RESEARCH PAPER

Spontaneous cracking of thermally toughened safety glass part three: statistic evaluation of field breakage records and consequences for residual breakage probability

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Received: 27 February 2018 / Accepted: 26 November 2018 / Published online: 18 December 2018
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Abstract The present paper is the third part of a small series of publications dealing with the problem of spontaneous breakage of thermally toughened glass. The HST standard EN 14179-1:2005 (revised 2017) made this glass significantly safer; the present small series aims primarily to bring new arguments into the discussion on its real safety. Another aim is to rectify obsolete (pseudo-)facts repeated again and again in respective publications although they are disproven since a long time. The present third part of the series deals with statistic evaluation of datasets from field breakages. In these, every inclusion has caused a breakage, contrarily to those in the 2nd part. The data have already been partly (and as one mixed dataset) published in 1997; since then, the author's working group continued accumulating breakage data. Today, finally, this data collection is sufficiently numerous to be split into the two parts, namely breakages recorded on buildings and in Heat Soak Test, and to be evaluated separately by means of statistical analysis. Like in the previous papers, also this evaluation results in a clear difference in breakage probability, very much higher in HST. As a matter of fact, the latter's breakages' devolution cannot

simply be extrapolated in order to obtain the safety of the tested glass on buildings; the result of this extrapolation is only a minimum limit, but factually far away from reality. Finally, the experience of the author's employer, Saint-Gobain in Germany, is described for the time since the introduction of said standard. Also this pragmatic approach reveals that the safety of Heat-Soak Tested Thermally Toughened Safety glass against spontaneous breakages is much better than forecasted from the HST breakages' evaluation.

Keywords Façade · Toughened glass · Spontaneous breakage · Nickel sulphide

1 Introduction

Since Ballantyne (1957) wrote his extensive report on spontaneous cracking of façade cladding made of toughened glass, significant research has been carried out on the subject. The state of the actual general knowledge is summarized in a well-founded review published recently by Karlsson (2017), but it cannot be ignored that some of the facts mentioned therein must be looked at to be outdated under today's point of knowledge; the present publication occupies with this, among others, in its summary. However, new arguments are urgently needed in the discussion on safety of the HST; this is the primary aim of our small series of three papers. The present one is part three thereof, and it is recommendable to read both other parts before this one.

Electronic supplementary material The online version of this article (<https://doi.org/10.1007/s40940-018-00093-z>) contains supplementary material, which is available to authorized users.

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Additionally, in order to understand the details presented here, at least basic knowledge on the problematics of spontaneous breakage of thermally toughened glass is presumed.

In 2009, when O. YOUSFI terminated his PhD study in Grenoble (France), he had found out that the α to β transformation remains incomplete for a certain number of nickel sulphide inclusions if the temperature is above 280 °C. His work was published (Yousfi et al. 2007, 2009, 2010a, b, c, 2011) and extensively discussed in the glass community, especially in both ISO and CEN standardization committees. In consequence, at the occasion of a routine review of EN 14179–1 and publication of ISO 20657 in 2016, the decision was taken to change the holding temperature regime from 290 °C to below 280 °C in order to allow every nickel sulphide inclusion to complete its transformation, and thereby to make the HST-glass safer.

Due to the C-shape of the time-temperature-transformation (“t-t-t”) curves measured by YOUSFI, this temperature change does not have a strong impact onto the transformation rates (even nearly not at all for pure NiS). Consequently, it is judged to be uncritical, and that the effect of complete transformation of every nickel sulphide inclusion preponderates largely as the benefit of this condition change. It was also decided on CEN and ISO level that the holding time should remain the same (2 h). A scientific reason for this is that, rather close to the reversal point of the C-shaped curves, Arrhenius’ law is not valid. Especially, the transformation rate does not increase anymore with increasing temperature; it even decreases beyond a certain point because complete transformation is no longer possible; it becomes even more incomplete with increasing temperature. Also this fact was already revealed by YOUSFI. Although facts seem to be clear, there is no full agreement, e.g. in Germany, and therefore, there’s a certain risk that HST-glass will strongly be restricted for façade applications.

The present publication series reveals, among others, that temperature prolongation is factually not necessary, and that anyway, the HST-glass is safer against residual breakage than estimated before. Two papers have recently been published (Kasper 2018; Kasper et al. 2018), and the present third one completes our small series dedicated to advance significantly the common knowledge and experience on nickel sulphide inclusion caused breakages.

The first among the mentioned papers (Kasper 2018) deals with the detailed effects of the crystallographic and physical properties of the nickel sulphide species contained in these inclusions; it demonstrates that solely under this compositional aspect, only c. 40% of the breakages in a Heat Soak Test (‘HST-EU’) according to, e.g., EN 14179-1:2005, are factually relevant for the façade. The reason for this is not only the difference in thermal expansion between NiS and glass, but also the fact that many inclusions contain, besides NiS, the indifferent phase Ni₉S₈. On the other hand, the same paper reveals that also other than nickel sulphide inclusions definitely cause breakages in HST. Their behavior therein is a nearly perfect mimicry of that of nickel sulphide inclusions although they are not subjected to the well-known and extensively discussed allotropic α to β phase transition. It is a given (see e.g. Karlsson 2017) and shall not be put into doubt here that the latter is one root cause for spontaneous breakages on buildings if the glass is un-soaked. However, the widespread, intuitive, but although unsubstantiated deduction thereof that the kinetics of its phase transition were “proportionally” responsible for the time-to-breakage devolution in the HST is revealed to be definitely wrong.

The second paper (Kasper et al. 2018) of the present small series focuses on a set of data produced by inspection of annealed glass. The automated method identified almost all present nickel sulphide inclusions correctly. The evaluation of the trials related with these inclusions proved what beforehand was only assumed, namely that the nickel sulphide inclusions in raw glass are found everywhere in the glass section, but they are not uniformly distributed, most probably due to gravitational settling. All of the inspected glass panes (not only those containing a nickel sulphide inclusion) were toughened and subject to a HST. Only 25% of the nickel sulphide inclusions identified beforehand led to breakage. Contrariwise, additional breakages (of panes without identified nickel sulphide inclusion, or polluted with other than nickel sulphide inclusions) were also not obtained. The paper discusses the reasons under the aspects of size and position of the inclusions.

The present third part of the series deals with statistic evaluation of datasets from field breakages. In these, every inclusion has caused a breakage, contrariwise to those in the second part (Kasper et al. 2018). The data have already been partly (and as one mixed dataset) published in 1997; since then, the author’s working group continued accumulating breakage data. Today,

finally, this data collection is sufficiently numerous to be split into the two parts ‘Building’ and ‘HST-EU’ (see glossary), and to be evaluated separately by means of statistical analysis.

2 NiS breakages from HST and buildings in Europe: long term collection and analysis

Due to long term occupation with the theme of nickel sulphide inclusions and related spontaneous breakages at SG, respective breakage departure centers were collected during more than 30 years. The breakage cause is always found in the inner surface of the so-called butterflies. However, it is not always a nickel sulphide inclusion leading to the formation of such a butterfly, see Kasper (2018). Regarding fracture-mechanics, the butterfly is a product of the glass toughening and the energy stored in this regard, but definitely not of the kind of its root cause.

It is, however, not possible to check the real breakage cause in industrial HS-testing. These breakages occurring in the glass processing factories always result in about two to three big buckets full of small cullet pieces. Within our R&D projects, we tried several times to find the breakage center therein but had to realize that this is virtually impossible.

On the other hand, we knew from experience from longer time ago that > 95% of the breakages in HST are due to nickel sulphide inclusions, reported e.g. in Kasper et al. (2000).

There is a reason behind this fact: Breakages in HST can be due to

- nickel sulphide inclusions > c. 50 μm ;
- other inclusions (stones etc.) > c. 500 μm (i.e. c. 10 times the size of the nickel sulphide inclusions);
- big bubbles in the same range of size as the stones.

Whilst the big bubbles and stones are reliably detected by the quality control units of any float line since a long time, the nickel sulphide inclusions are, even today, significantly too small for online detection, and they do not cause optical distortion. Therefore, they pass and, under normal conditions, they are definitely the major cause for toughened float glass breakages in the HST. This is not forcedly true for patterned glass, see Kasper (2018).

In previous SG publications in the early 2000’s, e.g. Kasper et al. (2000), no difference was made between

breakages in HST or on buildings. The same is true for all publications the author knows; also (Quiquampoix and Wagner 1977)’s review already mentioned above does not report this. The reason why this seemingly obvious fact was not accounted for is twofold, namely that, at the time, this difference seemed to be irrelevant and, on the other hand, the number of available butterflies from buildings was too low to make a statistically significant evaluation.

After 30 years of collecting activity, a sufficient number is available now in both groups: 225 butterflies from HST and 101 butterflies from un-soaked glass from building breakages. This considerable collection allows us to make a statistically relevant evaluation. The mathematical tools applied in the present paper are the same as already described in Kasper et al. (2018); besides histograms, the two-parametric Log-Normal function is applied successfully. Additionally, the GAUSSIAN function is used in the present paper as a part of the characterization of the position alignment of the inclusions over the glass section.

2.1 Positions in the glass section

For both datasets (see glossary: ‘HST-EU’; ‘Building’), size and position were measured precisely. Measurements were carried out using two different methods, namely standard light microscopy and Scanning Electron Microscopy (SEM). Both methods are doubtlessly so well-known that their description can be omitted in the present paper. The precision of both methods is certainly different, but predominantly, the specimens themselves have a non-negligible impact on the scattering of the measurements. The form of the glass fragments is irregular, the surface containing the origin of the fracture (“breakage mirror”) is never perfectly even. If a nickel sulphide inclusion or its empty calotte is found in such a surface, the diameter observed on the breakage mirror might in detail be different from the true maximum diameter of the inclusion. Regarding an inclusion, the hidden part inside the glass is even probably a bit larger than the diameter observed in the surface. If, on the other hand, the inclusion and the surrounding glass are polished for quantitative analysis, the observable diameter might also be smaller than the maximum. The inclusions discussed in Kasper et al. (2018) (and the same is true for many other publications, e.g. Barry and Ford (2001) make clear that they

Fig. 1 Positions of nickel sulphide inclusions, raw values as measured. Cumulative delineations (each number equals to one breakage event). (a) Breakages ‘HST-EU’. (b) Breakages ‘building’, un-soaked glass. (c) Midline of the glass

are often not of exactly spherical, but of elliptical shape; the fracture mirror defines the observation plane, and this is still true if the specimen is polished in order to obtain a flat surface for said quantitative analysis. Therefore, the resolution of the measuring devices is of minor influence in relation to the natural imponderability of the specimen’s dimensions.

However, estimating the average size of the nickel sulphide inclusions to be 200 μm and the best precision to be $\pm 1 \mu\text{m}$, the worst case to be $\pm 10 \mu\text{m}$, an average precision of c. $\pm 3\%$ in size is expected; concerning the position, a similar estimation for the position (9 mm glass/ ± 0.3 mm) leads to approximately the same relative error.

The measured positions in the glass section are displayed in Fig. 1. They should always have been measured in relation to the same glass side, namely (by in-house definition) to the bath side; the latter refers to -50% in the figure. During the long collection time of 30 years, it can, however, not be fully excluded that in some seldom cases the bath side was not correctly identified. This causes a small imponderability in the evaluation.

Three remarkable qualitative observations are made:

- There’s a point-symmetric structure in both datasets; in fact, all of the four branches look similar and systematically “wavy” in the same way. Particularly, the pivotal point is not situated in the middle of the dataset, but in the middle of the glass. This is an

important observation; it will be used below as a significant argument.

- A high number of breakage centers is found in a narrow range around the glass’ midline.
- There’s a significant dissymmetry in the position values. Very obviously, more breakage departure points are situated in the lower half of the glass section (towards bath side) than in its upper half.

Table 1 quantifies this finding. Inclusions within $\pm 1\%$ of the glass midline are counted extra. The visual impression was not misleading: The difference is (t-test) significant in both cases. The respective results of nickel sulphide inclusions identified in annealed glass (last line in Table 1, marked with *), already extensively discussed in Kasper et al. (2018), are added for comparison. Visibly, they correlate with the present findings. Contrariwise, the position alignment in the annealed glass does not show an accumulation close to the midline; reasonably, this either cannot be expected in view of the generation mechanism for these inclusions. The influence of gravitational settling onto the position alignment of the nickel sulphide inclusions was already discussed and evaluated in the mentioned paper. Independently, every result in Table 1 shows a similar trend. However, the numbers differ systematically in the different cases; also this is not unexpected because the datasets ‘Building’ and ‘HST-EU’ represent breakage cases, whilst the inclusions in the annealed glass are observed without this filter that obviously generates a limitation of the range ($\approx$ no breakages under compression) and a ‘Spike’ close to the midline. The physical cause behind the observed dissymmetry is the density difference¹ between glass and NiS and the fact that larger inclusions sink down more rapidly than smaller ones (“gravitational settling”; see Kasper et al. 2018). In order to prevent quality problems, most of the glass melting furnaces run with thickness-independent tonnage; therefore, the residence time of glass and the (precursors of the) nickel sulphide inclusions in whole furnace, but especially in the refining and cooling section of the furnace is always the same. Thickness is then adjusted by different lehr speed only, and consequently, the alignment profile of the inclusions is con-

¹ Density of glass: 2.5 g/cm³; of α -, β -NiS c. 5.4 g/cm³ (at ambient temperature).

