

FAILURE OF TOUGHENED GLASS

The processing of thermally toughened glass, in accordance with EN 12150-2:2004 [1] or EN 14179-2:2005 [2], results in a layer of surface compressive stress being generated, balanced by a core of tensile stress. The compressive layer will extend approximately 20% into the glass thickness (t) on either side, with the central 60% of the glass thickness being tensile stress. The stress profile will typically follow a parabolic profile, as illustrated below:

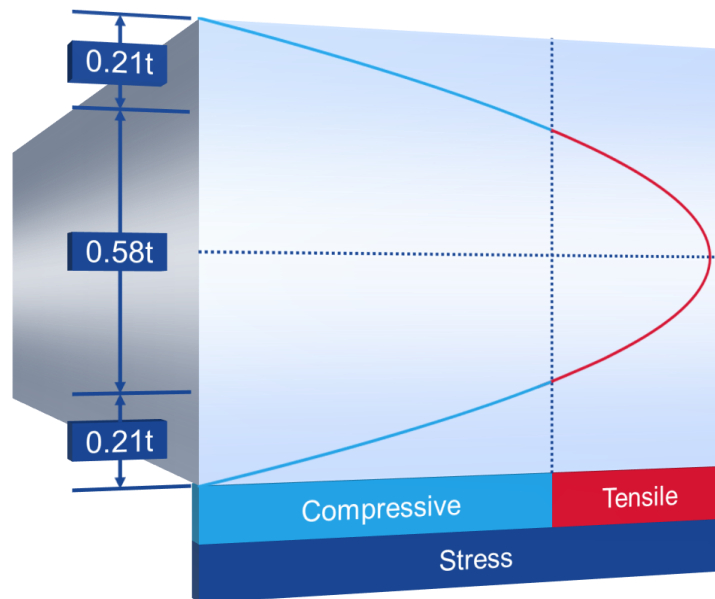


Figure 1 - Illustration of stress distribution within toughened flat glass

If the tensile stress is brought to the surface, either by penetration of the compressive stress layer or through a change in the stress distribution through bending of the glass, failure will result in fragmentation as fractures will rapidly propagate through the tensile region.

The failure of thermally toughened can occur for several reasons, including;

- Overstressing at the glass surface, due to excessive bending,
- Surface damage resulting from deliberate or accidental hard body impact(s),
- Edge damage, potentially from storage, handling or installation,
- Issues with the toughening process can also lead to failure of the glass, specifically inadequate toughening or the generation of "hot-spots", which can result in tensile stresses near the surface,
- Nickel Sulphide (NiS) inclusions, which have the potential to cause apparent spontaneous failure of the glass.

DETERMINING THE CAUSE OF FAILURE

Regardless of the mode of breakage, a toughened glass pane will typically have two “butterfly wing” or “double D” shape fragments at the failure origin.

When toughened glass within a fully edge supported frame does fracture, there is a chance that the glass being retained in the framing system. If this is the case, the fracture can typically be traced back to an origin by following the “spider web” pattern back to the epicentre. This then allows the origin to be removed, retained and analysed in order to determine the cause of failure.

