stress can physically exist and be found by the SCALP 5 by repeating the measurement without moving the SCALP 5 device.

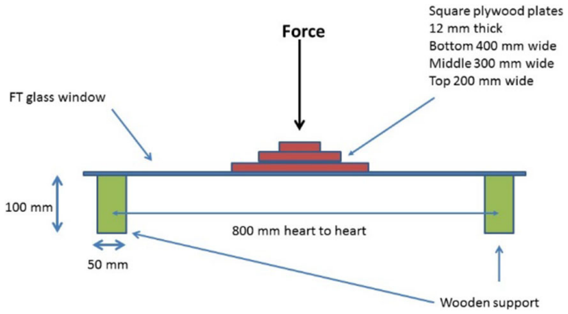
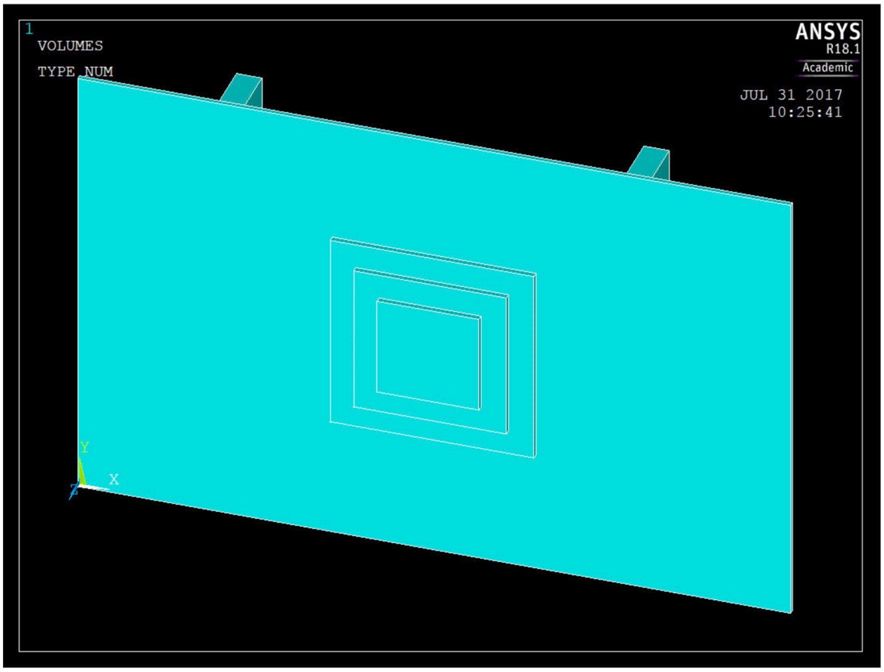


Fig. 4 Schematic illustration of the test setup used and ANSYS model

The compressive surface pre-stress was found to vary considerably across the width of the window forming bands similar to the bands of shards visible in the spontaneously failed specimens. As the variation is far greater than the 5% measuring error, the differences must be assumed to represent physical differences in the compressive surface pre-stress.

Figure 3 shows a typical variation in compressive pre-stress as measured with the SCALP 5 device. It should be noted that at the centre of the specimen, where the highest bending stresses occur, the compressive surface pre-stress is significantly lower. Depending

Fig. 5 Ansys model



on the specimen, local values for the compressive surface pre-stress as low as 20 MPa were measured. This is in sharp contrast with the values of 100 MPa measured in the properly thermally toughened zones.

5 Testing the windows

Three non-broken windows, were supplied to determine the failure stress. The windows were 1950 mm long, 1220 mm wide and 8 mm thick. As the pre-stress is highly variable, as indicated by the SCALP 5 measurements, an area with bands of high and low pre-stress should be tested to determine the weakest point. Normally this would be done with four point bending or ring on ring tests. Due to the size of the windows the tests could not be done with available 4 p bending or ring on ring set-ups. There was no time or budget to manufacture a ring on ring test rig in this size. A special setup, illustrated in Fig. 4 was designed and verified using a FEM calculation. Carved pine wooden supports were made to support the glass. Using a series of stepped 12 mm thick plywood plates the point load was introduced gradually into the whole plate to create a surface load. The force was introduced using a steel head pressing on a steel plate covering the top wooden plate. Displacement of the hydraulic cylinder and force were measured during the test.

Table 2 ANSYS input properties

Material	E longitudinal (GPa)	E transverse (GPa)	Poisson ratio
Glass	70	70	0.2
Pine	10	1	0.35
Plywood	8	8	0.3

A linear elastic finite element model was made using the ANSYS 18.1 mechanical APDL academic research finite element software. This was done using type 285 solid elements, to calculate the stresses under the stepped plate. The type 285 element is a four noded tetrahedral element, selected because it can mesh complex shapes easily. As the calculation is linear elastic and a fine mesh was used, it was not felt necessary to use an element with mid side nodes. The model is illustrated in Fig. 5. It is composed of volumes, which are glued together in the ANSYS pre-processor, each volume with the material properties specific for the material used. The various input properties are given in Table 2. The values for these properties were found in the CES 2016 database.

