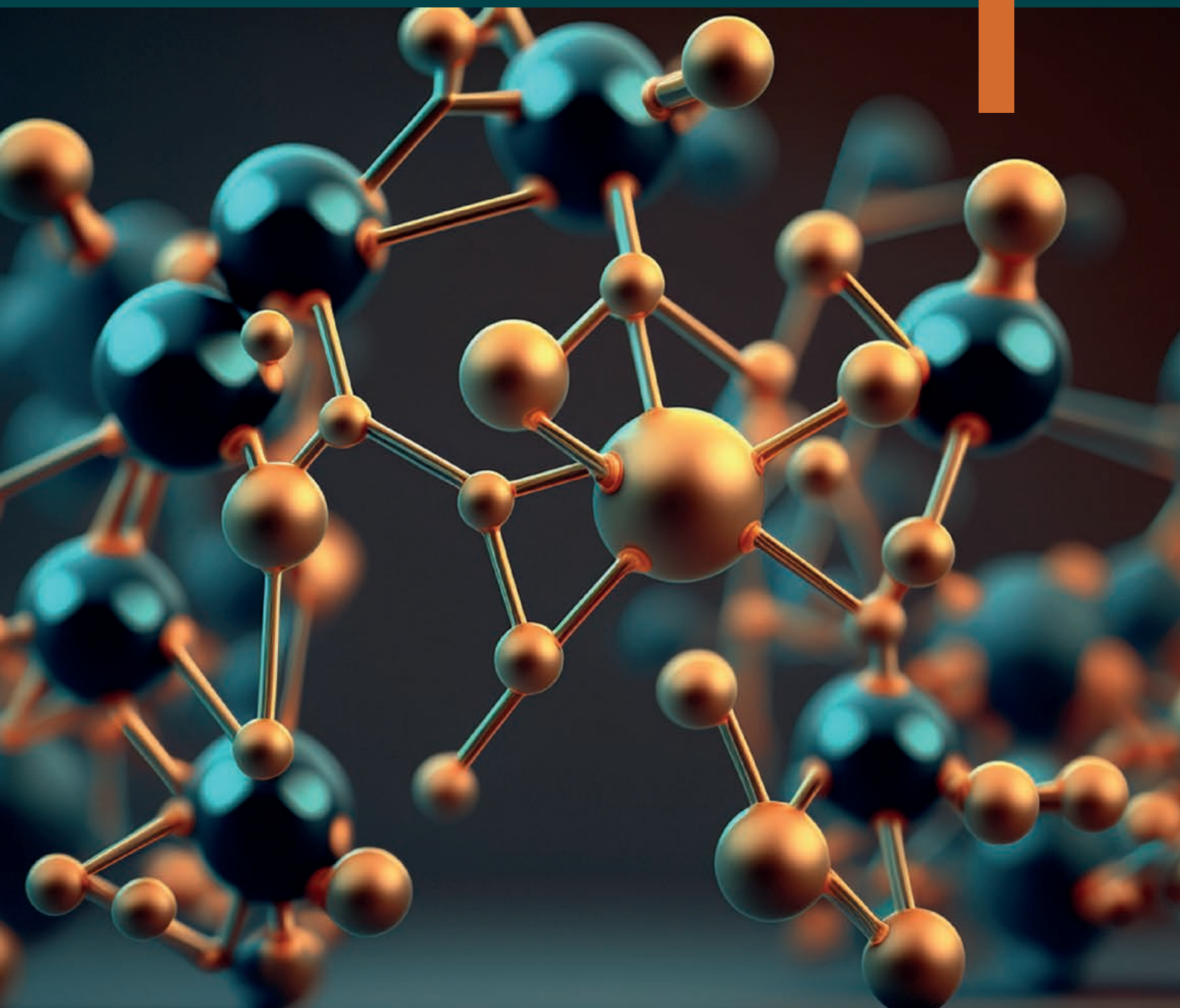


A technical paper

NiS in HS glass



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Breakage probability of nickel sulphide inclusions in heat strengthened glass

Dr. Andreas Kasper⁽¹⁾ – John Colvin⁽²⁾ – Frank Rubbert⁽³⁾ - Francis Serruys⁽⁴⁾

Abstract

Spontaneous breakage of toughened glass in facades is under control today when the heat soak process according to EN 14179-1 (2005/2016) is rigorously carried out. However, nickel sulphide (NiS) breakages are not only observed in toughened glass but also in heat strengthened glass.

Heat strengthened glass has a lower surface compression and thus a lower central tensile stress when compared to toughened glass. Consequently, bigger nickel sulphide (NiS) inclusions are required to initiate a crack, as crack initiation is determined by the inclusion's size in combination with the surrounding stress field.

The critical diameter "Dc" to cause spontaneous fracture of glass is determined. A good correlation is found with the diameter of reported nickel sulphide inclusions causing breakages on a building facade. Knowing the distribution and the diameter range of nickel sulphide inclusions in float glass, a breakage probability, on a statistical basis, of 1 breakage in 1,100 ± 200 tonnes of heat strengthened glass can be estimated.

Furthermore, the timescale for breakages in service is examined. 90% of the breakages normally occur within a period of 6 to 8 years after production, even in a cold climate. Although nickel sulphide breakages have been observed after more than 20 years, this remains an exception and it may be concluded that seldom a breakage happens more than 10 years after installation.

More data of broken heat strengthened glass, due to nickel sulphide inclusions, is required to confirm the breakage probability, which in reality might be much lower. Due to this low breakage probability it is not industry practice to carry out the heat soak process. Moreover, heat strengthened glass does not satisfy code requirements for safety glazing. Therefore, heat strengthened glass is most often used in the form of laminated safety glass. If a nickel sulphide inclusion would cause a breakage of a laminated heat strengthened glass, it would stay in place due to its specific fragmentation as required by EN 1863.

Keywords: Façade - Heat-strengthened glass - Nickel sulphide - Breakage probability

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1. Introduction

In 1961¹ Ballantyne (1961) was the first to describe the mechanism whereby NiS causes toughened glass to break. It took till the 1970s² to come up with a heat treatment, later on called the heat soak test (HST), to eliminate critical NiS inclusions, trying to avoid spontaneous breakage of toughened glass. The German standard DIN 18516, derived from this test, fixed a holding time of eight hours at constant oven temperature, but it was, however, very imprecise and could easily be misinterpreted³.

Therefore, in the mid-1990s CEN⁴ started to work on a new heat soaked toughened glass product standard, the result being EN 14179-1 Glass in Building - Heat-Soaked Thermally Toughened Soda Lime Silicate Safety Glass.

Since the beginning the focus of R&D has been on understanding and reducing the residual risk of spontaneous breakages due to NiS inclusions in toughened glass. Only on rare occasions were spontaneous breakages of heat strengthened glass mentioned or dealt with. (Bordeaux and Kasper (1997))

The knowledge gathered on the properties of NiS inclusions, their distribution and dimensions in soda-lime silicate glass is used to make an estimation of the breakage probability of NiS inclusions in heat strengthened glass.

Nickel sulphide is a complex material, which undergoes a phase change (a change in crystalline structure) at a transformation temperature between 282°C and 379°C, depending on the precise combination of nickel and sulphur (called the stoichiometry) (Colvin (2013)). The mechanism which causes failure in the façade is due to the phase change being accompanied by a volume change. The α -phase, which is stable above the transformation temperature, has a smaller volume than the β -phase, which is stable below the transformation temperature.

The toughening (tempering) process in glass requires the glass to be heated to around 620 °C, which softens the glass, followed by rapid cooling. Any nickel sulphide in the glass is converted to the α -phase at the high temperature, but the rapid cooling does not allow time for the conversion back to the β -phase. The nickel sulphide is thus "frozen" into the toughened glass in an unstable form.