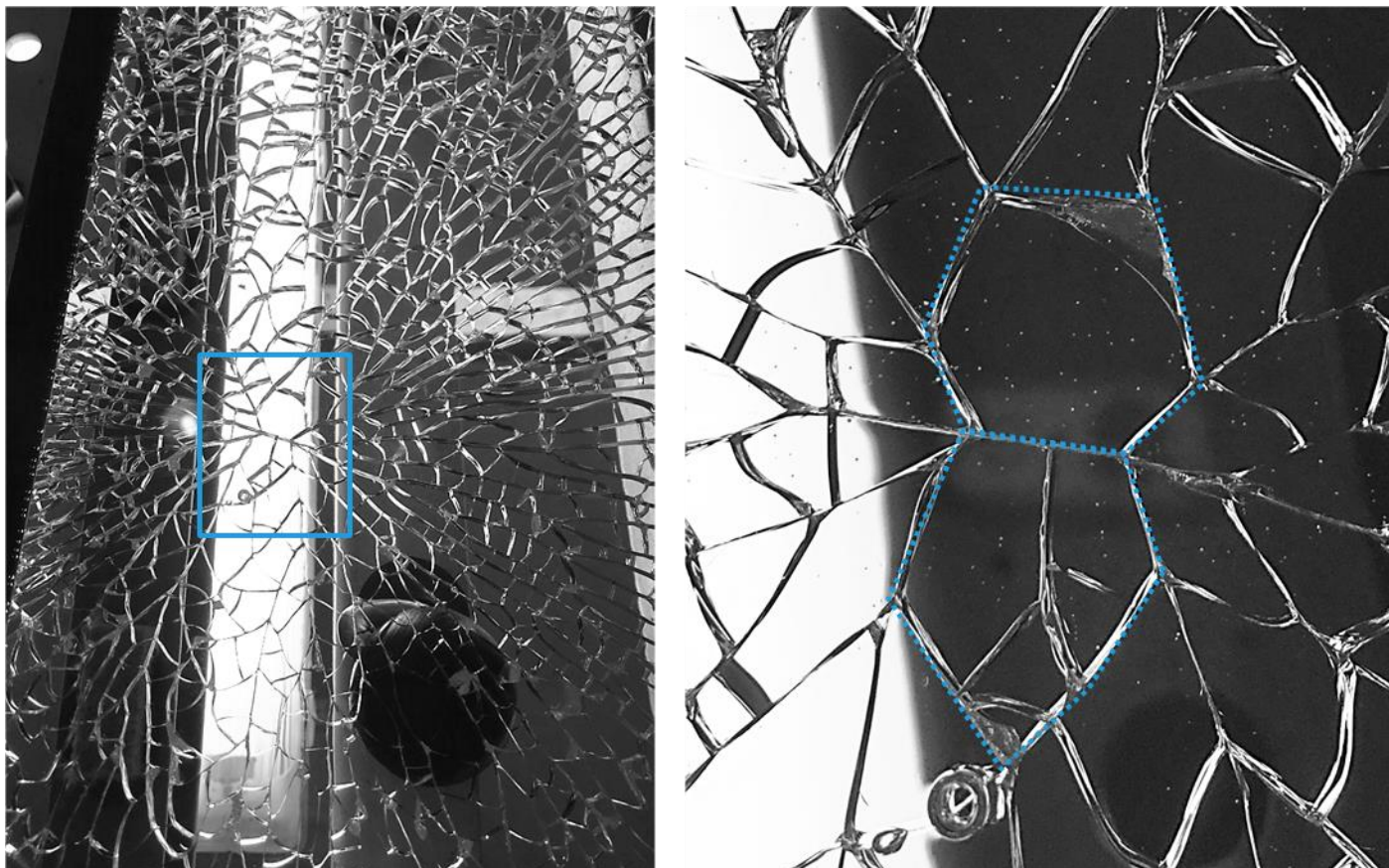


Figure 2 - Fractured toughened glass pane (Left), with origin region highlighted (Right)

If the resulting fragmentation results in the glass falling, the origin fragments may be difficult to locate, but can often be identified within the remains by their typical shape.

The origin fragments must be identified and analysed in order to determine the cause of failure of a toughened glass pane.

FRACTURE RESULTING FROM MECHANICAL LOADS

Glass failure will occur when levels of tensile stress at the surface exceed the strength of the glass. Although toughened float glass is typically considered to be approximately 4 to 5 times stronger than annealed float glass, failure due to stresses generated due to deflection can still occur when a load of a sufficient magnitude is applied.

Failure through externally applied loadings would be more likely if the toughening process has resulted in limited levels of surface compressive stress or “hot spots”.

FRACTURE RESULTING FROM DAMAGE

With toughened glass having a core with significant levels of tensile stress, any damage to the outer compressive layer will effectively bring this core closer to the surface. If damage is sufficient that it penetrates completely through the compressive layer, failure would be expected to occur instantaneously. If damage is limited, there is still the potential for failure of the glass to occur if loading “opens up” any surface damage.