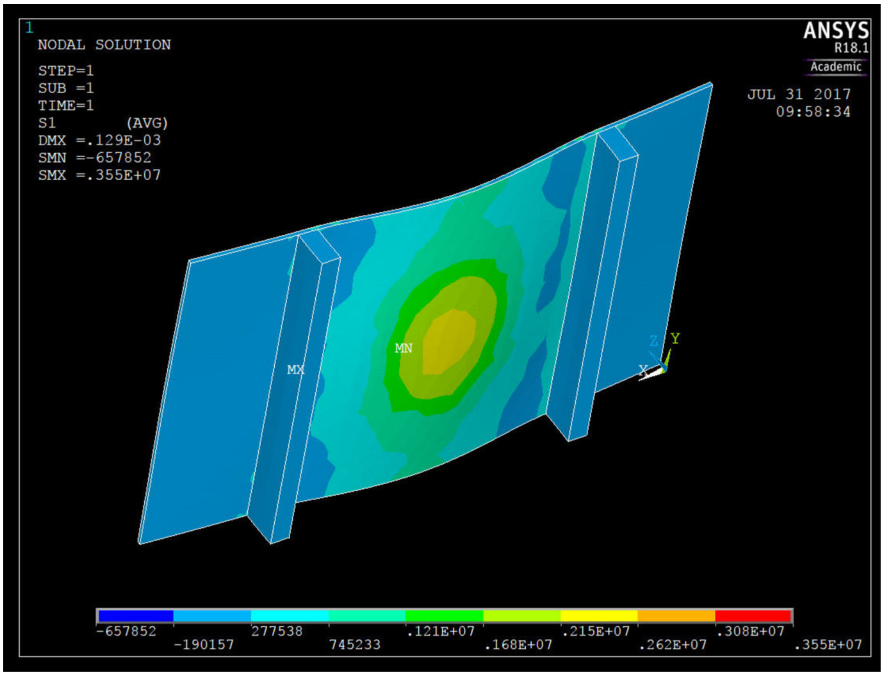


Fig. 6 Stress distribution according to ANSYS model

The lines on the long sides of the glass plates were specified as having 400 elements. The lines in the thickness of the glass and three plywood plates were specified as having 8 elements, ensuring there are at least 8 elements in the thickness of each plate. The glass plate was meshed first, the wooden supports next, followed by the plywood plate in contact with the glass, the middle plywood plate and the upper plywood plate. This resulted in a fine mesh with in total some 22.000 elements. No meshing errors, deformed element or other warnings were reported by the ANSYS program before starting the solver or after solving the model. Mesh refinement was thus not considered necessary. A homogeneous pressure was applied to the upper plywood plate corresponding to the applied load at failure. This simulates the head of the hydraulic cylinder and intermediate steel plate in the test setup. The failure stresses were determined by finding the maximum principal stress in the bottom of the glass plate. Figure 6 shows the calculated stress distribution. Effectively the stress distribution is comparable to that in a ring on ring test which implies the test should find the weakest point in the area of maximum stress.

The loading rate was 250 N/min. Table 3 gives the calculated failure stresses. After failure, all specimens showed the same pattern of bands of small and large shards as observed in the spontaneously failed windows and illustrated in Fig. 7. All failures originated in the area with the highest applied stress shown in Fig. 6. The bands of large and small shards visible in Fig. 7 corresponded to the bands of lower and higher compressive surface pre-stress measured with the SCALP 5 device.

Table 3 Failure stresses calculated using the linear elastic ANSYS model of the experiment

Window number	Failure stress (MPa)
1	72
2	65
3	56

6 Fractographic analysis of the butterfly fragments

The butterfly specimens of the two available spontaneously failed windows were carefully removed.

On one set no clear fractographic information was visible with a microscope. However, if the two pieces were put together it was clear that a small piece was missing. Presumably the piece of glass where the fracture originated was separated from the butterfly pieces and not recovered.