Over a period of time the α -phase slowly converts back to the β -phase, the conversion being accompanied by an increase in volume of 2.55 % at ambient temperature (Groenvold (1995)). If an NiS inclusion is sufficiently large and is in the central (tensile stress) zone of the toughened glass (Kasper et al. (2018)), then the expansion caused by the phase transformation can exert sufficient excess stress to cause a crack to propagate, leading to disintegration of the pane.

2.0 Properties of nickel sulphide inclusions

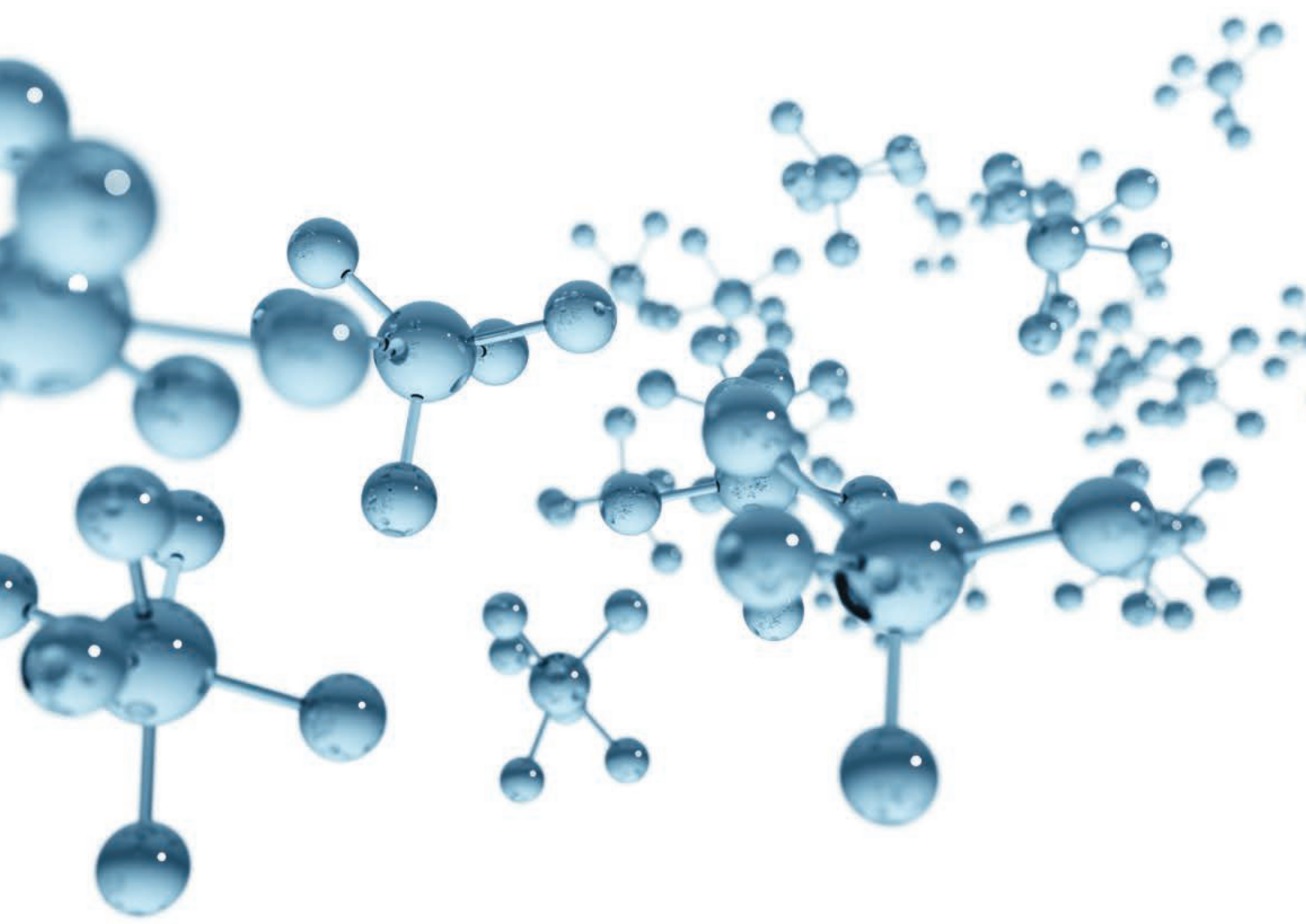
Kasper (2018 / 2020) and Kasper et al. (2018) wrote a series of four articles where the properties of nickel sulphide inclusions, their distribution in float glass, a statistical evaluation of field breakage records and consequences for residual breakage probability are dealt with in detail.

This article highlights the main points for better understanding the method used to estimate the breakage probability of NiS inclusions in heat strengthened glass.

2.1 Nickel source

NiS inclusion formation needs a particle source containing nickel metal. Nickel sources such as the nickel content of fuel oil of several ppm, or nickel containing thin coatings on glass at recycling, can factually be excluded as they provide highly dispersed ($< 1 \mu\text{m}$) oxidized NiO or NiSO₄ (from waste gas) or NiO (in coatings, but even if it is the metal, the local concentration is too small).

Consequently, only metal chip contamination contained in raw materials and cullet can reasonably be suspected to be the source of NiS inclusions. This source is, even today, not perfectly under control, and it causes a background breakage level of circa 1 in 10 tonnes of standard toughened glass in the HST (i.e. not on facades!).



2.2 Crystallization and composition of nickel-sulphide inclusions

The nickel-sulphur phase diagram is shown in figure 1. According to this diagram, every possible nickel sulphide is liquid in the glass melt, i.e. the inclusions are not solid bodies but small droplets of a liquid. Only when they enter the float bath (temperature < 1000°C), do they crystallize.

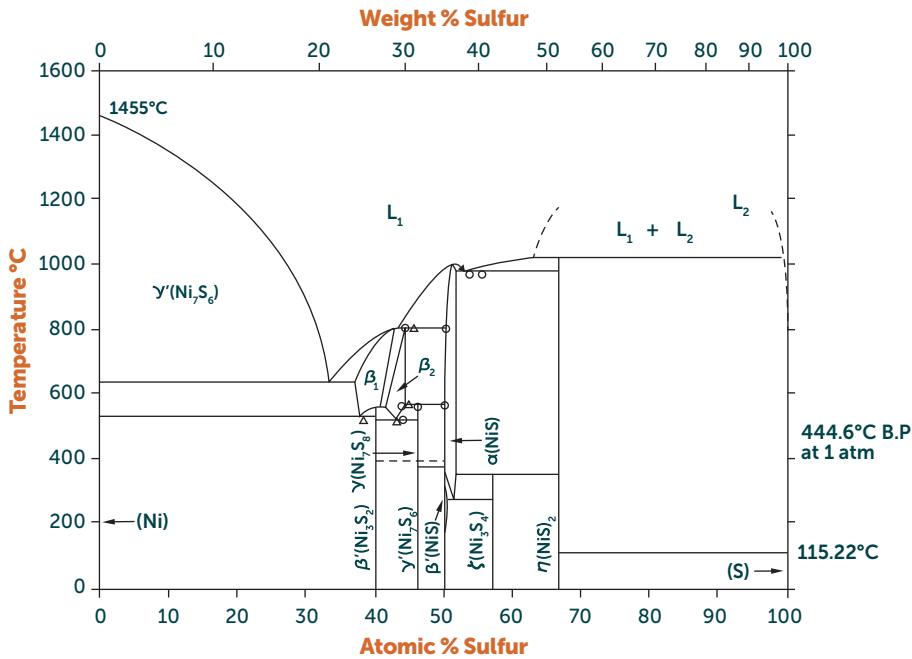


Fig. 1
Nickel - Sulphur
phase diagram

In the crystallising inclusion, crystals form according to the Ni-S phase diagram (fig. 2).