Glass edges are generally considered a weak point for thermally toughened float glass and any damaged to the edges, whether during handling or installation, or due to fixtures within the frame impacting the glass edge, can result in failure of the glass. For this reason, it is always recommended to avoid contact between metal fixings and glass.

FRACTURE RESULTING FROM STRESS IMBALANCES IN THE GLASS

Ineffective tempering, typically resulting from issues in the quench phase of the toughening process can lead to either a reduced level of surface compressive stress from a lack in quench pressure, or “hot spots” resulting from blocked quench nozzles.

At worst, areas of tensile stresses can be generated at, or near to, the surface of the glass. This acts as a weak point where and damage or localised stresses applied can resulting in apparent spontaneous fracture.

FRACTURE RESULTING FROM NICKEL SULPHIDE (NiS)

NiS as a cause of failure was highlighted by E.R. Ballantyne in the early 1960s [3], after reporting on the failure of a number of toughened panes on ICI House in Melbourne, Australia. However, it has been reported that the industry was, to an extent, aware of the issue as far back as the 1940s [4].

NiS inclusions are inherent in float glass as a result of a reaction between sulphur, present in the fuel for the burners that melts the glass, and nickel contaminants present in the batch materials. In annealed float glass, these inclusions don't present a problem due to their small size, typically in the region of between 50-600 µm [5, 6]. Due to the size of the inclusions, they are not detectable by the standard defect detection devices typically present on float lines.

However, when glass is thermally toughened, phase changes of the NiS upon heating and cooling of the glass can lead to subsequent failure as phase changes of the NiS continue.

PHASE TRANSITION OF NICKEL SULPHIDE

With NiS inclusions within glass, there are two temperature dependent crystalline phases to consider, the alpha high temperature stable phase (hexagonal) and the beta (rhombohedral) low temperature stable phase. The beta phase, is in the region of 2-4% greater in volume than the alpha phase [6, 7].

The temperature and rate at which the NiS changes between alpha and beta phases will be dependent on the stoichiometry (Ni to S ratio) and the presence of any impurities, such as iron (Fe) [8, 9, 10, 11]. Typically, the phase transition will occur in the region of 380°C [4, 6]. During the production of float glass, the low cooling rate within the annealing lehr allows the inclusion time to change phase, and so increase in size whilst the glass is still relatively soft, and so without imparting additional stress into the glass.

During thermal toughening, the rapid cooling of the glass prevents the inclusion from having time to complete the phase transition before the glass is fully cooled, in effect trapping the inclusion in an unstable phase within the glass.

Over time, the inclusion will continue to phase change, which will result in the generation of micro-fractures around the inclusion within the glass. If the inclusion is present within the tensile region of the toughened glass, the micro-fractures can propagate as a result of stress at the crack tips, and ultimately result in fracture, and fragmentation, of the glass.

GLASS STRESS AND INCLUSION SIZE

It is known that the critical diameter of a NiS inclusion, the size at which it will cause failure, is dependent on its size, location within the glass thickness and the level of stress within the glass [5, 12, 6]. From the equation shown below [5], it can be seen that the critical diameter (D_c) is inversely proportional to the level of tensile stress (σ_0) at the inclusion location. As such, higher levels of tensile stress require a smaller inclusion to result in fracture.