Due to the small size of the inclusions, the gravitational forces are weak, the dropping speed slow; but an effect is obviously visible and accessible to mathematical estimation.

Table 1 Dissymmetry parameters of breakage departure points, breakages definitely due to nickel sulfide inclusions

Dataset	Numbers of breakages from...Relation				t from STUDENT test	Significance of difference (%)	Inclusions accumulated close to the midline
	Lower half	Upper half	Lower half (%)	Upper half (%)			
Building	49	21	49	21	17	> 99.5	31%
Heat-Soak-Test EU	125	62	56	28	17	> 99.5	17%
In annealed glass*	85	50	63	37	23	> 99.5	n.a.

% values rounded for clarity. The number of the inclusions close to the midline ($\pm 1\%$ criterion) is identical with the ‘Spike’ length in Table 3 below (criterion “above parabola”)

*Discussed in Kasper et al. (2018)

served and only compressed simultaneously with the glass thickness to be produced. Also, comparing different furnaces, their tonnage is, due to economic reasons, normally close to the design tonnage or less than 10% lower, comprising a fixed, more or less universal relation of (throughput) to (glass mass in furnace). Therefore, logically, the absolute glass thickness has, if any, only a minor impact onto the relative position of the nickel sulphide inclusions over the glass section.

For more significant statistical evaluation, the measured parameter “distance to bath side” was transformed into “relative distance to the glass midline”. For this, the individual position value was divided by the individual glass thickness, and 50% subtracted. Then, all inclusions’ positions were projected into one half of the glass section. Mathematically, this transposition is trivial; all x values simply obtain the same (chosen negative or positive) sign, and the breakage numbers are collocated again in ascending order. Thereby, all inclusions seem to be in one quadrant of the glass section. The dataset’s endpoint is now exactly on the midline of the glass. The latter would lead to a visible misfit if it was not true; however, Fig. 1 and the congruency of the ‘Spike’ length in Tables 1 and 3 both reveal that it is indeed exactly like this.

Thereby, mathematical-statistically, the ranges of the data transform in the following way:

- $x \in \{0, 1\} \rightarrow x \in \{-1/2, 0\}$ & $y \in \{0, 1\} \rightarrow y \in \{0, 1/2\}$
(result: an ascending curve at left of midline)

or

- $x \in \{0, 1\} \rightarrow x \in \{0, 1/2\}$ & $y \in \{0, 1\} \rightarrow y \in \{1/2, 0\}$
(result: a descending curve at right of midline)

In the first case, all x values are negative, in the second positive; in comparison of both, the data are mirrored at the midline. However, the dataset remains a cumulative curve, admittedly only one half, and correspondingly, a (combined) fitting curve must be (composed of) cumulative GAUSSIAN’s and/or parabolas; it is not the derivative, even if it looks like (e.g. curve Fig. 2b).

This value transformation neutralizes a potential side confusion problem, and it has another genial effect: It approximately doubles the apparent precision of the datasets; they become more “dense”, more figurative, and curve fitting is significantly clearer like this.

2.1.1 Simple approach using one GAUSSIAN for best-fitting

The datasets prepared as described are—in a first order approach—fitted using one (half, see above) cumulative GAUSSIAN. This is the simplest approach taking into account only the symmetry of the data in relation to the glass midline.

The deviations from the best-fit half-GAUSSIAN look systematic, as already discussed above, and they are significantly higher than expected from the measurement precision estimation. PEARSON’s R^2 is below 0.9 in both cases; therefore, this simple fitting cannot be looked at to be a good approach. However, the STUDENT t value indicates a clear rejection of the null hypothesis and thereby a (possibly weak) functional correlation.

Anyway, hypothesizing that the GAUSSIAN is (in this first order approach) a possible way of fitting both datasets already allows drawing some interesting conclusions. The obvious systematic deviations thereof are discussed later.

Table 2 summarizes the respective fitting results from Fig. 2. The parameters of the GAUSSIANS do not

Fig. 2 Positions of nickel sulphide inclusions transposed into one half of the glass section. **A** Breakages ‘HST-EU’, $N = 225$. **B** Breakages ‘Building’, un-soaked glass, $N = 101$. (a) Measured data. (b) Cumulative GAUSSIAN (left 1/2 only), from least

square fit. (c) 99% limit [$\pm 2.58 \sigma$] (GAUSSIAN value = 0.5% on both sides). (d) Tensile stress parabola in thermally toughened glass, arbitrary scale [maximum (50 ± 10) MPa], for orientation

Table 2 Comparison of positions of nickel sulphide inclusions in ‘HST-EU’ and ‘Building’ Equations ref. to Kasper et al. (2018)

HST	Building	0% = glass midline; 50% = surface
26%	24%	Half width of (full) GAUSSIAN (calculated using EXCEL solver)
30%	28%	Zone in the glass middle where statistically 99% of the inclusions are found
29%		Zone in the glass middle with tensile stress
$\pm 3.6\%$	$\pm 3.1\%$	Relative scattering $\pm s$ on position (x) scale (from deviation from best fit curve)
$\pm 5.8\%$	$\pm 6.6\%$	Relative scattering $\pm s$ on number (y) scale (from deviation from best fit curve)
0.7984	0.8981	R^2 /PEARSON’s coefficient of determination, Eq. (1c)
30	30	t/STUDENT’s parameter for statistical testing, Eq. (1d)
$> 99.5\%$	$> 99.5\%$	Probability $(1 - P)$ against null hypothesis “ $R^2 = 0$ ” from t

differ a lot, and the scattering is comparatively high. However, at the limit, the ‘HST-EU’ fitting curve is broader. Breakages comprise $\pm 30\%$ of the glass section instead of $\pm 28\%$ for ‘Building’, whereas the point of stress neutrality is at $\pm 29\%$ for both (²; see also Nielsen et al. 2009) for more explanation). This means that under ‘HST-EU’ conditions, inclusions slightly within the compressive zone do already lead to breakage. On buildings a certain (small) tensile stress is needed for failure. This is logical but, in view of the scattering in x direction, not directly proven, at least not using this simple approach.

² Exact calculation: Stress neutrality point at 28.867...% for the ideal parabola. Due to natural fluctuations and measurement uncertainty of the real glass stress devolution, it is normally agreed to round this value to 2 digits, i.e. 29%.

2.1.2 More complex approach taking into account the stress parabola

The simple approach above is not really satisfying. Not only because the fit does not look good, but also under a theoretical point of view, the breakage probability should, to some extent, depend on the stress surrounding every single inclusion so that a correlation with the stress profile frozen in in the toughened glass should be visible somehow in the position’s diagram of the breakage departure points. On the other hand, a purely stochastic alignment like curve (b) in Fig. 2 virtually cannot be expected.

Both dataset curves look like a (half) helmet with a fade-out (like a brim) at the basis and a spike on top. Therefore, below, they are named the ‘spiked helmet’ curves as an acronym to make description shorter.

However, the argumentation in this chapter might perhaps be an over-interpretation. Therefore, the following evaluation and interpretation should be looked at to be a discussion contribution based on the data status presently available. In this regard, it could be a trigger to motivate other researcher's evaluations on similar, eventually already existing datasets whose evaluation will hopefully confirm the present proposal. Here-with, the author offers his help in this complicated evaluation on a strictly confidential basis.³

As already pointed out, it is logical to hypothesize that the parabolic stress profile in the thermally toughened glass controls the occurrence of the breakages, mainly its positive (tensile) part. Therefore, the stress parabola is added in Fig. 2, and it will help to interpret the 'spiked helmet'. The stress parabola's generally known fixpoints are, according to (15) and (16), its maximum at the midline with $y = (50 \pm 5) \text{ MPa} [\pm s]$, and its abscissa zero-points at $x = \pm 29\%$ of the glass section as already explained above.

Before trying a fracture-mechanic interpretation, the observations are described under these points of view.

Description Comparing the positions' devolution in Fig. 2 with the simple GAUSSIAN approach, systematic and similar deviations are observable in both datasets and even in each of the four branches of Fig. 1.

- The average derivation between the measurements and the simple GAUSSIAN is much higher in both x and y direction (c. $\pm 3.5\%$; $\pm 6\%$) than is expected from the measurement precision ($\pm 1.5\%$, see above and taking into account the impact of the folding of the data).
- The 'Spike', a pointy accumulation around to the glass' midline, appears in both cases; it is visibly much stronger in the 'Building' dataset. This effect cannot be explained by measurement uncertainty or related scattering of the data. It seems to be a systematic effect, although there is, at first sight, no evident reason.
- The 'HST-EU' curve seems to be broader. However, the fade-out is not precisely fitted in the simple approach; consequently, this result is questionable.
- In both cases, the measurements do not match the fit in the middle part. They differ much more than to

be expected from the measurement precision, and they show a bulge outside the GAUSSIAN.

If the 'Spike' is ignored (i.e. the GAUSSIAN end-point on the ordinate is lowered), this bulge is shifted entirely above the shifted GAUSSIAN. The latter resembles to a parabola running in parallel to the stress parabola.

In the following argumentation, reasons shall be identified for these deviations. However, note that this is a hypothesis. It cannot be entirely excluded that the 'Spike' is an artifact of the measurement imprecision although it looks too systematic and is similar in both independent datasets. To re-assure this finding, other researchers are invited to repeat the collection independently, applying precise measurements in every case. A repetition of the measurements on the samples of the present dataset is unfortunately not possible because in the meantime the respective laboratory was moving two times, and too many of the older samples were lost.

In order to explain the observations in more detail, it is important to keep in mind that the tensile stress is always maximal in the glass center. Referring to established theories, the stress profile shows a parabolic profile that is, consequently, relatively flat at its maximum⁴ in the middle of the glass pane. Contrariwise, the breakage departure point's devolution forms a spike in the middle. Also the rest of the curve is not looking like a simple GAUSSIAN, neither like a parabola. Obviously, reality is more complex and cannot be characterized by one single curve.

At closer sight, four different sections appear in both datasets, namely, from the glass' midline to its surface:

1. the 'Spike';
2. a section where the curve looks like a (reversed) parabola running parallel to the stress curve;
3. a section where the curve looks, at its basis, like a probabilistic 'Fade-Out' tail;
4. a part, between the end of the 'Fade-Out' and the glass surface, where breakage departure points are never located. (This part's "fit" is the zero line.)

These four sections might refer to different locations within the glass section where one specific property of the (glass/nickel sulphide inclusion) interaction preponderates. Although somewhat smeared by random

³ Everybody needing support is welcome to write a short note to Andreas.Kasper@Saint-Gobain.com or AKasper@gmx.de to establish contact without obligation.

⁴ According to general convention in materials science and physics, tensile stress has a positive sign, compressive stress is negative.

Fig. 3 ‘Spiked helmet’ curve fitting: Alignment of the breakage centers in broken glass panes. Fitted curve mirrored at glass’ midline for better visibility; not to be confused with derivative curves. Values’ compilation in Table 3. **A** Breakages ‘HST-EU’. **(B)** Breakages ‘Building’, un-soaked glass. (a) Measured inclusion positions, triple fit curve underlaid. (b) ‘Spike’ around glass’

midline, cumulative 1/2-GAUSSIAN underlaid. b1 End point of ‘Spike’. (c) Parabolic section of (a). (d) ‘Fade-Out’, cumulative 1/2-GAUSSIAN. (d1) Take-off of ‘Fade-Out’ (see definition in Kasper et al. (2018)). (e) Tensile stress parabola in toughened glass. (e1) Maximum at glass midline. (e2) Zero at 29% of glass thickness. (f) Intersection (c)/(d)

scattering, two discrete inflexion points are visible in both curves. In these, and at the Take-Off (for definition see (Kasper et al. 2018) of the ‘Fade-Out’, condition change has to be assumed, i.e., in the direction from glass surface to midline, in each point an additional effect seems starting to play a significant role.

Consequently, instead of one function, three of them are applied for fitting, namely a GAUSSIAN, a parabola and a second GAUSSIAN. The basis of this “construction” is chosen to be the parabola, and both GAUSSIANS are attached to it according to the range where they obviously appear.

According to the nature of the three single curves, nine variables are principally needed to adapt them to the data sets. Three among them (the midpoints of both GAUSSIAN’s and the impair factor / midpoint of the parabola) are, however, predefined zero because of principal symmetry conditions in the glass section. The

sum of the heights of parabola and ‘Spike’ is 100% so that only one among them is free.