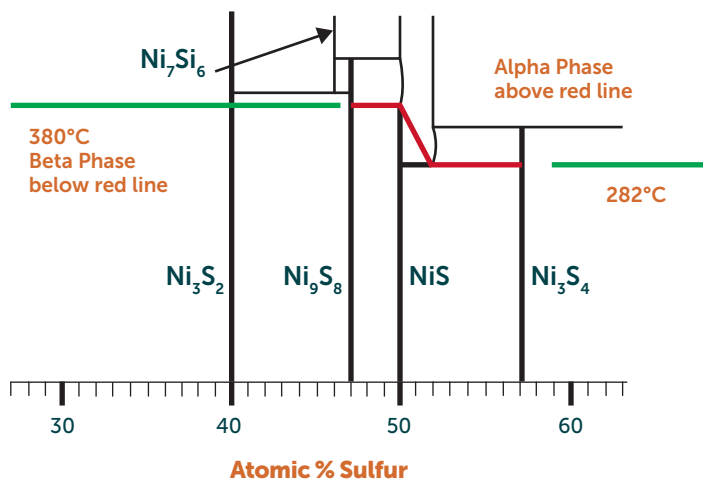


Fig. 2
The α to β phase
change line

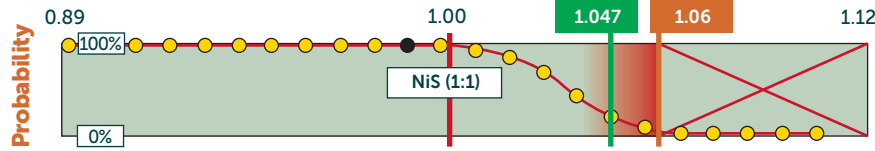
Simplifying, one can distinguish three main situations:

(a) Inclusions composed of Ni_3S_2 or $(\text{Ni}_3\text{S}_2 + \text{Ni})$: these do not cause spontaneous fracture, but also they have never been identified in float glass due to its long dwell time in the glass tank.

(b) A compositional range from (at ambient) Ni_3S_8 to NiS in random variable relation.

(c) So-called over-stoichiometric NiS $(_{1+x})$ where the inclusion contains a sulphur excess slowing down the transformation speed considerably with increasing x .

Only NiS inclusions containing (at least partly) the 1:1 composition and those with a small sulphur excess are of interest, because this chemical compound is the only one to be subject to slow α to β phase transformation at ambient temperature. According to the nickel-sulphur phase diagram, this composition can only form if $X_{\text{liquid}} < 1.06$. Then the solely interesting and solely observed phases in the crystalline high-temperature NiS inclusions are Ni_3S_8 and NiS_x where X_{solid} is in the range $1 \leq X_{\text{solid}} < 1.06$. The composition Ni_3S_4 is occasionally observed in the transformed β phase NiS inclusions as a subsequent product of slow diffusional transformation in situation (c).



Obviously the composition of a large majority of nickel-sulphide inclusions should deviate from the 1:1 composition. Distinction is made between:

(a) Under-stoichiometric inclusions

In this composition range, the crystallised nickel sulphide inclusion contains two separate phases i.e. NiS and Ni_3S_8 . Their volume proportion depends on the exact value of X_{liquid} . Below 400 °C Ni_3S_8 is not subject to a phase transformation relevant for glass breakage. Therefore, the volume fraction of NiS determines whether the inclusion is critical or not. If there is not enough NiS the α to β phase transformation does not make the volume of the entire inclusion grow enough to cause glass breakage at ambient temperature.

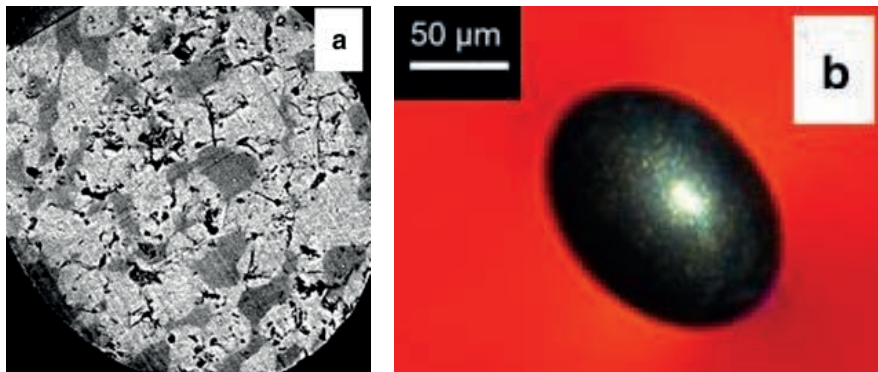


Fig. 3 Composition range of nickel sulphide inclusions eventually leading to glass breakage. Dotted (—o—) curve: estimated probability of occurrence, based on LAFFITTE's papers (limit value 1.06 for occurrence in glass), and YOUSFI's papers (limit value 1.047 for breakages)

Fig. 4 Nickel sulphide inclusion (HGI930) found in a glass pane. Size (140x190)µm. a. SEM micrograph of polished inclusion, NiS (1:1) (darker areas), c. 10% of area only. Rest: Ni_3S_8 (brighter area; more fissured). b. By light microscope, before preparation for SEM. Photographs: SG (2015)

(b) Over-stoichiometric inclusions

In the over-stoichiometric case, the crystallised high-temperature nickel sulphide inclusion only contain one phase, α -NiS_x. According to the NiS phase diagram, x_{solid} in this combination (but only in the α phase) can principally vary from 1.00 to c. 1.12. Yousfi et al. (2011) calculated a limit of $x < 1.047$, as with increasing value of x_{solid} the transformation needs more and more time. Therefore, x values above this value should be irrelevant, as phase transformation is too slow to cause breakage during the lifetime of a facade. On the other hand, NiS_x inclusions with $x > 0,06$ are definitely unstable in the glass melt (i.e., subject to spontaneous thermal decomposition) because of their exponentially increasing inner vapour pressure.

2.3 Nickel sulphide properties

	a-NiS	β -NiS	Glass	
Melting point	985		-	°C
Refining temperature	-	-	1470	°C
Softening temperature	-	-	620	°C
Sensity at ambient	5.52	5.38	2.49	g/cm ³
Thermal expansion	16.5	14.5	9	*10 ⁻⁶ /K
Thereof α to β vol. increase:	2.55%		at ambient T°	
	2.78%		at 290°C	

Table 1
Physical properties of glass and nickel sulphide inclusions

2.4 Position of nickel sulphide in the glass section

Both glass and the different nickel sulphide species have significantly different densities (2.5 g/cm³ and circa 5.4 g/cm³, respectively, at ambient temperature – Table 1).

The glass flow in the rear zone (“channel”) of every float or patterned glass furnace is almost free of turbulences and vortices because this would lead to optical glass defects (Jebsen-Marwedel and Brückner (1980)), and the dwell time of the glass in this zone of the furnace is, depending on the glass thickness and effective pull rate, in the range of several hours. Consequently, the nickel sulphide inclusions sink down due to the combination of gravitational settling and buoyancy, and the bigger ones (> 500 μ m) sink to the bottom and do not appear in produced flat glass.

Although NiS inclusions are found “everywhere” in the glass, all superficial NiS inclusions are located in the bath side, none in the atmosphere side. In short, the signature of a certain amount of gravitational settling is visible from examining the position of NiS inclusions in the glass section.

2.5 Size and size distribution

A dataset of 140 NiS inclusions identified in annealed glass were analysed (Kasper et al. (2018)). The following observations were made:

- Small inclusions (c. $< 140 \mu\text{m}$) are more frequent than big ones.
- Very small inclusions (c. $< 50 \mu\text{m}$) are rare.
- Inclusions are found "everywhere" in the glass.
- The signature of a gravitational settling is visible analysing the position of the NiS inclusions in the glass section.
- All superficial NiS inclusions are located in the bath side; none in the atmospheric side.

Figure 5 shows the histogram evaluation of the dataset using classes of $50 \mu\text{m}$. With exception of the classes of the smallest ($< 50 \mu\text{m}$)⁵ and maybe the biggest ($> 500 \mu\text{m}$)⁶ inclusions, all counted numbers per class seem to match with a decaying exponential curve.