$$D_c = \frac{\pi K_{IC}^2}{3.55 \sqrt{P_0} \sigma_0^{1.5}}$$

Where; K_{IC} = Stress Intensity Factor, 0.76 MPa.m^{0.5}

P_0 = Hydrostatic Pressure, 615 MPa

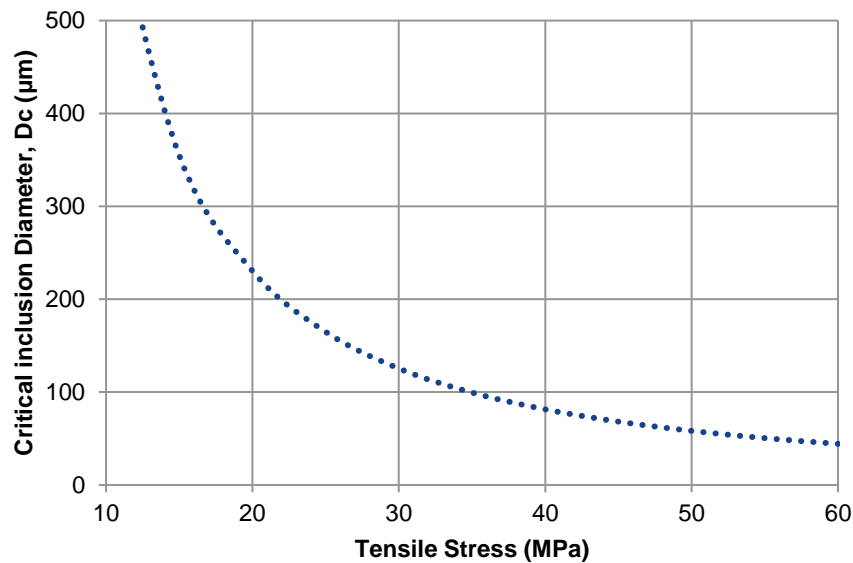


Figure 3 - Plot of predicted critical inclusion diameter vs. tensile stress at the inclusion location

The actual level of stress at the inclusion location will be dependent on the level of surface compressive stress, and depth into the glass, with the maximal tensile stress at the centre. This can be calculated using the following equation [12];

$$\sigma_0 = \sigma_c \left[1 - 3\left(\frac{y}{h}\right)^2 \right]$$

Where; y = distance from centre line

h = glass half-thickness

In order to cause failure, a NiS inclusion must be present within the tensile region of the glass, so approximately the central 60% of the glass thickness.

NICKEL SULPHIDE INCLUSIONS IN FLOAT GLASS

There is still much debate and disagreement as to the potential level of NiS inclusions in float glass, and as such, the likelihood of failure due to the presence of a NiS inclusion. Neither EN 12150-1:2015 or EN 12150-2:2004 [13, 1] provide any indication of the likelihood of failure rates due to NiS in non-heat soak tested thermally toughened glass.

Commonly stated values have been known to be in the region of 1 inclusion per 4 to 8 tonnes of glass [14, 4], and even as low as 1 inclusion per 38.5 tonnes of glass [14]. The following data [15] shows data from more than 25,000 tons from different production and tempering sites, and so provides an average, as well as an indication of the variability in potential rates of occurrence of NiS inclusions.

	Tons	Breaks	Tons per Break
A	13569	874	15.5
B	20262	3142	6.4
C	668	108	6.2
D	241	46	5.2
E	539	14	38.5
Total	35279	4184	8.4

Additional data [16], shown below, gives a higher occurrence, with an average of 1 failure per 6.5 tons.

	Tons	Breaks	Tons per Break
A	2987	292	10.2
B93	1725	372	4.6
B95	2043	300	6.8
B96	2080	275	7.6
B97	2419	383	6.3
B98	2484	344	7.2
B99	2891	588	4.9
C1	118	19	6.2
C2	693	103	6.7
Total	17440	2676	6.5

Due to the lack of available technology to readily detect NiS inclusions within float glass, there is very little data available that can be used to determine the actual occurrence rates. With consideration to speculation that NiS may occur in outbreaks, any occurrence rates stated should be considered with this in mind. For reference, the paper from which the above data is sourced suggests a conservative estimate of 1 break per 6 tons of glass.