The five remaining free parameters are then

- σ and height of 1st half-GAUSSIAN - ‘Spike’
- [Crest above abscissa of (reversed) parabola (a in $y = a - b * x^2$) fixed by spike height.]
- Largeness of Parabola (b in $y = a - b * x^2$) at abscissa level defines foot point of parabola.
- σ and height of 2nd half-GAUSSIAN - ‘Fade-Out’

These five parameters are obtained by iteration, using the EXCEL® solver and partial least square fits. The result is drawn in Fig. 3. The fitting parameters are summarized in Table 3, together with other calculated values.

The mean variations of the data around both triple-curve combinations and other statistical parameters are listed in the four lowest lines of Table 3. Now, the mean variation fulfills the expectance from measuring

Table 3 ‘Spiked helmet’ curve fitting parameters and uncertainty estimation: positions of nickel sulphide inclusions in ‘HST-EU’ and ‘Building’

Positions		Estimated tensile stress		Number of breakages in dataset	‘Spike’ length [A]	‘Spike’ length [A]	Crest [A]	Merging point w. 2nd GAUSSIAN [A] (“f” in Fig. 3)	Largeness at merging point [B]	Root point on abscissa	σ [B]	μ (Midpoint)	Take-off [B]	Total fit mean variation [B]/Eq.(1a)	R ² /PARSON’s coefficient of determination, Eq.(1c)	t/STUDENT’s parameter for statistical testing	Probability (1 - P) against hypothesis “no correlation”	1st GAUSSIAN ‘Spike’	Parabola	2nd GAUSSIAN “Fade-out”
HST-EU’	‘Building’	Uncertainty Eq.(1b)	‘HST-EU’	‘Building’																
225	101	—	50 ± 5 MPa	38.7 ± 4 MPa	37.6 ± 4 MPa	33 MPa	< 38 MPa	—	—	—	—	—	—	—	—	—	—	—	—	—
0.6%	0.5%	n.a.	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
zero	zero	n.a.	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
16.0%	29.5%	1.3%	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
84.0%	70.5%	1.3%	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
23.2%	21.6%	1.7%	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
14.4%	13.7%	0.3%	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
17.0%	16.8%	0.4%	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
5.9%	6.7%	0.2%	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
zero	zero	n.a.	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
29.7%	31.2%	0.7%	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
± 0.59%	± 0.72%	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0.9931	0.9983	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
179	243	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
> 99.9%	> 99.9%	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—

Position values calculated using EXCEL® solver. Stress estimated for average case (-100 ± 10 MPa) on surface, following Mognato et al. (2011). [A] in % of number of breakages in dataset. [B] in % of glass thickness. “n.a.” not applicable; “—” not defined. Applied equations refer to Kasper et al. (2018)

precision explained above. PEARSON's R^2 and STUDENT's t value are far better than with the simple fit, see Table 2. Besides the fact that the fitting curve nearly disappears behind the data points, all statistical characteristics indicate good fitting. However, in the logical sense, this is not a proof for the approach to be correct. Therefore, a reasonable physical cause has to be dedicated to each of the branches in order to explain why the total position's curve is so complex.

In the following sequence, the findings are first described. The description starts with the middle (i.e. parabolic) section of the curve. Then, the middle section is augmented into both directions. Finally, a possible interpretation is given.

- *Parabolic section (without fade-out)*

This section of the datasets comprises 61% ('HST-EU') and 49% ('Building') of the total number of breakages, respectively. Obviously, it is the dominant part in both curves.

A simple parabola is used for curve fitting; it is normalized onto the number of breakages contained in the parabolic section itself, i.e. neither including the 'Spike', nor the 'Fade-Out'. The only fitting parameter is its width 'a', without any trick. The good quality of this simple fit indicates that the correlation is probable.

Within error margin, a similar value for 'a' (16.8%/17.0%) is obtained in both datasets. It corresponds to a stress value of 33 MPa for both abscissa foot-points, i.e. the parabolas are similar for both the 'HST-EU' and 'Building' datasets. The reason for this is most probably that external conditions (mainly the temperature) do not change remarkably the stress in the glass. However, this does not mean that the breakage behavior in this dominating part of the curve does not change – quite the contrary is the case. Due to the difference in thermal expansion and other compositional effects of the inclusions and the thermo-mechanic effect of the HST procedure itself (see Kasper 2018), the curve must be stretched towards higher breakage rates in HST. This means that for a given (hypothetical) glass lot, the number of breakages in HST is higher in relation to the building situation, whilst, on the whole, the form of both position's curves is virtually similar.

- *Probabilistic 'Fade-Out' Tail*

The curve fade-out is, according to the list above, the 2nd GAUSSIAN, situated beyond the parabolic part. It needs two fitting parameters, namely (a) its half width

and (b) its midpoint. This part of the datasets comprises c. 20% of the total number of the breakages in both cases, and the fade-out is found in a stress region between +38 MPa and slightly negative pressure.

The latter means that the curve tail ingresses the compressive stress zone in both cases, but in the case of the 'HST-EU' it is a little more, namely –11 MPa instead of –5 MPa for 'Building'. Although this is statistically not forcedly significant, it goes into the logically expected direction and might then be a consequence of the influence of thermal expansion: Because at higher temperature a given inclusion is physically larger, it makes a longer initial crack and needs more compression to be stopped.

- *Merging point of probabilistic 'Fade-Out' tail with parabola*

The upper end of the probabilistic 'Fade-Out' is the visible beginning of the parabola, as discussed above. The corresponding stress values are calculated to be at 38.7 MPa ('HST-EU') and 37.6 MPa, respectively. Statistically, considering the mean variation obtained, they are not positively different. However, also in this evaluation their difference shifts into the logically expected direction: Lower stress for breakages in 'HST-EU', caused by e.g. the impact of the difference in thermal expansion. As mentioned above for the parabola, also here this similarity does not mean the HST having no significant increasing influence onto the number of breakages in a given glass lot; the contrary is logically true.

- *1st GAUSSIAN = 'Spike'/'Spike' length*

Visibly, this subset of the curves is very pointy in both cases. The author is convinced that it is not a measuring artifact. Summarizing both datasets, 66 out of 326 inclusions (20.2%) are located within $\pm 1\%$ of the midline—seemingly too many for a mistake, and a similar number as in the 'Fade-Out' tails. Therefore, the 'Spike' is obviously significant for the breakage behavior and cannot be ignored. Note that within this interval the tensile stress only increases and re-decreases by c. 0.1%.

Data measurement on the inclusions is easy—simply the dimension—and was carried out, during the time, by different persons, using three different measuring methods.⁵ It was done under different condi-

⁵ Stereo loupe, polarizing light microscopy and scanning electron microscopy

tions, namely, (a) in the case of the ‘HST-EU’ dataset, to c. 90% within a few weeks, using a factory collection of breakage butterflies, and (b) in the case of the ‘Building’ dataset, during c. 30 years, only making a small number or even one single examination at the same time. Another argument is that a significant part of the respective data is not situated exactly on the glass’ midline but at, e.g., 0.3%; 0.86% etc. besides it; these small differences also appear in the diagrams, above the parabola, and in Fig. 1. This shows that it is about values calculated from measured lengths and not simply estimation or stopgap for a forgotten measurement.

Thinking about a cause for this observation, an obvious statement is that it is the “symmetric case” of the breakages where the stress field is similar on both sides of the inclusions, just where the stress level is maximal in the glass section. Remarkably, the width of the ‘Spike’ is similar to the average of the inclusion’s diameters⁶ so that it is very pointy. A possible reason could be in the fact that the aligned stress (its vectors pointing parallel to the glass surface) interacts with the surface roughness of the inclusions, see Kasper (2018), and that in the “normal, dissymmetric” case, one side of an inclusion is subject to higher stress than the other one, whereas in the “symmetric case”, both sides feel nearly the same stress level, and that this fact makes the breakage probability increase in this narrow range. The “symmetric case” explanation is actually the best available for the ‘Spike’s existence; so far, it is simply phenomenology. Identifying its detailed physical reason is an important task for future fracture-mechanic simulation.

Comparison of the entire curves

In comparison, both datasets show only one substantial difference. In ‘HST-EU’, the relative ‘Spike’ length is significantly smaller than in ‘Building’, nearly only half. However, it is fact that more breakages occur in HST than on buildings, and this must be true for every single section of the curves, i.e. also for the ‘Spikes’. Consequently, they can be used as levelling poles, hypothesizing that on ‘Building’ the breakage probability in the ‘Spike’ curve section is at maximum the same as on ‘HST-EU’, i.e. this estimation is a minimum estimation for the relation in the breakage rates of both datasets. The calculation consists principally in stretching the y-axis of dataset “HST-EU” to an extent

that the ‘Spikes’ come to the same length. The stretching factor is then correlated with the relative number of breakages between ‘HST-EU’ and ‘Building’.

This hypothesis makes it possible to compare both datasets semi-quantitatively. Using respective figures from Table 3, a simple proportionality calculation reveals that in ‘HST-EU’ ($1 - 16\% / 29.5\% =$) 46% more breakages occur than on buildings. Because this is a minimum estimation, as explained above, it signifies that the HST makes all of the panes break that also (unsoaked) would cause breakage in building application. Additionally, roughly (more than) the same number of uncritical ones is again eliminated as “safety margin”.

This estimation does even not take the “missing fat” inclusions into account, see Chapter 2.2 below. Their position in the glass is unknown; however, it is logical to expect finding them preferentially within the ‘Spike’ because they are definitely the most critical. This is only true for the ‘Building’ dataset because the ‘HST-EU’ dataset is not truncated. Consequently, this effect makes the proportion estimated above increase even more. Hypothesizing that all of these lost inclusions were situated within the ‘Spike’ (this is certainly an over-estimation) leads to a maximum difference of ($1 - 16\% / 78\% =$) 79% more breakages, i.e. only 21% are necessary for safety because these inclusions would be dangerous in building application without HST, and nearly the fourfold number (more exactly: a factor of 3.76 of additional breakages) is then the “safety margin”.

It can, however, not be said that the factual safety margin is necessarily encountered between these two extremes. This estimate only takes the aspects of size and position into account. Other factors not yet accounted for here add even more safety margin. This point is discussed in more details in the conclusion of the present paper.

Fracture-mechanical approach into the observations The curves described above should forcedly be correlated to fracture-mechanics. Especially, the breakage probability should continuously increase with approach to the glass midline. Because the breakage probability is correlated with the derivative of any possible cumulative curve, the curvature of every section of the curve is therefore expected to be concave (if and when it reflects said probability), at the limit straight, but never convex. This is because the probability curve being convex means a physical impossibility, namely that the breakage probability would decrease. However,

⁶ Average glass thickness 9 mm $\rightarrow \pm 1\% = 180 \mu\text{m}$; average inclusion size c. 200 μm in both datasets.

Fig. 4 ‘Spiked helmet’ curve, abscissa transformed into stress coordinate. **A** Breakages ‘HST-EU’. **B** Breakages ‘Building’, un-soaked glass. **C** Principal devolution (sketch) of breakage probability, valid for both **A** and **B**. (a) No breakages in compressive zone. (b) (dotted) ‘Fade-Out’, cumulative GAUSSIAN with midpoint at crossing (b, c). (c) Parabolic section of (Fig. 3) transforms into straight line; trendline also drawn. (d) ‘Spike’, nearly like vertical straight

this is obviously the case in Fig. 3; therefore, something must be principally wrong.

The simple, but absolutely not self-understood explanation for this seeming fault is in the fact that not the position in the glass is determining breakage probability (naturally besides the intrinsic properties of the inclusion: size, chemical composition, roughness and the resulting orientation of the “initial crack”), but the stress field surrounding the nickel sulphide inclusion. Consequently, the diagram abscissa above has to be transformed from position scaling into stress scaling in order to correlate it to breakage probability. The result of this transformation is shown in Fig. 4A, B.

The advantage of this evaluation is that it correlates to fracture-mechanic principles.

- In the ‘Fade-Out’ curve part, increase according to a cumulative GAUSSIAN is still observed. In comparison with the former one (in Fig. 3), the new one should be the correct one, but due to data scattering a decision on which one is better cannot be made; in both cases the best fit is of sufficient quality in relation to the data scattering.
- As expected, the parabolic alignment transforms into a straight. Consequently, the breakage probability (= the derivative of the straight) is constant in this section of the curve, i.e. it does not depend on the surrounding stress.

- The ‘Spikes’ still appear; however, they look nearly like a vertical straight. This is due to the fact that the curves are the more compressed by the scale transformation the more they approach the midline. Tensile stress is nearly constant in the middle of the glass, whilst a high number of breakages is located within this curve part. The GAUSSIAN form of the ‘Spike’ is not visible anymore, contrariwise to Fig. 3.