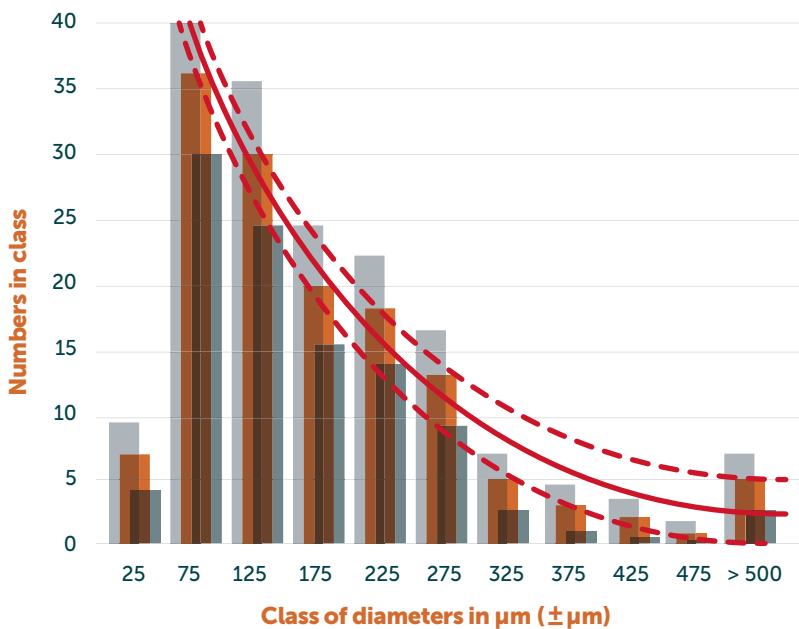


Fig. 5
Histogram of dataset "all inclusions".
Black columns: Counted numbers.
Grey columns: $\pm s$ (in numbers) calculated using the POISSON distribution. Red line: Exponential best fit curve, excluding class $< 50 \mu\text{m}$. Dotted red lines: $\pm s$ calculated from least square statistics.

Physical modelling shows that only medium size NiS inclusions arrive in the final glass whereas the smallest decompose quickly (or remain undetected)⁷, the biggest go to the bottom (gravitational settling), and a non-negligible fraction decomposes spontaneously under its own vapour pressure.

2.6 Form of nickel sulphide inclusions

Because the melting point is somewhat below 1000°C NiS inclusions do not crystallize before they enter the float bath; glass forming therein (stretching) is the reason why they sometimes look elliptical instead of spherical. This effect is more pronounced in thin glass than in thick glass since thin glass is stretched more.

Typical examples of nickel sulphide inclusions found in float glass are shown in figure 6. Some inclusions are detected in the bath side surface of the glass. This is related to gravitational settling.

Note that in every case the rough inclusion's surface structure is clearly visible besides the brass-like shine of the mineral and the exactly elliptical shape which is an important identification criterion for NiS inclusions.

Most of the inclusions show a weak brown halo that is often elongated into a brown ream (Fig. 6). This is a characteristic feature of NiS inclusions; it demonstrates that their chemical decomposition (i.e. the increase of x in NiS_x) never stops.

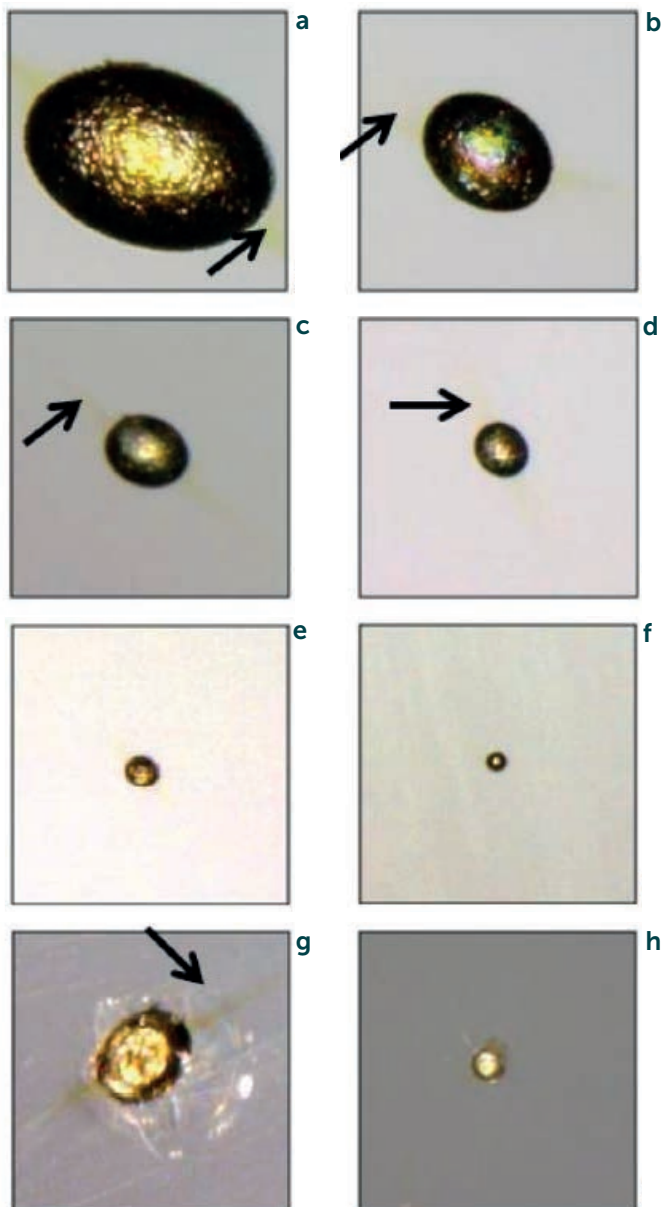


Fig. 6
Light microscopic photographs of nickel sulphide inclusions identified by SG C/K detection method. Size = average (longest/shortest axis). Magnification the same for every picture. Arrows: decomposition halo/ream around inclusion. **A–F** Different sizes (540 μm, 290 μm, 180 μm, 120 μm, 70 μm, 35 μm); among them are the very biggest and the very smallest inclusion identified. **G, H** Examples of inclusions found in the glass surface, all in bath side, sizes 220 μm, 60 μm [Kasper et al. (2018)]

3.0 Fracture mechanics

The inclusion's size and the surrounding stress field play the deciding roles for crack initiation. In other words, the position of a nickel sulphide inclusion is not really the deciding factor on breakage occurrence; the predominant factors are the local tensile stress combined with the size of the NiS inclusion.