INFLUENCING FACTORS

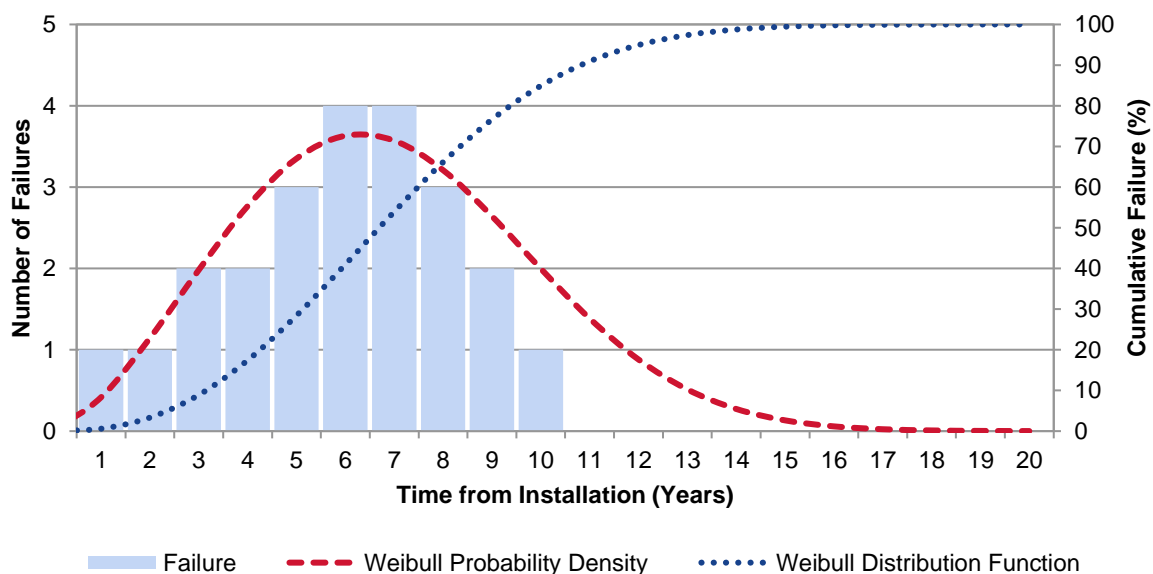
As well as the properties of the inclusion (stoichiometry, location and size) and the glass (tensile stress levels), other factors may influence the breakage rate, and the time to failure, for NiS inclusions.

The phase transition between alpha and beta is, to some extent, influenced by temperature, which is why the heat soak process will cause thermally toughened panes containing NiS to fail. Glasses with high levels of solar absorbance (body tinted or coated), and panes in spandrel or shadow box installations, will heat up to a greater extent when exposed to incident solar radiation. As such, the growth of the inclusion may occur at an increased rate, leading to failure at an earlier time.

FAILURE TIMELINE

If a critical NiS inclusion is present within thermally toughened float glass, then typically, failure will rarely occur within the first few months of the glass being processed. Previous commentary has stated that failures typically begin approximately 2 to 3 years after processing, peak at 2 to 7 years, and then rates reduce. Other sources suggest peak failures in the first 1 to 3 years [4, 17]. It has also been reported that failures from NiS have occurred up to 30 years after processing [17]. There will be influence, as above, from the service temperatures of the glass, as well as potentially the loadings the service glass is under from climatic conditions and occupancy.

Some predicative techniques have been proposed, based upon a Weibull distribution [4, 14], which would potentially permit the remaining number of failures to be predicted on a building once a number of failures have already occurred. The below example is for illustrative purposes only, and shows recorded failures recorded up to 10 years from installation. An appropriate Weibull distribution could be used to predict a further 2 to 3 failures on the building



HEAT SOAK TESTED THERMALLY TOUGHENED FLOAT GLASS

The potential for failure due to NiS inclusions can be reduced by carrying out the heat soaking process, in accordance with EN 14179-1:2016 and EN 14179-2:2005 [18, 2]. The heat soak process is carried out within a calibrated oven, with the glass being heated, at a controlled rate, to a temperature of $260 \pm 10^\circ\text{C}$ for a minimum of 2 hours, with subsequent controlled cooling to 70°C . The process will cause any NiS inclusions present within the glass to undergo an accelerated phase change, and associated volumetric increase. If the inclusion is of a critical diameter or above, failure of the glass would be expected to occur. As such, this testing is a destructive method.

FAILURE DURING THE HEAT SOAK TEST

Available data [16] shows the time to failure of glass within a heat soak test, for ovens of the same design. At time 0 hours, when the HST oven has reached the hold temperature, based on the available data, 79% of the total number of failures have already occurred. After a hold time of 2 hours, 99% of the total number of glass failures has occurred within the HST.