However, the scale transformation has a significant drawback. In fact, there’s no other way than to presume that the stress parabola is “the same” for every breakage event, but this is, however, not true. According to Mognato et al. (2011) the parabola’s peak value is (50 ± 10) MPa, i.e. it varies by $\pm 20\%$ ($\pm 2 \cdot s$), and the same is proportionally true for every stress estimation except for zero. Therefore, the transformation amplifies the uncertainty in the dataset,⁷ and this is, additionally to the compression, another reason why the fitting is now more diffuse. This is the reason why both evaluations are reported; Fig. 3 is used for the estimation of the characteristic points in Table 3 because of higher precision. Contrariwise, Fig. 4 is relevant under

⁷ Under the theoretical statistical aspect, this should lead to heteroscedasticity of the data: The variance should increase with approach to the midline. However, this is at least not visible; it was accounted for in the value estimation in Fig. 4.

the breakage probability and physical explanation point of view.

As shown in Fig. 4C, it is possible now to conclude qualitatively onto breakage probabilities. Quantitative conclusion ($\pm 20\%$) is only possible for the abscissa values (stress), but not for the ordinate values (probabilities). This is because a probability is always a relation (number of observed events/total number of all possible events). In the present case, both breakage numbers are known, but the references are not, and they are in all likelihood different in both cases.⁸ Consequently, the absolute levels remain unknown; Fig. 4C can only show the principal devolution, applicable (but not quantifiable, even not relatively) on both datasets. The relation of the breakage probability in ‘HST-EU’ and ‘Building’ is, however, significantly different and, as already proven, higher in HST. This relation is a priory unknown; however, it leads forcedly to a multiplication of the effect described in Chapter 2.1.2.1.

This fracture-mechanical approach allows interesting assessment of the data. Referring to Fig. 4C, the following argumentation⁹ is a probable interpretation.

In section (a)

The primary flaw size does not matter: Due to the surrounding compressive stress field, possibly growing primary cracks are getting orientated parallel to the glass surface (examples in Kasper 2018). Compressive stress stops any crack propagation into perpendicular direction, but only the latter could lead to glass failure.

⁸ If the real total number of nickel sulphide inclusions existing in both glass lots would be known, a real breakage probability could be given for each of the cases. For relativization, the reference could be the tonnage of the toughened glass lots where the breakages are observed in; division would then give an approximate relation between both cases. However, these were not recorded. An estimate is in fact available for the average breakage rate in HST (c. 1 breakage in 10 tons tested), but not for un-tested glass; the evidence that both must be different is one of the main themes of the present publication series.

⁹ This data assessment should be the subject to further fracture-mechanical calculation; to facilitate this, the author invites any competent institute to use the raw datasets published herewith. To the author's knowledge, respective actual calculative approaches are oversimplified under significant aspects and do not take into account the different sectioning observed and qualitatively interpreted here. Simplifications in current calculation approaches seem to be e.g.: Non-consideration of inclusion's rough surface; primary flaw formation assumed for every nickel sulphide inclusion's size, even for the very small ones; non-consideration of compositional impact of NiS_x ; non-consideration of additional external (e.g. thermo-mechanic; wind load) forces and/or fatigue impact by water diffusion.

Close to stress neutrality some (“very critical”) inclusions cause seldom breakage. Logically, this occurs more frequently in HST.

In section (b)

The breakage probability increases stochastically with increasing tensile stress.

Note that in this case, for probability, the correlating curve is a derivative 1/2-GAUSSIAN.

Between stress neutrality and the critical stress level of c. 38 MPa, size and orientation of the primary flaw (caused by the properties of the individual nickel sulphide inclusion and the surrounding stress field) play the key role, deciding on if or if not the situation becomes critical.

Small primary flaws¹⁰ do not forcedly lead to breakage, but large ones do. The surrounding stress and, in relation, the very direction of the flaw [see Kasper 2018 for explanation] determine what is “small” or “large”.

In HST, thermo-mechanical forces play a much more significant role, making also (in this sense) “smaller” flaws become critical.

In section (c)

Starting with the critical stress level of > 38 MPa, the breakage probability turns to constancy.

The tensile stress is high enough to make every critical primary flaw extend to “infinite”, i.e. to cause breakage.

In HST, thermal expansion, stronger thermo-mechanical forces and, possibly, sub-critical crack growth cause formation of primary flaws on a higher number of (even smaller) inclusions, in interaction with the notched surface of the inclusions.

In section (d)

At a stress level of (50 ± 10) MPa, very close to the glass' midline in a range that reflects the size of the nickel sulphide inclusions but by far not the increase (c. $+0.1\%$) and the variety ($\pm 20\%$) of the tensile stress

¹⁰ More precisely: Physically, interpreted as a vector, a primary flaw pointing into radial direction with reference to the coordinate system of the inclusion is (normally) a skew vector with reference to the coordinate system of the glass. In the relative parallelogram of forces only the partial force orientated parallel to the glass surface (and thereby causing crack opening) plays a role. For more details, see (Kasper 2018), impact of the roughness of the nickel sulphide inclusions.

Additionally, the first crack (at the weakest point of the inclusion, i.e. the greatest notch) leads to stress release around the inclusion due to the correlated physical expansion of the “bubble” so that a second crack into another direction cannot generate. The latter is, naturally, true for any of the sections a - d discussed here.

level within this range, the breakage probability jumps up. In the sketch, its width is exaggerated figuratively.

Possible causes have already been discussed above.

Summary and conclusions on the position's aspect

In short, the author's interpretation of these findings on the nickel sulphide inclusion's positions in breakage departure points is the following. Maybe it is not yet correct in every detail, mainly concerning the 'Spike'; therefore, it should rather be looked at to be a discussion contribution than "pure truth". More precise fracture-mechanical calculation to be carried out independently will certainly lead to deeper understanding or (also this shall not be excluded here) to falsification of some of the aspects discussed here. However, due to the partly surprising findings, it seems that the mechanism for spontaneous breakages is not yet understood in every significant detail and needs additional theoretical effort.

On the basis of the observations, both datasets are divided into four subsets.

- a) Roughly in the compressive zone of the toughened glass, nickel sulphide inclusions do never cause "normal" spontaneous breakage (i.e. breakage due to their α to β transformation).
- b) In the 'Fade-Out' region of both data subsets, the breakage probability increases according to a probabilistic law due to increasing tensile stress.
- c) Above a threshold value of c. +38 MPa (i.e. 76% of its maximum) the breakage probability stabilizes although the tensile stress still continues to increase with approach to the midline.
- d) In the 'Spike' subset, i.e. in a narrow range in the very middle of the glass, an unknown additional effect leads to sudden strong increase of the breakage probability.

Applying the probabilistic calculations explained above leads to the conclusion that minimum¹¹ 46%, but possibly more than 79% of the breakages observed in 'HST-EU' are "irrelevant", i.e. they do not occur in (un-soaked) building application.

Reversing this argument: The HST makes all panes break that are critical on building; additionally, the same number again, but possibly even up to three times more panes shatter as safety margin.

¹¹ "Minimum" means that in the estimation side effects like the truncation of the 'Building' dataset are not included, and that the standard deviation observed is used to make a worst case calculation.

2.2 Size of analyzed nickel sulphide inclusions

Like in the C/K trial (see Kasper et al. 2018), 'HST-EU' and 'Building' datasets can be fitted by aid of the Log-Normal function. Every inclusion in both datasets has caused glass breakage and was therefore a critical one, in contrast to the dataset "All Inclusions" in the 'C/K trial' where the number of uncritical inclusions preponderates. However, criticality has a different meaning in HST and in building application, respectively. The present chapter delivers another proof for this fact.

2.2.1 Evaluation of the breakage data using Log-Normal curves

The following figures are obtained if the evaluation method mentioned is applied; the referring histograms are shown in Fig. 8.

Figure 5 shows the cumulative size alignment of the collected data including their fit with aid of the two-parametric (i.e. not x-shifted) Log-Normal functions under multiplication with the maximum number in each dataset. The curves were obtained applying the least-squares method. The statistical key values [PEARSON's R^2 and STUDENT's t] derived thereof according to Eq. (1c)/(1d) in Kasper et al. (2018) reveals that this fitting leads to pretty good results; the fits match the datasets in a way that does not seem to be amendable.

Figure 6 compares the fitting curves obtained. As will be discussed below, significant truncation correction has to be carried out on the 'Building' curve in order to obtain comparability.

Concerning the definition of the "take-off" point (see also Kasper et al. 2018): Log-Normal curves normally show a sector at low values where the function values are close to zero (in the examples: c. < 40 μm / < 70 μm), and thereafter (at the "take-off") it raises visibly and rather steeply. Adopting the $\pm 3\sigma$ criterion of the GAUSSIAN (99.7% of the members of a dataset are located within this interval; 0.15% are lower, 0.15% higher), the probability (of occurrence, of breakage etc.) of 0.15% is defined arbitrarily by the author to be the take-off point of the curve. It calculates using the best-fit Log-normal curve of a given dataset, assigning a characteristic inclusion size x to said function value of $y = 0.15\%$.

Observations from Figs. 5 and 6:

- The forms of the curves differ significantly.

Fig. 5 Size alignment of identified nickel sulphide inclusions. **A:** Breakages from HST furnaces ('HST-EU'), collected in different factories. **B:** Breakages from Buildings ('Building'), un-soaked glass, collected from client claims and consultants asking for analytical expertise in the SG laboratories. (a) Scale-adjusted cumulative (LOG-NORMAL function based) curve, from least

square fit in **A:** $R^2 = 0.9964$; $t = 248$ ($> 99.9\%$ probability against hypothesis $R^2 = 0$) in **B:** $R^2 = 0.9969$; $t = 179$ ($> 99.9\%$ probability against hypothesis $R^2 = 0$). (b) Derivative (Log-Normal function based) curve, parameters from (a), arbitrary units. Error bars: $\pm 3 * s$ from least square fitting

Fig. 6 Comparison of fitting curves of datasets 'HST-EU' and 'Building'. **A:** Cumulative (LOG-NORMAL function based) curves, from least square fit. **B:** Derivative (LOG-NORMAL function based) curves, area = 1 for both. (a) Breakages 'HST-EU'. (b) Breakages 'Building', un-soaked glass. Take-Off points: (a) 43 μm ; (b) 68 μm (difference 25 μm)

- Breakages in HST "take off" at much lower diameters.

Already in 1981, Swain (1981) published that (literal citation) "NiS inclusions that had spontaneously fractured revealed that fracture is generally initiated from particles greater than 110 μm in diameter located in the tensile stress field in the middle of the glass plate. Inclusions within the range 80–110 μm are sometimes responsible for spontaneous fracture, whereas those inclusions of diameter less than 80 μm are not dangerous." In 1981 the HST was not yet invented; consequently, every inclusion SWAIN examined was coming from a building breakage.

This observation is in good correlation with the actual findings on buildings described in the present paper. The "Take-Off Point" of curve Fig. 7a is 68 μm .

Due to its definition (see here-above), this criterion is very strict and leads, therefore, to a lower estimation than SWAIN's observation. Contrariwise, the analogous estimation for curve Fig. 7b yields 43 μm , and the t-test reveals that the difference between both is to $> 99.9\%$ significant.

- Breakages in HST obviously contain significantly more coarse members

(visible by the more flat curve slope [curve B] at $c. > 300 \mu\text{m}$).

This third observation needs some discussion and leads to the necessity of statistical correction before more detailed data evaluation. According to the findings discussed in Kasper et al. (2018), breakages caused by coarser nickel sulphide (and other) inclusions should

Fig. 7 Comparison of Log-Normal curves of datasets ‘HST-EU’ and ‘Building’. **A**: Cumulative (LOG-NORMAL function based) curves, from least square fits. **B**: Derivatives of curves in (A) calculated using the parameters σ , μ from A. This observation: Compression of (B) to obtain $a = b$ in tail of curves, $n'_{\text{kor}} = 0.77$. **D**: Cumulative curves calculated using the parameters σ , μ from C. (a): Breakages ‘HST-EU’, original; reference curve form. (b): in A and B, (b) = (a), shifted with $x = 26 \mu\text{m}$

to adjust with basis of (c) in C and This observation, (b) merged with tail of (a) because breakage probability in HST must always and everywhere be higher than on building. c: Breakages ‘Building’, un-soaked glass, in A and B, compressed in y direction with $m_{\text{kor}} = 0.66$ to fit with (b), undoing the truncation. in C and D, compressed with $m'_{\text{kor}} = 0.51$. Difference between b and c (cumulative): truncation loss

occur nearly independently of the conditions, i.e. it should be equal on buildings and in HST, and in both cases happen with high probability. But obviously, as visible in Fig. 5, many of the coarse inclusions are missing in the ‘Building’ dataset. The reverse hypothesis, namely that the coarse ones cause breakage in HST only, is absolutely not logical and has therefore to be discarded.

The most probable explanation is that the coarse inclusions have caused “undiscovered” breakages during the time between production and installation. – The “normal” spontaneous breakages on buildings caused by nickel sulphide inclusions show an incubation period of (normally) more than 1 year whilst the inclusions have already started transforming, but they did not yet fill up entirely the hollow space around them.