3.1 Heat strengthening

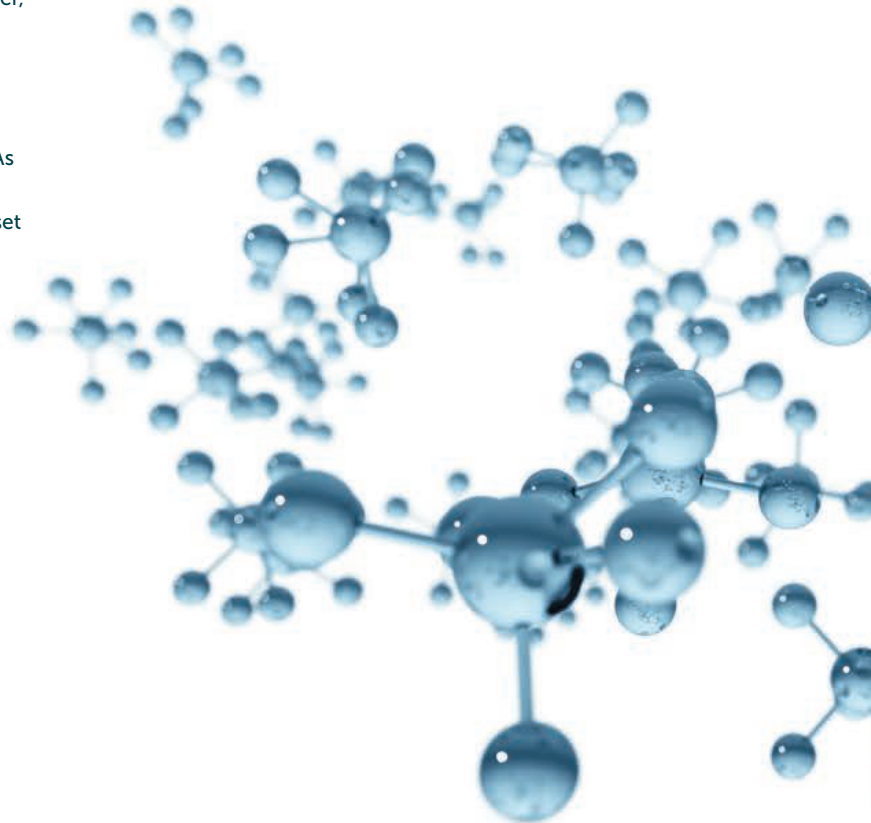
Heat strengthened soda lime silicate glass is defined in the European product standard EN 1863-1:2011 as glass within which a permanent surface compressive stress, additionally to the basic mechanical strength, has been induced by a controlled heating and cooling process in order to give it increased resistance to mechanical and thermal stress and prescribed fracture characteristics.

Although the minimum value for the mechanical strength is defined as 70 N/mm², EN 1863-1:2011 clearly states that the manufacturer shall ensure that the fragmentation always satisfies the requirements of the standard.

Mognato et al. (2011 / 2017) demonstrated that the minimum surface compression of heat strengthened glass should be 35 N/mm² in order to reach the characteristic bending strength of 70 N/mm². However, a surface compression of 55 N/mm² is confirmed as upper limit for still having a correct fragmentation. Based on this data the acceptable range of surface compression meeting the requirements of EN 1863-1:2011 should be $35 \text{ N/mm}^2 \leq \sigma_c \leq 55 \text{ N/mm}^2$. As the tensile stress at the glass' midline is half of the surface stress, the maximum tensile stress σ_0 can be set at 27.5 N/mm².

In opposition to the EN 1863-2011, other product standards are defining a surface compression, e.g. ASTM C1048 – 18 defines that heat strengthened glass with a thickness of 6 mm and less shall have a surface compression between 24 to 52 MPa (i.e. 12 N/mm² to 26 N/mm² tensile stress in the middle).

ISO 22509:2020 specifies that a minimum value for surface pre-stress of 25 MPa is needed. Additionally, the maximum value for surface pre-stress should not exceed 52 MPa to achieve the specified fragmentation behaviour.



3.2 Critical size of the nickel sulphide inclusion

According to Swain (1981) the critical radius of an inclusion with internal pressure P_0 in a tensile stress of σ_0 for spontaneous fracture is given by the equation:

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

Where:

- D_c critical diameter of the inclusion required to cause breakage [m]
- K_{Ic} stress intensity factor
- P_0 surface pressure exerted by the inclusion due to the α to β phase change and also to the different coefficients of thermal expansion between NiS and glass
- σ_0 (maximum) tensile stress in the glass [MPa]

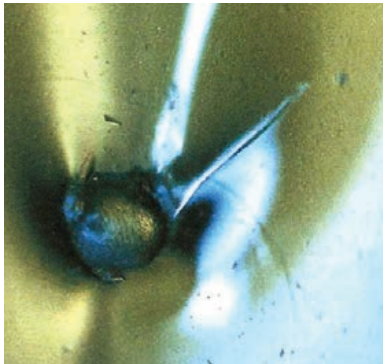


Fig. 7
Nickel sulphide inclusion with intact surface. Photograph taken on breakage mirror after spontaneous breakage. Notched surface structure from crystallization of NiSx of c. $\pm(1 \dots 10) \mu\text{m}$ is clearly visible. No trace of adhering NiSx is visible or detectable by e.g. EDX in the hollow calotte.

Special attention shall be paid to the parameters P_0 and K_{Ic} used by Swain:

I. Surface pressure P_0

a) The density difference between α and β phase is supposed to be 4%. Since then, it has been proven that the volumetric expansion is 2.55% (Groenvold 1995) and not 4%.

b) The difference in thermal expansion coefficients of glass and nickel sulphide is supposed to cause a negative pressure onto the glass during cooling. However, figure 7 shows that there's no cohesion between glass and NiS inclusions. The lack of cohesion between glass and nickel sulphide is related to the non-wettability and insolubility of nickel sulphide in the glass (melt). This fact is important because if there is no cohesion, the stress situation around the (untransformed) inclusion resembles that of a (notched) bubble, but not to that of a "normal" (silicate, silicon, other interlocked) inclusion. It is also important because an (untransformed) nickel sulphide inclusion, that is smaller than its surrounding bubble, does not exert force onto the glass.

c) The strength of glass surface surrounding the nickel sulphide, is affected by the notched nature of the surface of these inclusions. The superficial wrinkles are not in-line with Griffith's assumptions on the weakening of the glass strength due to surface cracks. But, even if the "tip" of the "crack" is rounded, the additional stress concentration due to such a structure is not negligible.

d) Finally, the combination of all these means that a transformation ratio at ambient temperature up to 55% is needed before the first breakages appear (Kasper (2018a)).

Therefore, the surface pressure P_0 is lower than 615 MPa, as determined by Swain. Calculating P_0 using the new data gives a value of 266 MPa at ambient temperature (i.e. for facades).

II. Stress intensity factor K_{Ic}

K_{Ic} is commonly measured on glass using macroscopic methods. These methods reveal the strength of glass under controlled atmospheric conditions, i.e. such as under the influence of water vapour. However, at ambient temperature the gap between the nickel sulphide inclusion and the surrounding glass might really be a vacuum. This means that K_{Ic} increases.

For the time being a stress intensity factor K_{Ic} of 0.79 has been estimated for the situation in facades (at ambient temperature).

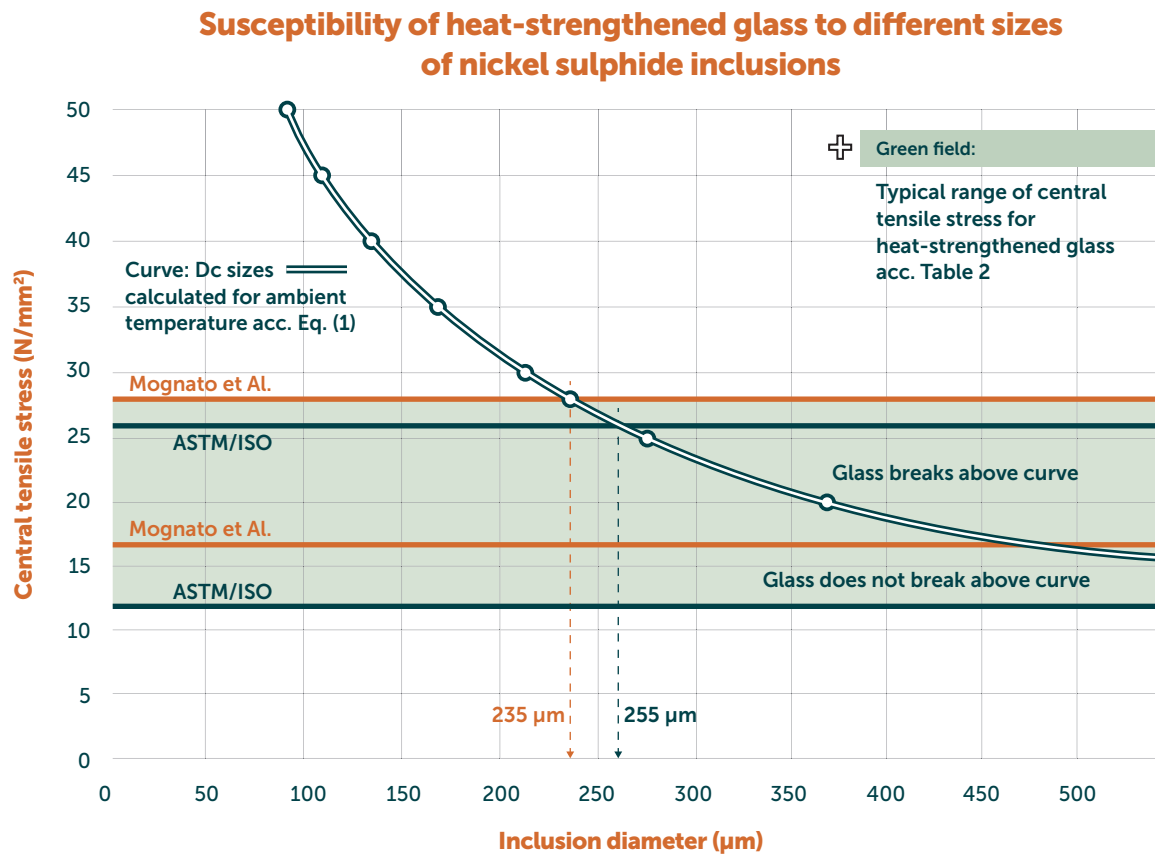
Table 2 shows the minimum and maximum surface compression as specified in official standards and the corresponding critical diameter D_c . The surface compression measured by Mognato et al. (2011 / 2017) is also given with the corresponding critical diameter D_c .