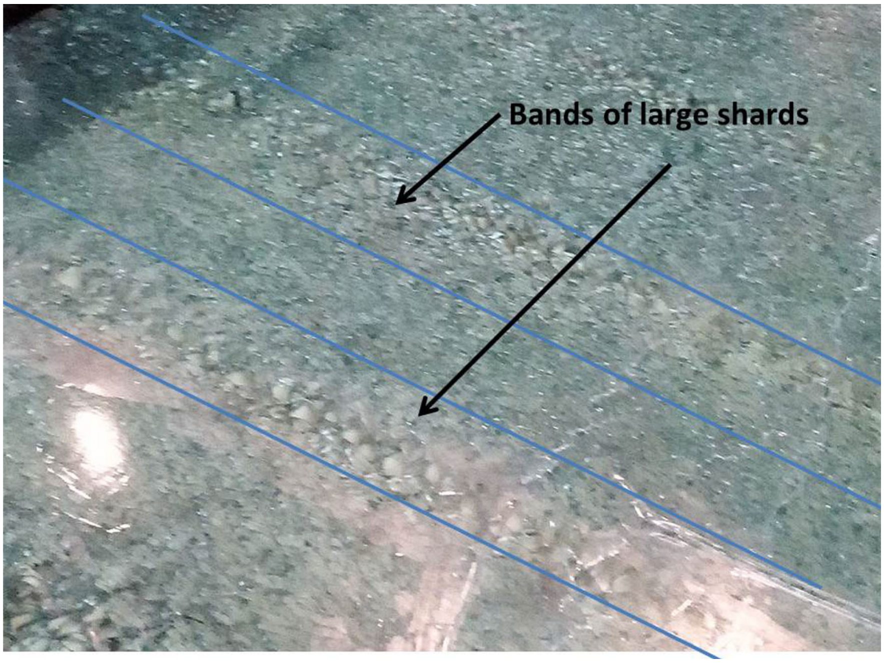


Fig. 7 Specimen after failure, drawn bands are approximately 40 mm wide

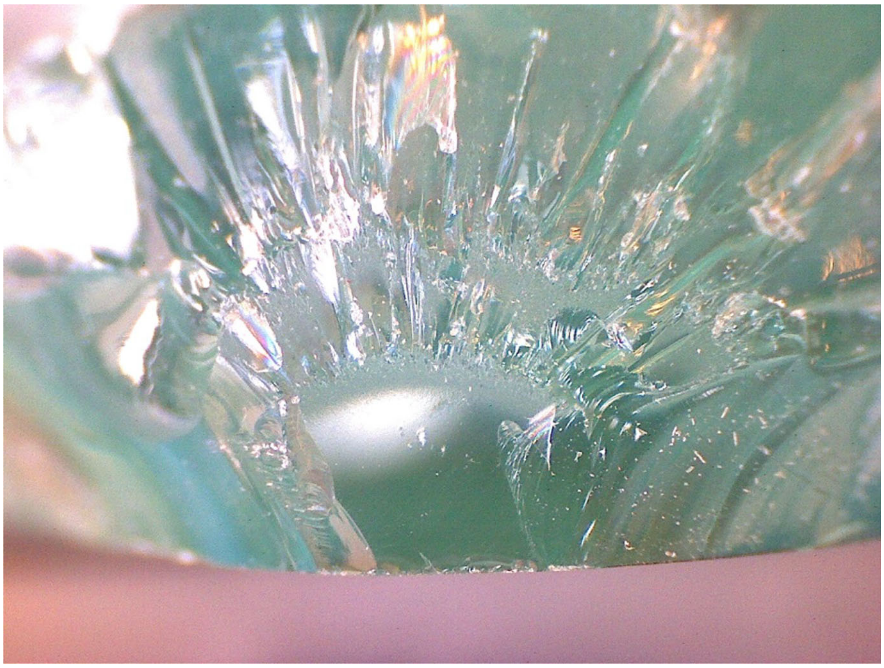


Fig. 8 Fractography of butterfly piece, typical mist, mirror, hackle zones

The second set of butterfly pieces showed a clear fractographic pattern under a microscope. This is shown in Fig. 8. The source of failure is clearly at the surface of the specimen and not in the interior, where a NiS failure would originate. The failure pattern is similar to that for an overload as given by Ono (2004), showing the typical mist, mirror and hackle pattern. This pattern alone makes it clear that no inclusion caused the original "spontaneous" failure.

7 Discussion

The two failures that were investigated were spontaneous in that they occurred during normal usage and unexpectedly to the vehicle users. The two failures are not spontaneous in that there is no detectable change or deterioration in the material which causes sudden failure.

The patterns of bands of large and small shards in both the failed windows and the windows tested in bending shows that both had uneven tempering. The SCALP 5 measurements on the windows that were later tested in bending shows the presence of longitudinal bands with decreased compressive surface prestress. As the windows were slightly curved, the bands are probably a shadow of the supports on which the hot glass rested during bending and which masked the glass from the cold air during tempering. These supports were visible on photographs of the manufacturing process shown by the manufacturer to the author.

The characteristic strength for thermally toughened, heat strengthened and annealed glass is given as 120, 70 and 45 MPa in the Guidance for European Structural Design of Glass Components.

The actual values found by testing for the bending tests of the windows in this investigation are thus reasonable or below the values for heat strengthened glass range and thus well below the strength expected for thermally toughened glass.

Presumably the windows, which are sliding vehicle roof windows, were designed with the assumption that the glass was homogeneously thermally toughened with corresponding strength. If the actual strength is half or less of the design strength it is not surprising that during normal usage an accidental overload can occur unexpectedly.

Although this is a spontaneous failure from the point of view of the end user, it is actually a quality control problem in the glass processing.

8 Conclusions

From the results, the authors conclude that:

> The spontaneous failures investigated are not due to NiS or other inclusions.
> Uneven tempering caused local zones with low compressive pre-stress.
> The result was a significant decrease in bending strength as demonstrated by the results of the bending tests.
> Fractographic analysis shows that the failures were the result of overloading the glass.
> As the strength was much lower than the strength expected by the designers during normal usage an overload situation could unexpectedly occur.
> The butterfly pattern in this case was not the result of NiS inclusions.

Compliance with ethical standards

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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