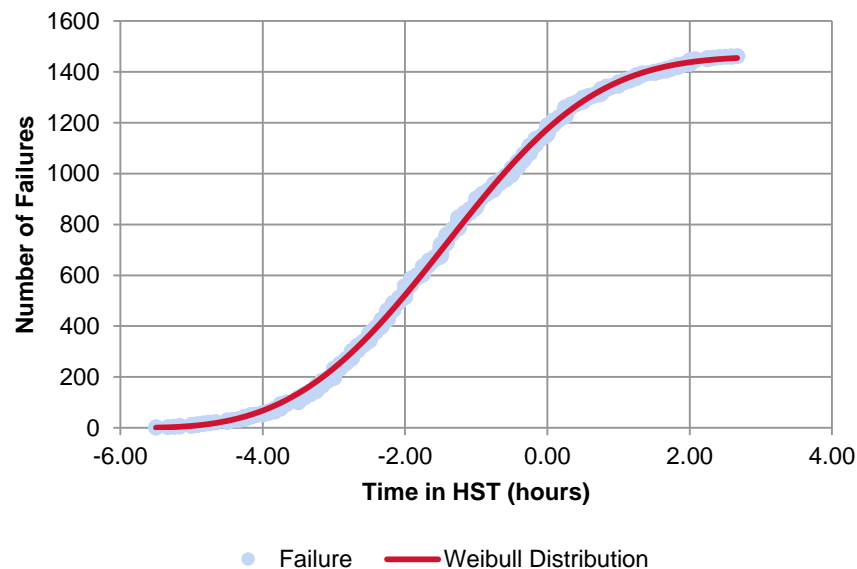


Figure 4 - Profile of glass failure during HST

PREDICTING FAILURE RATES FOR GLAZING

A Weibull distribution can be fitted, and this can be used, as per an associated article [15], to predict the probability of failure for glass that has been heat soak tested, based on glazing unit area, glass thickness within the unit and the total area of glass. Based on the Weibull distribution of the heat soak, the probability of failure during one year, for a single pane of glass can be determined, as follows;

$$P_1 = \frac{m_1 \cdot P_{HST} \cdot (1 - WEIBULL(t))}{L}$$

Where;

m_1 = distance from centre line

P_{HST} = Probability of failure on the heat soak test (1 per 6 tons)

L = Estimated lifespan of the glass (50 years)

If the probability of failure over the whole life of the glass is to be considered, then the lifespan can be removed as a function of the probability;

$$P_1 = m_1 \cdot P_{HST} \cdot (1 - WEIBULL(t))$$

Based on the probability of failure for a single pane, the failure probability for a whole façade can be determined as follows;

$$P_n = 1 - (1 - P_1)^{A_T/A_1}$$

Where; A_T = total glazing area
 A_1 = Area of pane

WORKED EXAMPLE FOR FAILURE RATES

To illustrate the above, as an example, we can consider 5000 glazing units, 2 m² in area, and comprising of two panes of 8 mm thick heat soak tested thermally toughened glass (2 hour hold time). This will give 10,000 panes of glass weight 40 kg, or 0.004 tons, and a probability of failure of each pane in the heat soak of 0.67%, based on 1 break per 6 tons.

Based on the previous equations, the probability of failure of a single pane (P_1) over its entire life is estimated to be;

$$P_1 = 0.004 \cdot \frac{1}{6} \cdot (1 - 0.9837) = 0.0108\%$$

For 10,000 panes of glass, this will be 400 tons of glass, and would result in a probability of a failure occurring on the building over its entire life of;

$$P_n = 1 - (1 - 0.000108)^{5000/2} \approx 42\%$$

This can then be extrapolated, as below, to consider the probability of multiple failures on the same building, based on the probability of a single failure.