As the threshold of inclusions in float glass can be set at 500 μm due to the gravitational effect in the glass melt, it is unlikely to find critical nickel sulphide inclusions for a tensile stress $\sigma_0 < 17 \text{ N/mm}^2$ or a surface compression $< 33 \text{ N/mm}^2$

Standard/ origin	Minimum surface compression [MPa]	Maximum surface compression [MPa]	Critical diameter D_c linked to the min. surface compression [μm]	Critical diameter D_c linked to the max. surface compression [μm]
EN1863-1:2011	No surface compression is defined, see Mognato et Al.			
ASTM C1048 - 18	24	52	814	255
ISO 22509: 2020	25	52	766	255
Mognato et Al.	35	55	463	235

Table 2 Minimum and maximum surface compression in official standards and according Mognato et Al. (2011/2017) with the corresponding critical diameter D_c

In Fig. 9 the curve Dc calculated for ambient temperatures (i.e. facade application) is drawn as well as the typical range of central tensile stress for heat strengthened glass.



Typically, NiS inclusions having a diameter of more than 235 µm are required to break heat strengthened glass, produced in accordance with the EN 1863-1:2011. This corresponds to records collected of the diameters of nickel sulphide inclusions found at the origins of glass breakages on buildings.

Fig. 9
Susceptibility of heat strengthened glass to different sizes of nickel sulphide inclusions. The curve represents the lower limit (1% breakage probability) referring to the inclusion's size if its position is exactly in the middle of the glass (position out of the middle further reduces the probability). Below the curve, the inclusion's size is supposedly too small to make the glass break. Above the curve, breakage might be caused with increasing probability.

3.3 Breakage probability

Knowing the distribution and the diameter range of NiS inclusions in float glass a breakage probability can be estimated (Kasper (2018)).

Only about 24% of the NiS inclusions found in float glass are bigger than 235 µm. Breakages caused in heat strengthened glass by smaller inclusions are rare and can be disregarded.

Because of the lower stress regime, it can be estimated from the diagram above, in combination with their estimated frequency of occurrence, that in comparison with toughened glass, 31% of the NiS inclusions would be critical even under heat soak test conditions.

Finally, Kasper (2018) came to the conclusion that only 10% of the breakages observed in the heat soak test would happen on a building (i.e. only 10% of the critical nickel sulphide inclusions in the heat soak test are relevant for the building).

Consequently, a qualified but conservative breakage probability of 1 breakage in 1,100 ± 200 tonnes can be estimated for heat strengthened glass.

Table 3 summarizes the estimation process of the breakage probability of NiS inclusions in heat strengthened glass.

Alleged breakage rate in the H.S.T.	6.5		tonnes of glass per breakage (reasonable lower limit; mostly better)	From Kasper – Serruys (2002)
Relevance: Less than	10%		of the breakages in the H.S.T. are relevant for facades	From Kasper (2019 – a,b) and Kasper et al. (2020)
Pure number	24%	20%	of the inclusions causing breakage in the H.S.T. are > 235 / 255 µm	From Kasper (2019 – b)
Impact of tensile stress	31%	25%	of the inclusions are critical (i.e. 69% / 75% arte not critical)	Derived from Figure 9 (i.e. surfaces below / above the curve)
ASTM / ISO		1300	tonnes of glass per breakage are expected for HS-glass	
Mognato et AL.	920			

Table 3 Estimation of the expected maximum probability of breakage in heat strengthened glass in facades

This is still lower than the level of residual risk stated in the heat soaked toughened glass standard ⁸ EN 14179-1:2016, i.e. 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 ⁹ of heat soaked thermally toughened soda lime silicate safety glass.

3.4 Interpretation of the breakage probability

Random events occur in clusters, so in any particular quantity of glass there may be more or less critical NiS inclusions than might be expected.

To give a feel for how randomness affects the numbers of incidents, the diagram in figure 10 shows 100 points randomly distributed (using a computer random number generator) in 100 squares, averaging 1 per square. There is one square with a cluster of 4 points and many squares have none. This is not an atypical event, but has given rise to the idea of NiS inclusions occurring in batches. (Colvin (2016)

The breakage probability has to be interpreted in this way, i.e. it is a statistical approach of an extremely large amount of data gathered over several decades. Therefore, it is not possible to claim that no breakages or a maximum of one breakage will occur because the quantity of glass used for the building is less than $(1,100 \pm 200)$ tonnes of heat strengthened glass.

Moreover, other types of inclusions may cause breakages which are not considered in this study. (Kasper (2018)

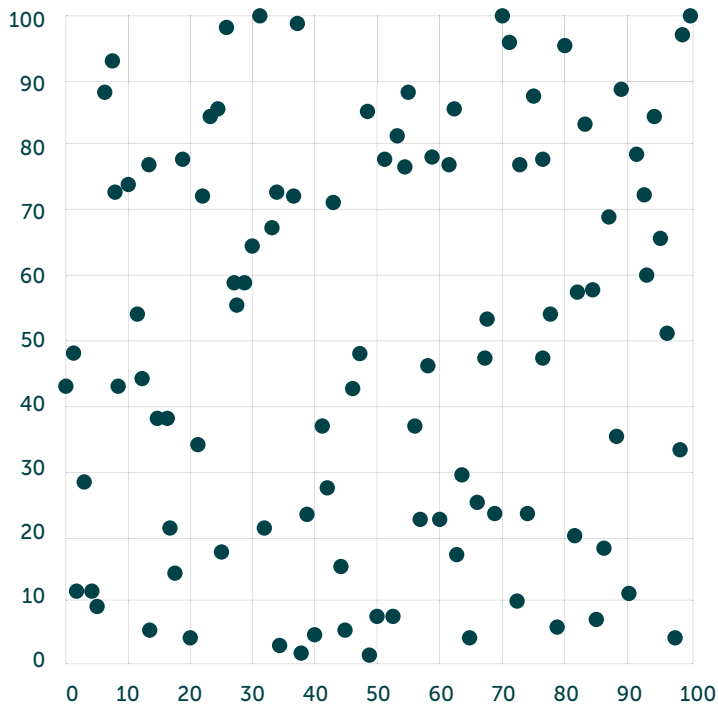


Fig. 10
100 points randomly distributed (using a computer random number generator) in 100 squares, averaging 1 per square

4.0 Timescale for breakages in service

Breakages in service start to occur some time after the glass has been manufactured. In general, one or two years of “incubation time” are usually observed before the first breakages.