This hollow space is known to be due to the different thermal contraction of glass and NiS during glass production. Consequently, stress is not yet induced into the glass during this period; premature breakage is only possible because the “NiS bubble” constitutes a weak point in the glass, and this is most probably because it is “large” (estimated: $> 300 \mu\text{m}$) and, at the same time, “notched”, and because the stress concentration factor around such a bubble is more than threefold, see Kasper (2018) for more details.

There’s also another point to be mentioned: Yet larger nickel sulphide inclusions (estimated: $> 500 \mu\text{m}$, but depending on glass thickness) can already cause breakage during the toughening process. This means that most of these “over-critical” inclusions, i.e. the “very large” ones, neither will arrive in the HST nor

on a building. This effect limits the observable size of the inclusions and is another reason (in addition to settling due to gravity, see Kasper 2018) for the relatively small and highly variable number of large inclusions generally found. It can also be the reason for the observation that the breakage rate in HST (i.e. the breakage number per ton) increases significantly with increasing glass thickness. “Very large” has a different meaning in a thin glass plate of e.g. 6 mm than in a thick one of e.g. 15 mm thickness. Said decrease is indeed observed in a significant extent, see Kasper et al. (2018).

Because, in summary, the large nickel sulphide inclusions are able to cause premature breakage without α to β transformation and, if they are transformed, they are for certain the most critical ones, probable reasons for only finding a small number of large nickel sulphide inclusions in breakages of un-soaked glass in ‘Building’ are:

- A part of the glass panes containing a large nickel sulphide inclusion already fails during its stay on the rack in the factory or during transport to the building site, due to additional transport-related agitation/jerk.
The panes are replaced without reflecting—glass is, naturally, brittle, so that this kind of breakage is looked at to be normal loss.
- Another part breaks shortly after installation. On a building site, nobody relates this to a nickel sulphide problem. Cullet is mopped up and the pane is replaced.
- If a problem with spontaneous breakages unveils a year or two after completion of the building, the first numbers of the breakage series will not be identified as such.¹² Again, cullet is mopped up and thrown away. It is most probable that the first, say, three breakages of a nickel sulphide inclusion epidemic are not realized as such, or at least the respective inclusions are lost and never find their way to a laboratory. Also in this case, the most dangerous (i.e. the largest, at the same time maybe situated close to the midline of the glass) nickel sulphide inclusions are the most suspicious to be lost.
If, after these first lost breakages, the case starts to be more obvious and a nickel sulphide problem is suspected, in most cases the panes of the build-

ing concerned are secured with an adhesive foil. If, thereafter, more panes shatter, the breakage centers can easily be collected for analytical clarification.

This allows two important qualitative conclusions to be taken into account in the following quantifications and discussion.

1. The explanation for the missing coarse nickel sulphide inclusions means that the most critical “largest” inclusions are the first to cause breakage. This has also to be true regarding the breakages in HST, see Fig. 10 and Chapter 3.
2. The ‘Building’ curve is, in the statistical sense, truncated.

The following evaluation has to take into account the fact that the largest inclusions are missing in the present ‘Building’ collection. Under a theoretical aspect, this means that the ‘HST-EU’ curve is complete, also because breakages between toughening and HST are not reported; contrariwise, a part of the population is missing in the ‘Building’ dataset. For certain, this concerns the coarsest inclusions, but *a priori* there’s no clear limit between “the coarsest” and “the others. However, truncation-undoing identifies where this overlaying curve of “the coarsest” starts to be relevant, and how cardinal it is.

2.2.2 Approach into undoing the truncation of the ‘Building’ curve

A first and simple approach to solve the truncation problem of the ‘Building’ dataset is hypothesizing that both complete (un-truncated) curves are similar in the geometrical sense. The thinking behind this is that the size effects leading to breakage on building and in HST would be similar, and that both the original ‘HST-EU’ and the reconstructed ‘Building’ curves can then be made overlaying “exactly” by simple x-shift, i.e. they are parallel. Furthermore, hypothesizing that “the smallest” are unaffected by truncation means that said x-shift merges the bases of both curves.

However, logically, the hypothesis of parallel curves is the absolute minimum condition. This argument is resumed below for continuative argumentation.

Consequently, in this first and most simple approach, the first step of truncation-undoing is basis (i.e. “take-off point”) adjustment of the cumulatives, Fig. 7A. The shift value is found to be 26 μm , minimizing the

¹² At least it was like that during the collection time of the samples in dataset ‘Building’. However, today, sensitivity has increased significantly.

curve's differences between 55 μm and their bifurcation. Superposition is nearly perfect in this lower section of the curves. The bifurcation point is found at $x \approx 200 \mu\text{m}$; m_{kor} gives an idea of the relative number of undiscovered breakages on buildings, 34%. This seems to be high; however, it looks reasonable taking into account the arguments above and, additionally, those discussed in Kasper (2018).

In the second step, Fig. 7B, the linked derivatives are drawn. This transformation reveals that the 'Building' derivative curve is partly situated above 'HST-EU'. Generally, the derivative curves represent the number of breakages per size interval (like in the histograms: $\Delta y/\Delta x$, but this time with "infinitesimal" intervals dy/dx). Applied on the present case, the 'HST-EU' derivative curve has always to be above (minimum: exactly merging) because, due to physical reasons, the breakage probability in 'HST-EU' is always higher. Consequently, the derivative (b) in B is multiplied with a factor < 1 (i.e. compressed) so that the tails of both curves coincide.

This operation yields Fig. 7C. The compressing factor $m_{\text{kor}} = 0.77$ is applied onto the 'Building' derivative in order to merge the tails. The calculations were carried out using EXCEL®-Solver and least-square fitting. By the way, the truncation loss remains the same, 34%, in this operation.

Note that this is an absolute minimum correction because the breakage probability in HST might be far higher but in no case lower, even in the range of big inclusions; just the logical minimum condition is applied. In the last step, Fig. 7D, the cumulatives are simply reconstructed from the parameters of their derivatives, Fig. 7C. Their y values at 600 μm are at the same time (in good approximation) the surfaces of the linked derivatives. Consequently, 34% of the breakage departure points in 'Building' are lost and minimum 23% of the breakages in 'HST-EU' cannot occur in (reconstructed) 'Building'. As already mentioned, the latter is most probably much higher, but the proportion is unknown.

This evaluation gives also an idea on the minimum size of nickel sulphide inclusions causing breakage under the different conditions. In correlation with experimental findings (Fig. 5), the breakage probability is aligned with the size of the smallest nickel sulphide inclusion ever recorded in our laboratory in 'HST-EU', namely 54 μm . The relative occurrence rate, calculated

from Fig. 7, curve (a), is then 0.61%,¹³ i.e. in average one out of 164 inclusions of this size causes breakage. Applying the same rate of 0.61% onto curve (b) yields in a diameter of 76 μm . For control, application onto the original (truncated) dataset, Fig. 5B, yields in a diameter of 78 μm , i.e. virtually the same value; obviously, truncation-undoing does not affect this section of the curve significantly. Finally, in 'Building', the smallest inclusion recorded in practice has 75 μm in best correlation to these statistical estimations.

Simplifying, only to illustrate the magnitude of the effect observed, a nickel sulphide inclusion of diameter 200 μm and NiS(1:1)-composition shall be discussed as an example for this surprising finding. In both cases, it has caused a breakage and, therefore, consists already of the β phase. Due to the x-shift discussed above its "property difference" between ambient and HST condition is said 26 μm . However, this cannot be due to thermal expansion alone: $x_{\text{expansion}} \approx 1.004$ calculates from the known thermal expansivity of NiS ($16 \cdot 10^{-6} / \text{K}$) and the temperature difference ($\approx 240^\circ$). This means that in the HST, the example inclusion expands to a diameter of 200.8 μm . Obviously, thermal dilatation explains only $(0.8 \mu\text{m} / 26 \mu\text{m}) \approx 3\%$ of the difference factually observed and is therefore nearly negligible. Even the size increase due to α to β transformation would be unable to explain the difference if it would not already have taken place. Note that this effect is said to be the most significant breakage cause, blowing the example inclusion up to maximum 206 μm and, thereby, making it figuratively responsible for only $(6 \mu\text{m} / 26 \mu\text{m}) \approx 23\%$ of the difference observed. Consequently, additional forces and effects must be the preponderating cause for the difference observed, and with regard to the small influence of thermal expansivity, these are responsible for 97% of the difference. Among others, transient thermo-mechanical forces and, possibly, sub-critical crack growth in the HST, but not on buildings, have been identified for this, as discussed extensively in the previous part papers of our small series, Kasper (2018) and Kasper et al. (2018). Also the present evaluation reveals that the special impacts of the HST are absolutely not negligible.

¹³ i.e. the occurrence rate is four times higher than that of the "Take Off" defined above, but, due to the steepness of the curve, this effect onto the inclusion's diameter is only small with $c. \Delta x = 6 \mu\text{m}$.

Fig. 8 Comparison of Log-Normal curves of datasets ‘C/K Trial’, ‘HST-EU’ and ‘Building’, from least square fit. **A** Cumulative (LOG-NORMAL function based) curves, including curve (b) from Fig. 7 = curve (a) x-shifted with constant $\Delta x = -26 \mu\text{m}$; $m_{\text{korr}} = 0.66$ applied to obtain curve (c). **B** Curve (a) x-shifted with size-dependent parameter [$\Delta x = (-17.3 - 0.18 \cdot \text{size}) \mu\text{m}$]; $m_{\text{korr}} = 0.45$ applied to obtain curve (c). **C**: Derivative curves with reference to (B). **D**: Compression of (C) to get $a = b$ in

tail of curves, $n_{\text{korr}} = 0.32$ applied. (a): Breakages ‘HST-EU’, original curve. b = (a), but shifted to adjust with basis of (b) (different between **A** and **B**, see above). (c): Breakages ‘Building’, un-soaked glass, compressed in y direction with m_{korr} to fit with (b), for undoing truncation (m_{korr} different in **A** and **B**, see figures in diagrams). (d): Breakages from ‘HST-C/K’ (see Kasper et al. 2018, original curve

The huge difference in the devolution of the size curves identified here is absolutely significant; but to the author’s knowledge such has never been accounted for in fracture-mechanics modelling.

2.2.3 Further estimation and comparison of breakage probabilities from size spectra, including the results of the ‘C/K trial’s result

In the first approach above, the ‘HST-EU’ and ‘Building’ curves are only aligned. In the following second step the results of Kasper et al. (2018) are integrated into the evaluation.

In Fig. 8A, both (reconstructed) cumulative curves are drawn again, normalized to the unity. However, this is still not fully logical. As already discussed above, this condition is the absolute minimum for the differ-

ence estimation because, among others, it disregards the “lever rule” referring to the length of the primary crack. The basic curves are assumed only to show a constant offset of $26 \mu\text{m}$. The physical expression of this is that nickel sulphide inclusions behave in ‘Building’ “as if their diameter (necessary to cause breakage) was increased by $\Delta x = 26 \mu\text{m}$ ” in relation to the ‘HST-EU’.

However, if effects similar to thermal expansion are accounted for the difference, its condition is proportionality. The physical expression for the latter is that “their diameter (necessary to cause breakage) increases by a factor $x_{\text{expansion}} > 1$ ”. Logically, the effect observed is not (only) related to x-shift but is (at least partly) proportional to the nickel sulphide inclusion’s size. The restriction in brackets refers to the fact that the HST also adds breakage causes based on the variabil-

ity of the chemical composition of the inclusions (the latter are irrelevant for ‘Building’) and, relatedly, different expansion coefficients of the additional species (Ni_9S_8 , Ni_3S_4) as discussed in Kasper (2018).

To take this into account, the C/K-Trial’s results are integrated into the following argumentation, especially those of the HST carried out within this frame (‘HST-C/K’). Figure 8A curve (d) (taken from Kasper et al. (2018), Fig. 7B) integrates the latter’s result into Fig. 7.

Surprisingly at first sight, the bases of ‘HST-C/K’ and ‘Building’ match nearly exactly. Above the basis region the truncation-undone curve (b) is situated between the ‘HST-EU’ and the ‘HST-C/K’ curves. But the explanation of this observation was already discussed in Kasper (2018). The glass panes’ sizes used in the C/K trial are “thin and small”. Exclusively breakages due to nickel sulphide inclusions are observed therein although also other inclusions of sufficient criticality had been identified, able to lead to breakages in analogy to the findings in Kasper (2018). Consequently, the dataset ‘HST-C/K’ cannot be matched directly to the “normal” ‘HST-EU’ curve.

Putting it simple reveals the following list of significant differences and similarities.