Number of Failures	Probability of Occurrence (%)
0	58.14
1	41.86
2	17.52
3	7.33
4	3.07
5	1.28
6	5.38×10^{-1}
7	2.25×10^{-1}
8	9.42×10^{-2}
9	3.94×10^{-2}
10	1.65×10^{-2}
15	2.12×10^{-4}
20	2.72×10^{-6}

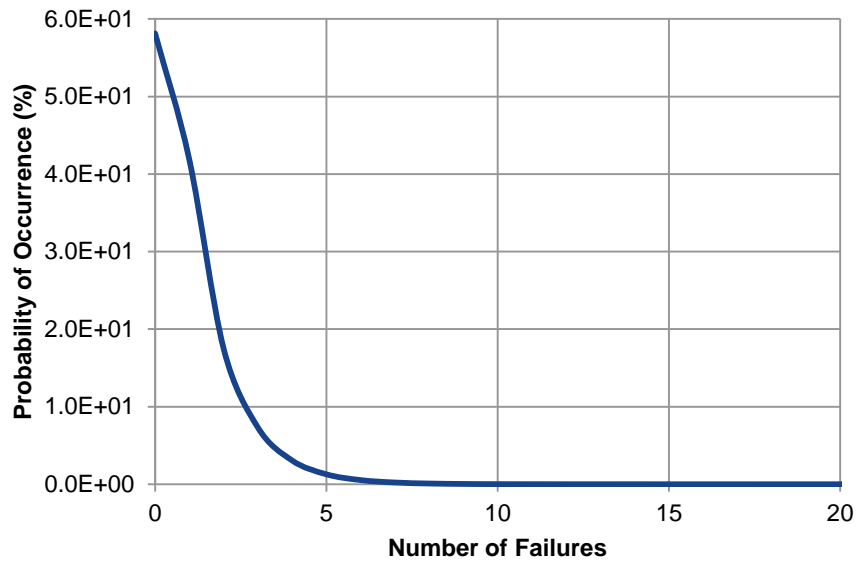


Figure 5 - Worked example, probability of multiple failures

GENERALISED ASSESSMENT OF RISK OF FAILURE

Whilst the heat soak test does not guarantee that no subsequent breakages due to NiS will occur, the standard does state that the “risk of spontaneous breakage of heat soaked thermally toughened soda lime silicate safety glass, on a statistical basis, due to the presence of critical nickel sulphide inclusions, is no more than one breakage per 400 tonnes of heat soaked thermally toughened soda lime silicate safety glass.”

If an assessment is required with varying glass configurations and sizes, a more generalised estimation of the risk of failure can be carried out, based on a Poisson distribution, and with consideration to the failure rate of no more than 1 per 400 tons of glass. Whilst Poisson distributions are common for determining failure rate probabilities, the use of this method by the Claimant’s expert witness, in the case of 125 Old Broad Street [19], to some extent sets a precedent for its application to risk determination for glazing with regards NiS failure.

The below is based on 100 tons of glass within a project, and a failure rate of 1 per 400 tonnes;

Number of Failures	Probability of Occurrence (%)
0	77.9
1	19.5
2	2.43
3	0.20
4	1.27×10^{-2}
5	6.34×10^{-4}
6	2.64×10^{-5}
7	9.43×10^{-7}
8	2.95×10^{-8}
9	8.19×10^{-10}
10	2.05×10^{-11}