The timescale of the curve depends on the air temperatures in service. The higher the average ambient temperature, the faster is the rate of conversion from the α phase to the β phase.

Over time, the rate of breakages rises to a peak and then tails off to nearly zero, after circa 10 years for facade glazing. Fig. 11 shows a typical curve of the cumulative number of breakages plotted over time.

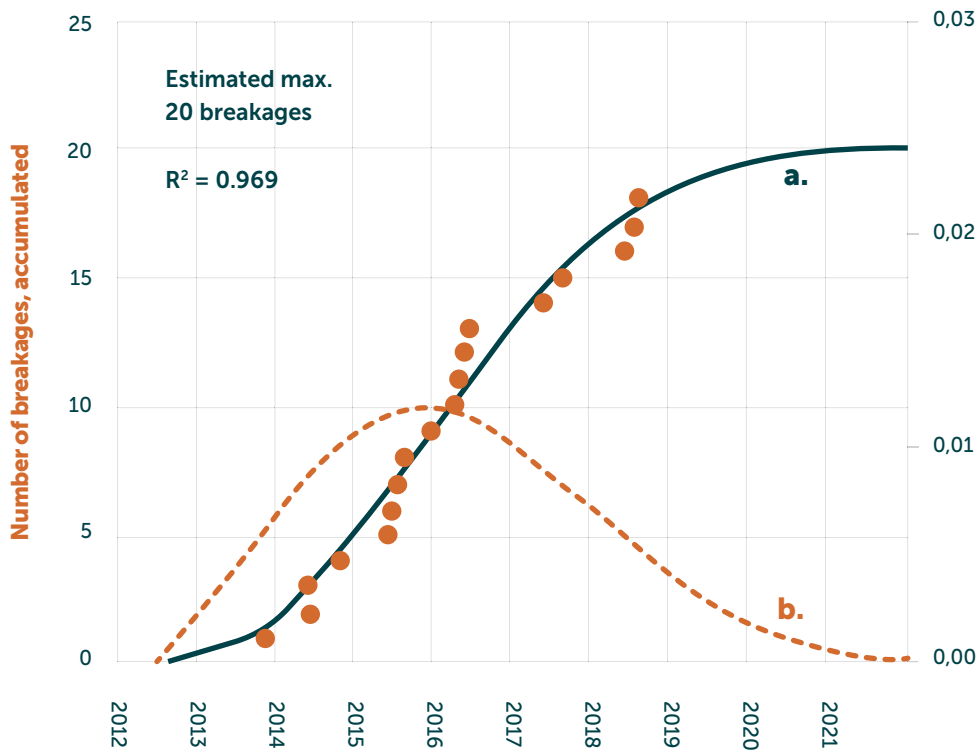


Fig. 11
 Typical time-to-breakage or cumulative NiS breakage curve. Case from practice where toughened glass was produced in 2012 but not heat soak tested. **a.** best-fit of the cumulated breakage data using the Weibull function, multiplied with the fit-estimated maximum breakage number. **b.** Derivative of the Weibull function corresponding to **a.** used to estimate the maximum of the breakage rate.

Fig.12 shows some examples of nickel sulphide breakages identified in heat strengthened glass.

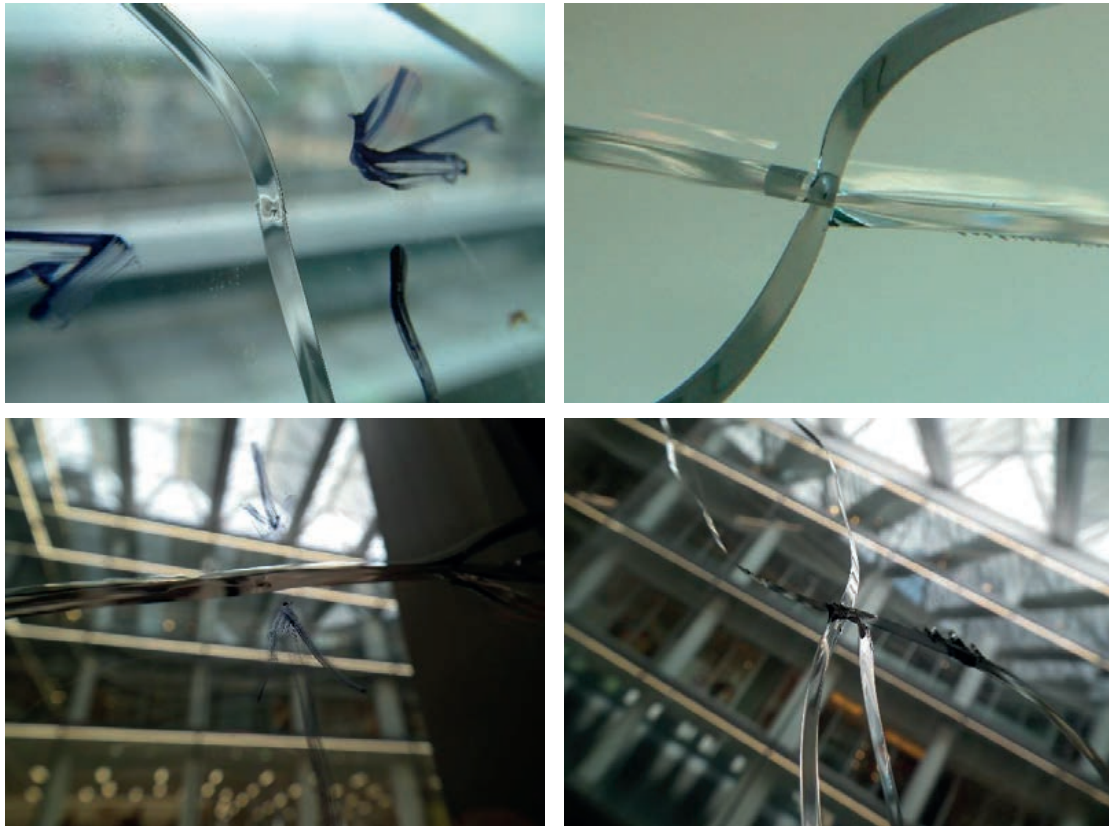


Fig. 12
NiS breakages in heat strengthened glass. All heat strengthened glasses were processed into laminated safety glass. The failure origins were examined using a scanning electron microscope (SEM) fitted with an energy dispersive spectrometer (EDS).

Vision glass does not get as hot in the sun as spandrel panes and the peak rate¹⁰ of breakages in vision glass is around 4 to 6 years after manufacture.

Insulated spandrel panes, which are subject to solar radiation, can reach high temperatures and the peak rate¹⁰ of breakage is about 2 or 3 years after manufacture.

For glass indoors or in cladding which does not receive solar radiation, the peak rate may be around 8 to 10 years after manufacture.

Breakages due to NiS inclusions have been known to occur more than 20 years after the glass was manufactured.

5.0 Conclusion

Although the residual risk of heat soak tested thermally toughened safety glass is very low, heat strengthened glass is often preferred under the assumption that it does not suffer from spontaneous breakages due to NiS inclusions.

This is definitely untrue. The lower tensile stress in the centre of the heat strengthened glass makes the glass much less vulnerable to spontaneous breakages compared with toughened glass (not heat soak tested). However, a conservative breakage probability of 1 breakage in 1,100 ± 200 tonnes is estimated for heat strengthened glass.

More data of broken heat strengthened glass due to NiS inclusions found on facades is required to confirm the breakage probability, which in reality might be much lower.

Heat strengthened glass is not a safety glass and is most often processed into laminated safety glass. If a NiS inclusion caused a breakage of a laminated heat strengthened glass, it would stay in place due to its specific fragmentation as required by EN 1863. Hence the low breakage probability, in combination with its usage as laminated heat strengthened glass, means the heat soak test is usually not done.