- *Differences between ‘HST-C/K’ and ‘Building’*
  - Temperature ($\Delta T \approx 200^\circ$)
    - impact of dilatation and chemical composition; sub-critical crack growth, if relevant;
    - average size, thickness, color;
- *Similarities*
  - In both, no significant temperature gradients over glass surface
    - nearly no thermo-mechanical stress.
- *Differences between ‘HST-EU’ and ‘Building’*
  - Temperature ($\Delta T \approx 200^\circ\text{C}$)
    - impact of dilatation and chemical composition [sub-critical crack growth, if really relevant];
    - Temperature gradients over glass surface
      - strong thermo-mechanical stress only in ‘HST-EU’
- *Similarities*
  - Average size, thickness, color

Obviously, the “significant difference” is in the thermo-mechanical stress that only appears in the ‘HST-EU’, but neither in ‘Building’ nor in ‘HST-C/K’. Chapter 2.3 in Kasper (2018) and Chapter 5.2 in Kasper et al. (2018) reveal that this additional stress is a strong impact factor in ‘HST-EU’, but it is negligible for the ‘HST-C/K’. Conversely, the hypothesized influence of sub-critical crack growth is most probably of minor influence because its impact cannot depend on the glass panes’ size, but, if observable, only on temperature.

The argumentation in the previous chapter reveals that the impact of thermal dilatation is nearly negligible in relation to the effects observed – only 3% of the impact is explained thereof. Taking this serious, and combining it with the result of Kasper (2018) Chapter 2.2.2, means that $> 98\%$ of the breakages in HST are irrelevant for building application.

Reversing this argument: The HST makes all panes break that are critical on building, and additionally, as a safety margin, 49 times more panes; the latter are only critical in HST, but not on building.

Therefore, the result of the ‘HST-C/K’ is used as a reference for the trend in ‘Building’; eventually, a small negligible difference (-3%) has to be expected due to the additional thermal expansion effect in ‘HST-C/K’. Again, the result must be looked at to be “the upper limit” of the size impact as already mentioned in relation to Fig. 7. So, Fig. 8B–D show the parameters and the result of this calculation. The following parameters apply:

- $m_{\text{kor}} = 0.45$ onto the original ‘Building’ curve (→ c), but no x-shift, in the first step.
- size-dependent x-shift (formula in diagram legend) onto the ‘HST-EU’ curve (original curve a), adjusting it onto curve (d), and thereby taking into account part proportionality for adding the effect of the “lever rule” → curve (b)
- No correction at all onto the ‘HST-CK’ curve (d); it serves as the reference.

This brings/keeps the basis of curves (b), (c) and (d) at the same place, and they show nearly identical devolution for $x < 200 \mu\text{m}$. The bifurcation point between curves (c) and (d) remains at c. $200 \mu\text{m}$.

Admittedly, although they base on facts and calculations obtained independently, the calculations related to Figs. 7 and 8 are in detail somewhat speculative because they only base on one set of data for each condition. Therefore, in order to not to over-stress them,

Fig. 9 Comparison of sizes in datasets “C/K trial” ‘HST-EU’ and ‘Building’. Histograms, classifying every dataset into classes of 50 µm. (A) Basic size spectrum, from C/K trial, 140 inclusions

in total. (B) Size spectrum identified in ‘HST-EU’ dataset, 212 inclusions in total. (C) Size spectrum identified in ‘Building’ dataset, 101 inclusions in total; truncated

the explicit figures (such as m_{kor}) should not be taken too precisely, possibly not better than $\pm 10\%$. However, they reveal that the breakage rate in ‘HST-EU’ is much higher than in ‘Building’, and thus, they allow to make a serious minimum estimation.

In analogy to the argumentation in relation to Fig. 7, the deduction of Fig. 8 is that (only under the aspect of the inclusion’s sizes) the percentage of irrelevant breakages in ‘HST-EU’ is 68% and 86%, respectively. This estimation is higher than the one made from the positions (Chapter 2.1.2). But in the latter, undoing truncation is not possible; this is another reason for this estimation to be a minimum estimation leading to the conclusion that the factual difference between HST and building application is most probably larger than observed here.

2.2.4 Figurative comparison of size datasets

Figure 9 compares the histograms of size spectra of nickel sulphide inclusions identified in annealed glass in the ‘C/K trial’ with ‘HST-EU’ and ‘Buildings’. In this figurative comparison an interesting significant development of the smaller sizes becomes visible. The results from ‘HST-C/K’ are not accounted for here because of their special properties discussed above, and because their member’s number is small and statistically not exploitable here. Sizes $> 200 \mu\text{m}$ are not discussed in view of the truncation problem revealed in Chapter 2.2.3. However, this knowledge is used to fix the reference group, see below.

The comparison of these datasets (on the side of smaller size) shows figuratively what happens during the glass processing and if the glass is, or is not, subject

to a HST. It also makes the calculations above (Chapter 2.2) less abstract, explaining them in a more intuitional way.

The smallest class ($< 50 \mu\text{m}$) is generally disregarded. Admittedly these inclusions cannot lead to breakages under any condition; consequently, whatever their number factually is, they are irrelevant.

The 4th class (150 µm to 200 µm) of all histograms is used as reference. According to the calculation and estimation results above this class is most probably not yet subject to truncation.

- In the basic size spectrum (‘C/K trial’), the highest number of the inclusions is found in the 2nd class (50 µm to 100 µm). Also 3rd class members are more numerous than those of the 4th.

In reference to 4th class, 2nd + 3rd classes contain

the (3.04)-fold number.

- In ‘HST-EU’, the relation has obviously changed.

The 2nd class only contains half of the members of the 3rd.

In reference to 4th class, 2nd + 3rd classes now only contain

the (1.72)-fold number.

- In ‘Building’, the relation has changed again.

The 2nd class is practically empty; the 3rd contains c. less than the 4th.

In reference to 4th class, 2nd + 3rd classes now only contain

the (0.74)-fold number.

- The ‘HST-CK’ result is, in the frame of the present evaluation modus and as already mentioned, statistically not significant, and the respective histogram is therefore not shown here (but see Fig. 6 in Kasper (2018)). To be complete, 2nd + 3rd classes therein contain 3 members and the 4th class 6 members, i.e. their respective relation is (0.5)-fold. Consequently, it is not far away from ‘Building’, but statistical significance is low.

This means that the HST sorts out larger-sized inclusions from the basic size spectrum, i.e. the smallest lead with lower rate (probability) to breakage. Contrariwise, the inclusions must be (in average) significantly larger to cause breakages on buildings. The difference is clear, figuring the relations 12 : 7 : 3 (: 2), in relation to the 4th class.

Taking this as an indication leads to the estimation that at minimum ($1 - 0.74/1.72 =$) 57% of the breakages in HST do not occur (un-soaked) on building, in best correlation with the estimations obtained in Kasper (2018) and up to here in the present paper. Also the present estimation turns out to be a minimum one because it presumes the breakage rate in the 4th class to be similar in both datasets whereas, in reality, the one in ‘HST-EU’ is significantly higher as already discussed earlier.

Obviously there’s a strong impact of the size (chapter 2.2), but also the position (chapter 2.1) seems to play a significant role in the difference between conditions in HST and on buildings. However, it should not be forgotten that chemical composition plays also a certain role (see Kasper (2018), Chapter 2.2.2). The latter is hidden in the datasets of the present paper because they result from factual breakage events.

Future modeling calculations by independent institutes will lead to a better approach, taking into account all impact factors simultaneously.

3 Projection onto the real case

3.1 Relation to previously published data

In 2000 the author has published (Kasper 2000) data from five consecutive years, showing the times to breakage in HST’s carried out in Europe. This dataset, containing 1462 times to breakage, became the main source for the constitution of the international HST Standard EN 14179-1. The sigmoid curve in Fig. 10 was

Fig. 10 Application of new findings onto data published in Kasper (2000). Total number of breakages recorded: 1462. Among these, number during holding time: 290 (= 19.8%). A: Breakages during early heating-up are “strictly necessary”. B: Breakages during middle heating-up are “possibly necessary”. C: Breakages during late heating-up are “most probably irrelevant”. D: Breakages during holding time of HST; “irrelevant”. (a) Limit of 33% of breakages. (b) Limit of 67% of breakages. (c) Beginning of holding phase (every pane > 280°C); 80%

published and precisely fitted by one single WEIBULL curve.

In Fig. 10, only 290 among the breakages (19.8%) are observed during holding time. Note that the following projection is strictly phenomenological and subject to statistical scattering. Size, position, chemical composition and the thermo-mechanical forces induced in the HST (see Kasper 2018 and discussions above) have a probabilistic influence, but not at all the same in HST and in building application. Generally, but a little simplifying,¹⁴ the sequence of the breakages in HST essentially reflects their (naturally hypothetical) sequence in building application, i.e., early breakage in HST essentially signifies early breakage and highest criticality of the respective inclusion in un-soaked glass. The later the glass breakage is observed, the less critical was the inclusion in HST, and so would it have been on the buildings. This matching goes up to a threshold range where the breakages in HST do not have counterparts on building any more. Beyond, they do not cause breakages on a building. These breakages in HST (the last ones) are classifiable “needless”. This hypothesis does not fully count for the breakages during actual (admit-

¹⁴ The HST adds a random element since the thermo-mechanical stress built-up does not fully follow this logic.

tedly too high) holding temperature; the latter need a closer look, see below.

According to the results obtained in the previous chapters, the percentiles of breakages are grouped into four sections as shown in Fig. 10. For sure it is not black-and-white like this because of mentioned statistical scattering [see also discussion on (section C) and (section D) below], but truth cannot be principally different. Naturally the precision of the numbers should not be overrated; the four sections should be looked at to be figurative, chosen to explain different impact factors.

The data in section (A) of Fig. 10 are recorded early in the heating-up phase of the HST. Denoting these breakages as “early” means that the respective inclusions lead easily to breakage. According to what was discussed in Kasper (2018), this group of inclusions must predominantly contain crystallites of pure (fast transforming) NiS, possibly mixed with crystals of (indifferent) Ni₉S₈, and/or be large and/or be situated in the high risk middle zone of the glass. The difference in their time to failure consists in small deviations of composition (possibly traces of iron or other foreign elements), individual position (= surrounding tensile stress, including the stress induced by heating-up) and size.

It is clear that this NiS population is also dangerous in building application. Therefore, these breakages in HST are qualified “strictly necessary”.

The data in section (B) are recorded in the middle of the heating-up phase. Regarding their α to β transformation, most among them have already turned over into the β phase, see [Kasper 2018, Fig. 17], no further transformation will occur in this majority. According to the values of “irrelevant breakages” above, they are possibly critical regarding breakages on buildings. Consequently, this group of breakages is qualified “possibly necessary and in any case adding value for good product safety”.

The data in section (C) are recorded during the late heating-up phase. According to the values of “irrelevant breakages” above, they are almost probably not critical at all, regarding breakages on buildings. Regarding the process time, it is about the last c. 35 minutes of the heating-up period. Consequently, this group of breakages is qualified “most probably irrelevant and probably not adding value for product safety”.

The data in section (D) are recorded at temperatures $> 280^\circ\text{C}$. According to Yousfi et al. (2007) this tem-

perature is too high. Under this physical condition, a part of the inclusions (namely those with some overstoichiometry, NiS_x with $x > 1.012$) is unable to reach entire α to β transformation; these will therefore show post-transformation and possibly cause spontaneous breakages at ambient temperature. In this curve section, the assumption that the criticality of the inclusions continues to decrease is not fully true anymore because temperature is too high. However, under the new conditions of the HST with reduced holding temperature level, the inclusions formerly not transforming entirely in section (D) will now be enabled to transform.

On the other hand, Chapter 2.3 in Kasper (2018) (HST breakages by stones) reveals that the breakages in this section of the curve are mainly caused by thermal stress or also, possibly, fatigue under the impact of water diffusion. In relation to this, the slow transformation of some single nickel sulphide inclusions with exotic composition is most probably only an inferior breakage cause. It is therefore most probable that this part of the HST is not needed at all for sufficient safety of HST-glass.

However, just exactly the invalid data from section (D) of Fig. 10 have been used and are crucial for the present extrapolative long-term safety prediction, independently on how this calculation was done in detail by the authors.

Both serious safety estimations published up to date KASPER and Serruys (2002) and Schneider et al. (2012) must therefore be revised. Both hypothesize the breakage behavior in HST and on buildings to be the same, i.e. that there is no systematic offset due to the conditions. Therefore, the resulting safety calculation under-estimates the factual safety by far, or, in other words, a lot of glass is destroyed in the HST ovens all over the world without any factual impact onto product safety.

Strictly speaking, nobody disposes actually of a dataset representing the new conditions of the HST. Therefore, the necessity to collect respective data anew has to be seriously discussed in order to eliminate any doubt. Eventually, it is even not necessary because the present small series of publications reveals that the crucial processes in the HST occur during heating-up, a long time before reaching the holding temperature. But anyway, before engaging into the laborious and time-consuming effort of new data collection, a new mathematical evaluation of the existing datasets could be tried, based on modeling and frac-

ture mechanics, at the same time taking into account the new thoughts, findings, estimations and calculations published in the present paper series. A neutral institution, such as a competent, strictly neutral professorship free of commercial or other interest, should therewith assess the hypotheses made in the present papers in a mind of constructive criticism, but avoiding lunatic safety demands.