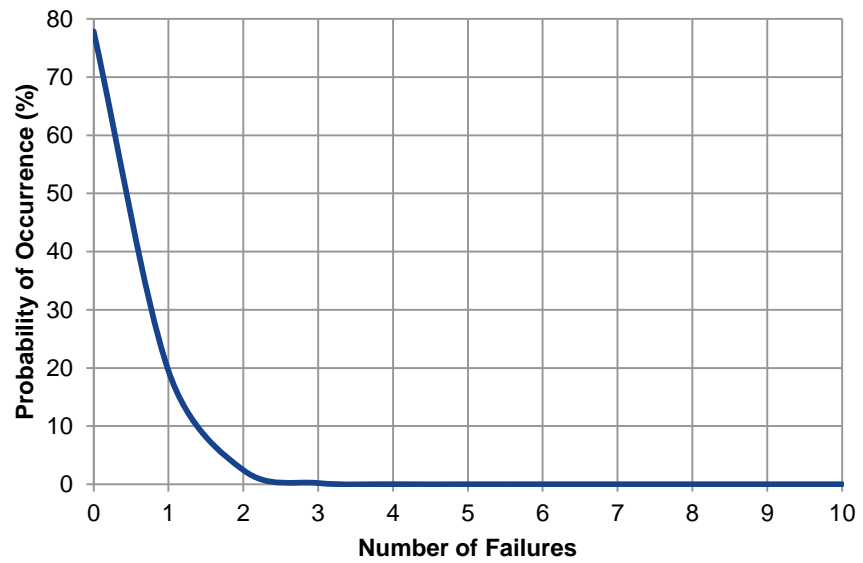


Figure 6 – Poisson distribution, probability of multiple failures

REFERENCED MATERIAL

- [1] European Committee for Standardization, *EN 12150-2:2004 - Glass in building. Thermally toughened soda lime silicate safety glass. Evaluation of conformity/Product standard*, CEN, 2004.
- [2] European Committee for Standardization, *EN 14179-2:2005 - Glass in building. Heat-soaked thermally-toughened soda lime silicate safety glass. Evaluation of conformity/product standard*, CEN, 2005.
- [3] E. Ballantyne, "Fracture of Toughened Glass Wall Cladding," CSIRO, Division of Building Research, 1961.
- [4] L. Jacob, "A Review of the Nickel Sulphide Induced Fracture in Tempered Glass," *Glass Processing Days*, pp. 108-110, 2001.
- [5] A. Kasper, "Spontaneous Glass Breakage Caused by Nickel Sulphide (a Review)," *Glass Performance Days*, pp. 600-602, 2009.
- [6] L. Jacob, "Factors that Influence Spontaneous Failure in Thermally Treated Glass - Nickel Sulphide," *Glass Processing Days*, pp. 323-327, 1997.
- [7] C. S. e. al., "Phase-controlled synthesis of α -NiS nanoparticles confined in carbon nanorods for High Performance Supercapacitors," *Scientific Reports*, vol. 4, 2014.
- [8] D. Bishop, P. Thomas and A. Ray, " $\alpha - \beta$ Phase Transformation Kinetics in Nickel Sulphide," *Journal of Thermal Analysis and Calorimetry*, vol. 56, pp. 429-435, 1999.
- [9] C. Sakai and M. Kikuta, "Adapted Heat Treatment for Phase Transformation of NiS Inclusion in the Heat Strengthened and Tempered Glass," *Glass Processing Days*, 1999.
- [10] M. Swain, "Nickel sulphide inclusions in glass: an example of microcracking induced by a volumetric expanding phase change," *Journal of Materials Science*, vol. 16, no. 1, pp. 151-158, 1981.
- [11] J. Barry and S. Ford, "An electron microscopic study of nickel sulfide inclusions in toughened glass," *Journal of Materials Science*, vol. 36, no. 15, pp. 3721-3730, 2001.
- [12] D. Gelder, "The Significance of Sub-critical NiS Inclusions," *Glass Processing Days*, 2001.
- [13] European Committee for Standardization, *EN 12150-1:2015 - Glass in building. Thermally toughened soda lime silicate safety glass. Definition and description*, CEN, 2015.
- [14] L. Jacob and I. Calderone, "Nickel Sulphide Inclusions - Important Issues for the Designer," *Glass Processing Days*, pp. 228-231, 2003.
- [15] A. M. Kasper, "Heat Soaking Avoids Spontaneous Cracking of Thermally Toughened Safety Glass," in *Glasstech, Asia*, 2002.
- [16] A. Kasper, "Nickel sulphide: supplementary statistical data of the heat soak test," *Glasstech. Ber. Glass Sci. Technol.*, vol. 73, no. 11, pp. 356-359, 2000.
- [17] J. Bowler-Reed, "The disintegration of thermally toughened glass by nickel sulphide inclusions," *Glass & Glazing Products*, vol. March, pp. 67-70, 2002.
- [18] European Committee for Standardization, *EN 14179-1:2016 - Glass in building. Heat-soaked thermally-toughened soda lime silicate safety glass. Definition and description*, CEN, 2016.
- [19] *Technology & Construction Court - Case Number HT2015.00104 - 125 OBS vs. Lend Lease Construction (Europe) Limited*, 2017.