Finally, the timescale for breakages in service can be limited to a period of 10 years. Indeed 90% of the breakages normally occur within a period of 6 to 8 years after production, even in a cold climate. Although nickel sulphide breakages have been observed after more than 20 years, this remains an exception and it may be concluded that seldom a breakage happens more than 10 years after installation.

Notes:

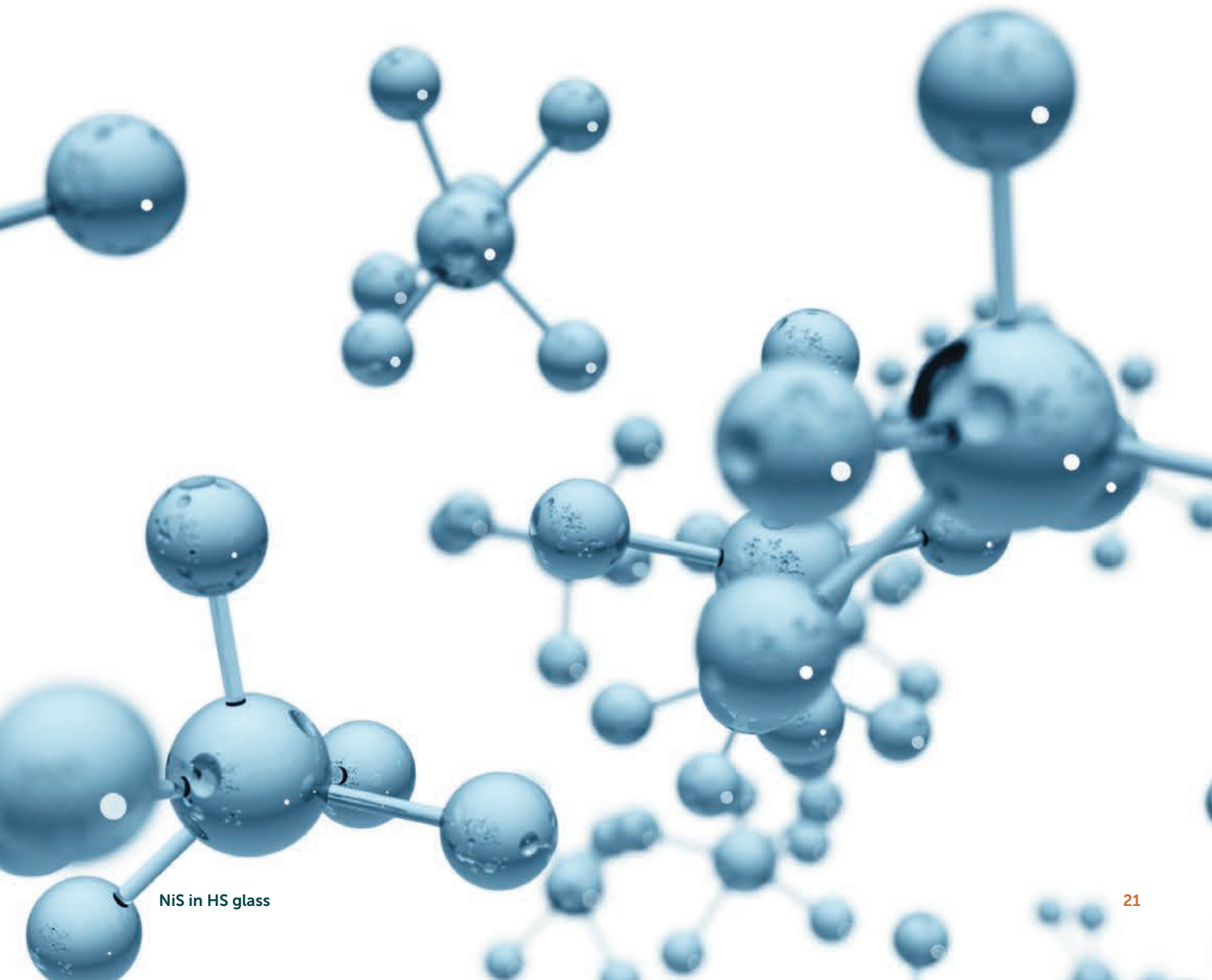
- ¹ ICI House building in Melbourne, Australia, was one of the first buildings that used toughened glass to avoid thermal breakages in the façade. The installation was completed in September 1958. However, it was not until 1960 that the toughened glass started to break. Ballantyne identified the cause of the glass failure to be of one particular type of inclusion, nickel sulphide.
- ² For the construction of the Deutsche Bank twin tower in Frankfurt, NSG Pilkington's German department developed the heat soak test.
- ³ Depending on oven design and glass stacking, an oven air temperature of 290 °C could leave parts of the glass at temperatures below 220 °C for the whole duration of the heat soak test.
- ⁴ European Committee for Standardization
- ⁵ The reason is doubtlessly due to both the accelerated decomposition of very small inclusions and the detection limit of the detection method applied. (Kasper et al. (2018))
- ⁶ The classes > 350 µm only contain a small number of members and are therefore subject to high "statistical scattering". Though out of 457 nickel sulphide inclusions documented in the Saint-Gobain laboratories, only one is > 600 µm and only three > 500 µm. (Kasper (2018 a, b) and Kasper et al. (2018))
- ⁷ Model calculations e.g. in Bordeaux and Kasper (1997), Swain (1980,1981) reveal that inclusions smaller than 50 µm are unable to cause glass breakage. Therefore the design limit (detection limit) chosen by Kasper et al. (2018) is 40 µm which is low enough to record every potentially harmful inclusion.
- ⁸ Exactly: "Glass in Buildings: Heat Soak Tested Thermally Toughened Safety Glass". This is a product norm describing in detail how the safety glass is made, and how its quality is assured.
- ⁹ Kasper (2018) and Kasper et al. (2018; 2020) estimate the residual risk of only 1 breakage in 10,000 tonnes of really heat soak tested glass.
- ¹⁰ 90% of the breakages are normally done 6 to 8 years after manufacture.

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The authors



John Colvin

John Colvin joined Pilkington in 1969 with a Maths degree from Cambridge University. He started in the Technical Sales Laboratory, which was effectively a R&D department for glass usage, subsidiary to the Marketing function. He was involved with formulating calculations for the strength of glass for inclusion in British Standards and was involved in developing the structural design of Pilkington 'Armourplate Assemblies', leading to the development of Pilkington 'Planar'. He was also responsible for background work which, in BS 6262, considered laminated glass effectively the same wind load resistance as monolithic glass of the same thickness, and which was used in the load charts he developed for glass products incorporated into BS 6262 and BS 5516. He subsequently became manager of Pilkington Glass Consultants, an internationally recognised glass consultancy, which led the glass industry in widening the usage of glass in buildings. In 1998, he joined Hansen Glass, a glass processing company, as Technical Director, before becoming an independent consultant in 2004. John is a valued member of several British, European and ISO glass standard committees and has made major contributions to the development of the standard EN 14179 for heat soaked toughened glass.



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Andreas Kasper is a chemist retired from Saint-Gobain Germany. He worked there in R&D for more than 32 years. Besides, he was a lecturer at the Aachen University on glass technology and basics for more than 15 years. His work at Saint-Gobain mainly focused threefold on emission and combustion of the melting tanks, glass formation in the float bath and quality of the molten glass with one focus on nickel sulphide inclusion's formation and spontaneous breakage of glass panes. During his working time, he has published numerous articles in professional journals and held many speeches on different expert conferences. In acknowledgement of his merits, the German Society for Glass Science and Technology (HVG / DGG) awarded him the Adolf-Dietzel-Price (2002) and the Golden Gehlhoff Ring (2014).



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Frank Rubbert, Group Industrial Hygiene Manager at Saint-Gobain, has been working for nearly 30 years in the flat glass industry in several management positions in production, R&D and quality management.

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NiS in HS glass

A technical paper