3.2 Reference to practical findings

Most of the estimations elaborated above are minimum estimations based on conservative assumptions, or they consider only one in many aspects so that it is sure that more safety has to be added. This is the reason why it is impossible to derive thereof a precise number for the overkill of glass plates taking place in the HST ovens working worldwide. The way how the different impact factors combine is actually unknown; it seems, however, that the relation of breakages between buildings and the HST is low. Theoretical calculation is difficult, was never done up to date in sufficient quality and is—if at all—only possible using sophisticated, detailed finite-element simulation tools and accounting for each of the properties discussed in the different parts of the present small series of papers.

A definitive statement could be obtained in a pragmatic way by evaluating the breakage rates observed on buildings after assuredly proven HST. However, this is difficult if countable events (i.e. breakages on buildings) do not occur over years. Therefore, the practical experience of SG during the last years shall be described as an example; other glass producers are more than welcome to publish their own experience for support.

All producers of glass panes according to EN 14179-1 are obliged to make available statistical data referring to HST-glass production. In Germany this is even already mandatory since 2002, the year of the factual introduction of the new HST standard through the respective prEN. Numbers, sizes, masses, glass kinds (clear, tinted, coated etc.) and the related breakage numbers in the HST furnaces are systematically recorded in the factories concerned. The four SG affiliates in Germany are under one quality directory. The respective data are systematically information-technically merged since 2011, and only since that time they are available for cross-plant evaluation. Thus the production of

17209 t of HST-glass was concretely recorded between 2011 and mid-2017 (both years included), and 2793 breakages (plus some collateral damages) were counted therein (Rubbert 2018). This refers to 1 spontaneous breakage in 6.2 tons of glass, a common breakage rate also already published in e.g. (KASPER and Serruys 2002). Extrapolated onto the whole time during which the new HST was factually applied (from 2002 to 2017 included), c. 42,000 t of HST-glass were produced in these four facilities, including c. 6900 breakages in the furnaces.

In spite of the proven too high holding temperature, only four breakages on façades were claimed out of this complete 16 year's production. Curiously, they all originate from the same factory, the same production period (2009) and are concerned to the identical built-in situation where HST is not forced by law since there's no danger at all to life. Many coincidences at the same time; however, a small number of unreported single breakage cases cannot be entirely excluded, even if this is not probable in view of the high sensitivity of both the clients and the public media. "Glass rain on a building" is always a good lead story, often enough read before and shortly after 2002, discrediting the use of toughened glass, and being the core cause for the development of EN 14179-1. Indeed, every customer complaint at SG concerning a spontaneous breakage (except the small series mentioned above) dates from before the factual introduction of pr-EN 14179-1; remember that spontaneous breakages of un-soaked glass can take place up to 30 years after toughening. Retrospectively, the factual introduction of pr-EN 14179-1 marked a turning point in the history of HST-glass.

Consequently, hypothesizing that the four reported breakages are indeed breakages after HST, that there might be a dark figure of three more,¹⁵ and that breakages after HST do not show the normal latency period of c. one year, leads to the estimation of a statistical residual breakage risk of one in 6000 t of HST-glass. This means that the formerly envisaged goal of one breakage in 400 t (see e.g. KASPER and Serruys 2002) is in reality excelled by a factor of fifteen. A number of 6900 breakages in HST is accompanied by only (estimated) seven on building, i.e. the relation is 0.1% only, contrariwise to the 1.5% prospected in the

¹⁵ There is no concrete reason for the additional number of three; it was just chosen arbitrarily to obtain a "round lot" in the following calculation.

mentioned publication by extrapolation of the breakages recorded during the holding time of the HST. Said 1.5% refer to c. 100 breakages; however, this counts only for SG products in Germany; the corresponding number from competing HST-glass producers would have to be added, so that we would expect much more, eventually up to 500 breakages only in Germany. This high number would certainly not escape to the alertness of clients and public media.

In summary, based on the practical finding at SG, the efficiency of the HST is estimated to be 99.9% instead of the prognosticated (extrapolated) ratio of 98.5%, and the statistical residual breakage risk of Heat Soak Tested Thermally Toughened Safety Glass is estimated to be one in 6000 tons of HST-glass.

4 Summary and conclusions

This chapter summarizes en masse the three parts of our small series of publications.

Put simply all new findings and interpretations, our small series of papers on spontaneous breakage of HST-glass should cause a paradigm shift in the assessment of the safety of HST-glass. It gives really new insights into spontaneous glass breakages, revealing that there's a factually huge quantitative and qualitative difference between the spontaneous breakages observed on buildings and those forced in the HST. Chapter 2.2 in Kasper (2018) reveals that one of the main reasons for this is due to the higher temperature in the HST in comparison to facades, causing almost all nickel sulphide inclusions¹⁶ to initiate breakage, independently of their detailed composition; in relation to this, only 39% of (hypothetically) the same population of nickel sulphide inclusions could cause breakage on façades.

The evaluation of a dataset obtained already in 1999 on toughened and heat-soaked Cathedral glass (a patterned glass; Chapter 2.3 in Kasper (2018)) reveals that not only nickel sulphide inclusions, but also refractory stones lead to significant breakage in HST. Surprisingly, the general form of both time-to-breakage curves is nearly identical although the inclusions in the Cathedral glass definitely do not show a phase transition like nickel sulphide. This proves that the virtual nature of

the inclusions themselves is not the factor controlling the time-to-breakage curve in the HST. Contrariwise, the inevitably heterogeneous heating-up of the glass in the HST, inducing strong thermo-mechanical forces (= temporary stress) into the glass, has a crucial influence on both the breakage devolution and the breakage rate. In particular, this makes clear that the α to β transformation speed of the nickel sulphide inclusions is not the process determining the time to breakage. This observation solves a long-existing question, namely why the time-to-breakage curve in the HST cannot be explained (i.e. calculated by combining and summing up a reasonable ratio of different NiS_x species) by the measured phase transformation speed of the different relevant NiS_x species.

A trial in the Saint-Gobain laboratories in China and Korea ('C/K trial'), described in Kasper et al. (2018), proved what beforehand was only assumed, namely that nickel sulphide inclusions are found everywhere in raw glass section, but their alignment is not exactly uniform. A signature of gravitational settling is identified in all respective datasets ('C/K-trial', 'HST-EU' and 'Building'), and a model is developed on the basic position localization and size spectrum curves in order to explain settling by different influencing factors. Subject to the HST after thermal toughening, only 25% of the nickel sulphide inclusions identified beforehand in the annealed glass lead to spontaneous glass breakage in the C/K-Trial, virtually because the survivors are too small or located in the compressive zone of the glass. A significant conclusion from this is that a HST cannot eliminate every nickel sulphide inclusion, but the critical ones do lead to breakage in HST although they are the minority, even here. Important to mention that, additionally to the α to β transformation, also strong thermal stress induced in HST during heating-up is leading to breakage due to otherwise "subcritical" inclusions of any kind (naturally also including smaller and more laterally-located nickel sulphide inclusions).

Additionally, the breakage ratio curve as a function of the nickel sulphide inclusion size is derived. It could serve in a future precise simulation of spontaneous breakages.

The same is true on buildings if un-soaked toughened glass is used. Also in this case, only the "critical" nickel sulphide inclusions are able to make the glass break. But there's a significant difference: "criticality" has a quantitatively different meaning in building application. The external conditions differ significantly; the

¹⁶ This is, naturally, only true if they fulfill the other necessary conditions for breakage initiation, namely being situated in the tensile zone of the glass and having a diameter making them critical in their real position.

main relevant differences are twofold, namely (a) the temperature regime and (b) the fact that the glass in the HST is subject to strong transient thermo-mechanical stress since this treatment is far from being isothermal, as already mentioned above. Consequently, breakages occur much more frequently in HST than (with hypothetically the same glass) on buildings, even taking into account that wind load is playing a certain role there.

This means that the HST according to EN 14179-1(2005) sorts out many more glass panes than virtually necessary. On the one hand the evaluation of the physical properties of possible nickel sulphide inclusion compositions, on the other hand the statistical comparison of datasets ‘HST-EU’ and ‘Building’, allow us now to make an estimate of respective proportions.

Namely, according to the estimations out of the different aspects of the difference between HST and on-building conditions onto breakage (summarized in Table 4), many, or even (looking at some of the calculated figures) nearly all breakages in HST are “irrelevant”, i.e. not necessary to assure the safety of HST-glass in its façade application. Additionally, it is known since a long time (Kasper 2000) that 80% of the breakages in the HST occur during its heating-up period. This leads to the conclusion that under HST conditions the holding time could be completely needless for adequate safety of HST-glass. So, keeping the glass at constant $(290 \pm 10)^\circ\text{C}$ for 2 h in excess to the real need certainly amends the HST-glass safety, but most probably to such an extent that this is virtually not relevant in practice, and the same is even true for the late part of the heating-up period. This is also a striking argument for that the assumption that breakage behavior in HST and on buildings would be similar, as applied in former safety estimations (e.g. Schneider et al. 2012; KASPER and Serruys 2002), is definitely not true.

To prevent misunderstandings: The present paper does not ask for changes in the HST process although such could possibly be derived of it. It intends to demonstrate that the HST, as it is made since 2005 and will be made after its current amendment, leads to an extremely low risk of residual spontaneous breakages in HST-glass. Today, vandalism or a construction or design fault (e.g. glass-metal contact due to unforeseen movement of the glass in the frame) seem to be the most frequent causes for façade glass failures (Weko 2017).

Due to previous scientific findings [(29) YOUSFI], the conditions of the HST standard have been modified by the European standardization committees in 2016.

Table 4 List of “irrelevant” breakages estimated from different findings

Paper, part	Chapter	Keyword	Irrelevant breakages	Remark
One	2.2.	Crystallization of liquid NiSx	> 61%	Other effects (size, position, roughness) to be added
Two	3.2.	Thickness impact in H S T	> 89%	All major effects included
	3.3.	Thermal dilatation impact + Crystallization	> 98%	All major effects included
Three	2.1.	Position (HST-EU): Spike	> 46%	Other effects to be added: Impact of big inclusions → truncation correction
		Ditto, including truncation correction	> 79%	
	2.2.	Size (HST-EU)	> 46%	Average of both estimations. Only size impact; estimation by mathematic-analytic evaluation
		Size histograms	> 57%	Only sizes < 200 μm evaluated; other effects to be added
		Minimum // maximum	46% // 98%	

Paper One: Kasper (2018), Paper Two: Kasper et al. (2018), Paper Three: The present paper

Mainly, the average holding temperature is reduced to be safely below 280 °C. This was found to be necessary to allow complete α to β transformation of every potentially dangerous nickel sulphide inclusion, including the seldom ones with exotic over-stoichiometric composition. Therefore, temperature reduction makes the HST safer.

To reassure this, the present findings and evaluations should be checked by calculation based on fracture-mechanics simulation, carried out by an independent institute. Another possibility would be to build up a “Bernoulli tree” of known influencing (probability) factors in order to quantify the differences between the conditions of HST and Building. Naturally, this check has to be based on (or to correlate with) the known facts, but also on the new findings and observations described in the present series of papers. The latter disallow some simplifications of the existing models, e.g. disregarding the strong impact of the rough surface of the nickel sulphide inclusions, the relatively complicated position alignment curve at breakage and the influence of the diversity of their chemical composition.

New presentations and concepts in the present paper series are:

- Ideas on properties of nickel sulphide inclusions in glass depending on their detailed composition, including the presence of Ni_9S_8 , Ni_3S_4 and over-stoichiometric NiS_x ($x > 1.012$). Consequences from this on their different criticality on buildings and in the HST.
- Experimental proof that also refractory stones can lead to massive breakages in HST, and that their time-to-breakage curve is—except for some minor details—a sigmoid curve like for nickel sulphide inclusions with even identical expansion into holding time.

Conclusion that the time to breakage does not really depend on the transformation kinetics of NiS_x , but on temperature heterogeneity in the HST oven and, possibly, on fatigue due to water vapor diffusion in the primary cracks caused by the inclusions or stones.

Obviously the processes in the HST cause increased breakage rate of the toughened glass, in relation to the situation on buildings. The same is true for sufficiently large refractory stones.

- Experimental proof that the nickel sulphide inclusion repartition in raw glass is, to some extent, ran-

dom, but not uniform because the signature of gravitational settling is observed.

- Statistical-experimental proof that only nickel sulphide inclusions with size $> 54 \mu\text{m}$, situated in the middle glass portion, are able to lead to breakage in HST.

On buildings, this limit is higher, $> 76 \mu\text{m}$. The difference (+ 41%) in the results is absolutely significant.

- Large inclusions ($> 450 \mu\text{m}$) always lead to high breakage risk $> 90\%$.
- Experimental proof that only a minority of the nickel sulphide inclusions factually existing in raw glass, but more than c. 1/4, leads to breakages in the HST carried out during the detection trial in China/Korea (dataset ‘HST-C/K’).

Breakage rate in a standard HST (dataset ‘HST-EU’) is higher, mainly because the glass panes are larger and thicker.

- Experimental and statistical proof that the HST destroys a significantly higher number of glass panes than ever would break on buildings. “Criticality” of an inclusion is different under the different temperature conditions. Approximately, 23% to 98% of the breakages observed in HST are useless.
- Proof that the safety of “Heat-Soak Tested Thermally Toughened Safety Glass” is actually strongly under-estimated. Out of the reasons enumerated above, the respective calculation is obsolete and needs independent revision. However, under its present form it gives a lower limit of the HST-glass safety, but, after all, far below its true value.

Another topic shall eventually be regarded. Like any science and research, also knowledge about spontaneous cracking of toughened glass was and still is subject to development. As an example, at the beginning of the problems with spontaneous cracking of toughened glass, in the late 1950’ies, the possibility to make accurate thermodynamic calculations in short time was not yet developed by lack of electronic computers. A calculation that, today, can be carried out precisely within 10 minutes using, e.g., the combination of an EXCEL sheet and HSC® (Roine 2011), as done by the author of this paper, took, at that time, several days and was made by a human computer, using a mechanic calculation machine, a pen and several big sheets of paper. This is the reason why, instead of making exact calculations, estimations (e.g. based on the well-known ELLING-

HAM diagram developed for steel production) were made without taking into account exact concentrations, ignoring that the latter can reverse a chemical equilibrium under certain circumstances.

As already pointed out, driving our theme forward is one of the main aims of the present paper series. During long-time occupation with it, from the beginning on based on scientific research like the measurement of the α to β transformation of NiS_x (e.g. Bordeaux and Kasper 1997a), precise thermodynamic calculations (e.g. Kasper and Stadelmann 2001) and accurate statistical evaluation of breakage events on buildings [e.g. Bordeaux and Kasper 1997b] and in HST [e.g. Kasper 2000], the author of the present paper has come to the conviction that some of the hypotheses published earlier do no more correspond to the actual state of knowledge and experience.

It is, however, not possible here to disprove all of the misinformation and misleading personal opinions published in numerous articles in non-scientific glass journals. But even in scientific papers, some errors persist and can lead to a wrong assessment of the problem. The following list of examples is certainly not complete.

- The opinion that nickel sulphide inclusions are solely and simply composed of NiS(1:1) and overstoichiometric NiS_{1+x}, and keeping quiet about the real (admittedly much more complicated) situation and its consequences is misleading. The compositions possibly present in these inclusions in float glass are NiS, NiS_{1+x}, Ni₉S₈ and, after transformation, Ni₃S₄. Thereof, solely NiS and NiS_{1+x} are subject to a relevant α to β transformation and can lead to spontaneous breakages. Ni₇S₆ often mentioned [e.g. Karlsson 2017] is not existing at ambient temperature because it is subject to a fast, unquenchable transition into Ni₉S₈ at 400 °C.
- The suspicion that nickel sulphide inclusions could generate from diluted/solvated NiO components can be refuted by thermodynamic calculation applied onto the conditions of float glass melting and almost all soda-lime-silica glasses. The reverse is thermodynamically proven and even obvious, looking at the nickel sulphide inclusions' photographs: They are unstable in this glass melt and are subject to continuous digestion due to the oxidizing nature of the (float) glass.

On the other hand, float glass coloration using NiO is not leading to increased numbers of nickel sul-

phide inclusions and widely used in the flat glass production as already mentioned earlier here.

- The same is true for the nickel content of the fuel oil. The author is convinced that there is no way to make nickel sulphide inclusions thereof, as already explained earlier here, and gas fired float furnaces face the identical problem of nickel sulphide inclusions. One example of the latter is given in the present publication series.
- Thermodynamic calculation makes it also more than doubtful that minor changes in the glass composition could lead to safety from spontaneous breakages (Karlsson 2017). Under the objective chemical point of view, as long as sulphate is used as the fining agent and it is not guaranteed that no nickel containing metal particles are polluting the glass batch, nickel sulphide inclusion formation cannot be excluded. One example for a glass matrix with significantly higher melting temperature and, in spite, proven nickel sulphide inclusions, is given in Gelderie and Kasper (2008).

On the other hand, if no sulphate is used (e.g. in borosilicate glass), there are proven no nickel sulphide inclusions. But, at least from today's point of view, eliminating sulphate from the float glass batch is unimaginable.

- Another unsubstantiated hypothesis is that the formation of nickel sulphide inclusions could be prevented by making the glass batch and the melt sufficiently oxidizing Brungs and Sugeng (1995). To the authors knowledge, this idea was disproved a long time ago Quiquampoix and Wagner (1977) but probably not explicitly published, just like many other "negative" results.
- The author found out that the seemingly different literature values of the expansion of NiS at α to β transformation are, at second sight, caused by the different temperature levels during the respective trials and the interference of the thermal expansion there-between. E.g., high temperature XRD measurement of pure NiS results in the net expansion at 379 °C, whereas the density measurement carried out on the (quenched) species at ambient temperature includes the different thermal expansion of both on cooling. It is, consequently, of crucial importance to use the right expansion coefficients in simulations.

- The stability limit of (pure) β -NiS is, indeed, 379°C. But, at least before Yousfi's publications already mentioned, it is often not mentioned that, depending on the detailed composition, the over-stoichiometric NiS_{1+x} can be stable down to 280°C. Another important aspect is that the so-called "limit temperature" is, thermodynamically spoken, the temperature where α and β phase are present in the substance to 50% each, and the transition follows a sigmoidal curve with, a priori, unknown steepness and significant tailing into both directions. Consequently, in order to obtain complete transformation, it is necessary to keep a safety distance to the "stability limit". Ignoring this lead, in the past, to the idea of a "short time" HST at 350°C, with the (non-published) consequence of numerous breakages on one building. This trial should not be repeated.
- The present publication proves, among others by thermodynamic facts, that the safety of HST-glass cannot be estimated correctly by simple extrapolation of time-to-breakage data of the HST. Regardless doing this results in a strong under-estimation of the real safety of the HST-glass. The latter could maybe serve as an upper limit, but in reality its safety against spontaneous breakages is much better.
- The pretention that the safety of HST-Glass against spontaneous breakages is not or not much better than that of un-soaked glass is occasionally heard in certain regions of the world as an oral argument against its application, but (to the author's knowledge) never published officially and explicitly. This myth is proven not true.
It can, however, be debated about "Is Heat Soaking worth it" [Voelker et al. 1991, not really on the actual state of R&D but, however, still interesting to read], and the arguments that the spontaneous breakage problem is not frequent on façades (sic!) and that the HST is expensive and destroying, are doubtlessly striking. On the other hand, up to date, no other prevention method has come out of its fledging state. Some proposals exist (Karlsson 2017), based on laboratory trials, but up to date, none among them has come to ripeness of use in industrial application. Consequently, it can be said that the HST is the only serious, accepted and standardized prevention for the nickel sulphide inclusion caused problem of spontaneous breakage.

Not specifying the HST in certain critical cases, even where its application is not strictly prescribed by building regulation, is risky and can lead to severe consequences as described e.g. in (Brungs and Sugeng 1995) for the Waterfront Place Building in Brisbane; more such but unpublished examples exist definitely. If the replacement of a series of broken glass panes causes a high multiple of the production costs, or if replacement is virtually impossible due to the façade design, applying the HST should be the voluntary standard because it is, in relation to the risk, not really expensive, and it guarantees the panes' safety against spontaneous breakages.

To do's/open questions:

- Quantification of breakage probabilities based on fracture mechanics computer model, taking into account the findings mentioned above, especially the clear differences between HST and on-building conditions.
- Critical testing of hypotheses of the present paper by independent scientists' cross check.

The best way for this would be checking based on independently gathered datasets. The author offers help for simultaneous evaluation if wanted.

The database of the present publication can be made available for own calculation, check and discussion.

Please mail to Andreas.Kasper@Saint-Gobain.com or AKasper@gmx.com. Alternatively, a data file can be downloaded from the Journal's website.

- More measurements and evaluation of samples produced in the C/K trial (only non-broken remain) are currently carried out, among others in our own laboratories, in order to verify/falsify said hypotheses, and to allow more precise calculation. Publication is planned.
- Possibly, new time-to-breakage data from HST according to EN14179-1(2016) could be collected, quantifying the impact of the new HST conditions, mainly the lower holding temperature and the limitation of the heating-up rate.

On this background, ISO 20657:2016 Annex E, the on-line HST practiced e.g. in Japan, appears in a different light. Therein, the relevant temperature range is $(220 \pm 20)^\circ\text{C}$, and the time the glass temperature transits this interval from above is only (minimum) 12 minutes.

Also if the HST is made under these, in comparison with EN 14179-1, very different conditions, no problems with spontaneous breakages arise in practice (Sakai 2017). The conclusion of this is that a safe product is also made applying much lower temperature and much shorter treatment time; this supports the conclusions of the present small series of paper.

In reference to practice, and in spite of this to-do list, one thing is sure. The previous safety estimation for the HST in Schneider et al. (2012) is not totally invalid. Since it is strongly under-estimating the factual safety, it fixes its lower limit. The present paper reveals that this safety estimation is an absolute worst-case scenario. Coarsely spoken, more than half of the breakages in HST are irrelevant and (un-soaked) would not lead to breakages on buildings. However, this does not mean that the real safety of the HST is only the double of the value actually estimated by extrapolation of the breakages at holding time.

Factually, the HST makes all panes break that are critical on building, and additionally, as a safety margin, approximately the same number more; the latter are only critical in HST, but not on building. This argument leads to the conclusion that the residual probability of spontaneous breakage eventually cannot be deduced quantitatively from the HST results. It might be – not zero, this is not possible – but nearly infinitely low.

Consequently, it is of minor concern if the holding temperature is reduced by 30°, or if it is 2–4 h.

Regarding the conflict concerning the obligation of periodical third party surveillance of the HST parameters on EU level, the present results reveal that some incertitude in the exact holding temperature can doubtlessly be tolerated in order to find a respective compromise. It seems, however, much more important to assure that the HST is really and truly done as a complete 100%-control of the toughened glass, without any single exception. Statistical records of the factories certified according to EN14179-1:2016 can be a proof of this; also, application of thermochromic colors, positively proving that the HST was carried out, is disputable and could be further developed.

It is more than probable that the new conditions of EN 14179-1:2016, including a scientifically founded reduction of the holding temperature and a limitation of the heating-up speed to three degrees per minute, will allow to produce a really safe “Heat-Soak Tested Thermally Toughened Safety Glass” that thoroughly

fulfills all expected reliability demands for its typical applications in buildings.

The authors thank R. Bink, J. Hilcken, A. Minne, F. Rubbert, J. Schneider and A. Schusser for fruitful discussion, H.W. Kuster for unbureaucratic leeway and S. Kasper for uncomplaining personal support.

Compliance with ethical standards

Conflict of interest The author states that there is no conflict of interest. However, he pronounces that he is a member of industrial research and development. The results published in the present paper refer to an industrial procedure practiced in the factories of his employing enterprise. Eventual practical consequences drawn from the present results can impact public safety. He is aware of the entrepreneur responsibility resulting thereof. However, he disallows any personal liability. He assures that every fact and result of the present paper has been compiled and evaluated with highest diligence, but he also emphasizes that accidental errors or misinterpretation can never be entirely excluded.

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Glossary

$\pm s$	Standard deviation.
σ, μ	Characteristic parameters of the Log-Normal function.
α -, β -NiS	High and low temperature phase of nickel sulphide, respectively. Another phase exists at even lower temperature but does not play a role in the frame of this publication.
‘Building’	Dataset analyzed in the present publication; breakage centers obtained on buildings in Europe
C/K trial	Trial carried out in China and Korea, leading to identification of 140 nickel sulphide inclusions.
EN	European Standard
HST	Heat Soak Test, if not otherwise mentioned carried out according to EN 14179-1:2005; Standard first revised in 2016 with significant changes.
‘HST-EU’	Dataset analyzed in the present publication; breakage centers obtained from HST’s in Europe

- ‘HST-C/K’ Dataset analyzed in Kasper et al. (2018), obtained from SG trials (‘C/K trials’) in China and Korea where a HST was carried out on samples containing previously detected nickel sulphide inclusions.
- HST-glass Acronym for “Heat-Soak Tested Thermally Toughened Safety Glass according to EN 14179-1”
- MPa Mega-Pascal (pressure unit)
- N Number (dimensionless)
- NiS Nickel sulphide, atomic composition 1:1
- NiS_x Nickel sulphide with non-stoichiometric composition, or variability in composition of nickel sulphides
- P Probability
- R² Coefficient of determination (a measure of the precision of curve fitting)
- SEM Scanning Electron Microscope
- SG Saint-Gobain (company producing, among others, flat glass)
- t STUDENT’s parameter for statistical testing; t is the difference between two values in terms of number of corresponding standard errors.
- x Molar or atomic fraction (e.g. in NiS_x)
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